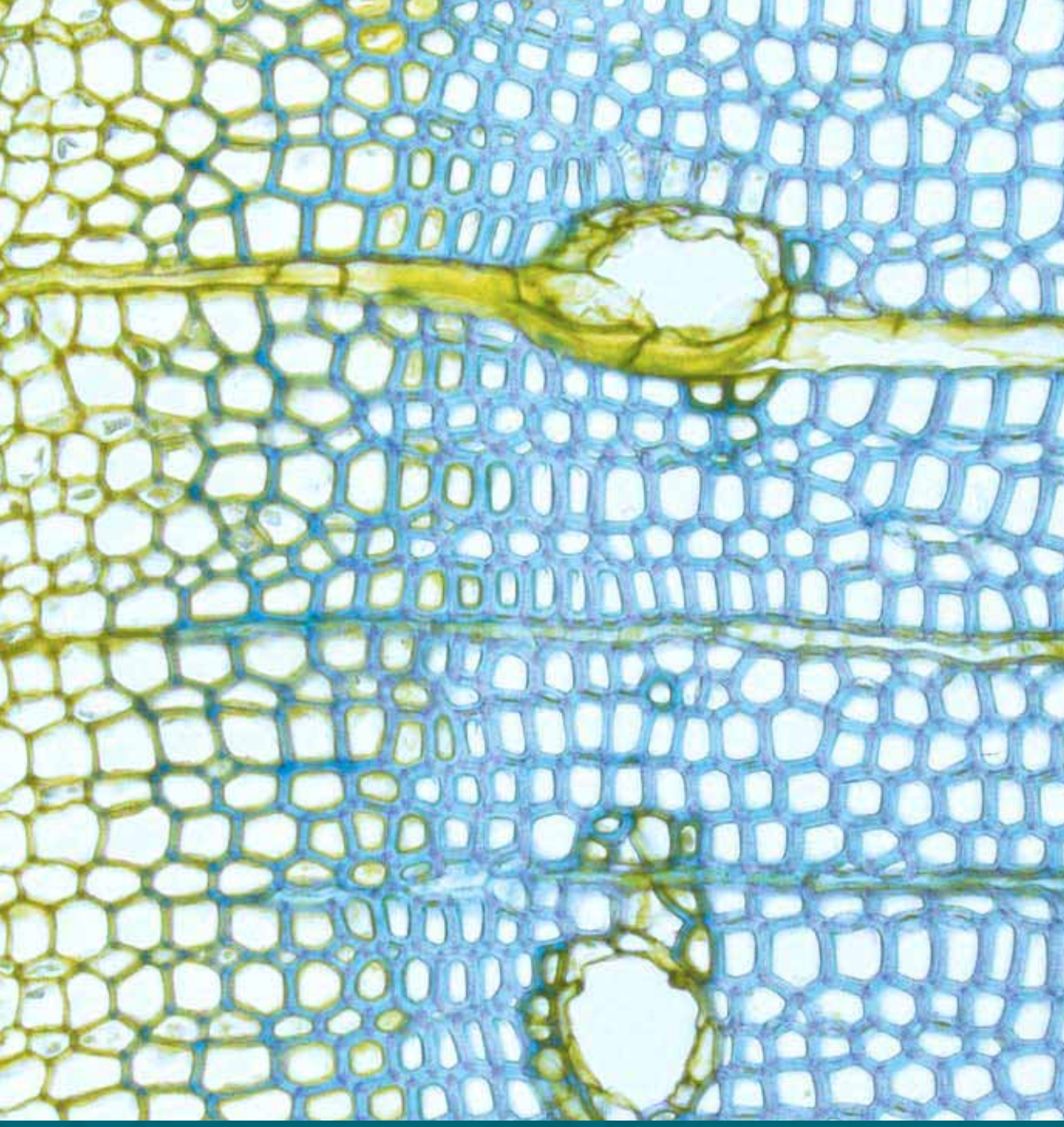

Wood Material Science
Research Programme

Wood Material Science and Engineering Final Report

Finnish-Swedish Research Programme 2003-2007

Final Report

Final Report

Wood Material Science and Engineering Research Programme

Wood Material Science
Research Programme

Stockholm 2007

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Foreword

In discussing the competitiveness of nations or entire continents, catchwords like 'competition with competences' and 'R&D infrastructure' keep cropping up more and more often. This reflects the fact that world-class companies, in striving to exploit lead markets, prefer to establish their core functions in countries where they have access to top-ranking knowledge and the facilities to apply it. At the same time, it is widely recognised that there is a need to strengthen the scientific base in key competence areas and to intensify the co-operation between universities, research institutes and industry.

Moreover, the present national funding systems easily lead to duplication of projects in neighbouring countries: the same topics are concurrently financed in similar projects, which may lead to weakly cumulative knowledge and a waste of financial and human resources.

Realising these needs and shortcomings, five key public funding organizations in Finland and Sweden, i.e., the Academy of Finland, the Ministry of Agriculture and Forestry in Finland, the Finnish Funding Agency for Technology and Innovation (Tekes), the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning (Formas) and the Swedish Governmental Agency for Innovation Systems (VINNOVA), initiated preparations for a joint programme in the area of wood material science. Started at the beginning of the new millennium, this effort resulted in the launching of the Finnish-Swedish Wood Material Science and Engineering Research Programme (programme period 2003–2006, subsequently extended to the end of March 2007).

Bringing together five funding organisations, 30 research institutes and universities and 50 enterprises, the programme has been a unique undertaking from its very conception. Apart from fulfilling the specific goals set for it, the programme has served as a pilot for the European Union in introducing concepts such as the European Research Area (ERA), Technology Platforms and ERA-Nets. Comprising 17 projects with a total volume of 20 million euros, the programme has established a solid knowledge base in the field of wood material science. At the same time, it has strengthened the co-operation between key public funding agencies in Finland and Sweden, and created cross-border competence networks, while bringing industry closer to research by engaging it in project preparation and realisation.

Drawing on the experience gained in this Finnish-Swedish co-operation programme, the concept of the trans-national wood material science programme was expanded to European Union level with the launching of the ERA-Net programme "WoodWisdom-Net" in the autumn of 2006. Involving eight European countries, it will constitute an important new financial tool for forest-based industries.

On behalf of the Wood Material Science and Engineering Research Programme I would like to thank the funding organizations, programme board members, programme directors and co-ordinators, researchers and company representatives in projects for their support and contribution to the programme and its development. I hope that the programme, for its part, will serve as a foundation for new knowledge-based growth in the forest-based industry sector.

Lahti, March 2007

Pekka Peura

Chairman of the Programme Board

Contents

THE SUB-PROGRAMME FOR BASIC RESEARCH PROJECTS

1 Identification of EST polymorphisms and candidate genes related to growth and wood properties in silver birch (IDEST)

FINAL REPORT

Name of the research project	**Identification of EST polymorphisms and candidate genes related to growth and wood properties in silver birch**
Coordinator of the project	Tapio Palva

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Identification of EST polymorphisms and candidate genes related to growth and wood properties in silver birch**
Project period	1.7.2003–30.6.2007
Organization in charge of research	Skogforsk
Sub-project leader	Bo Karlsson
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Skogforsk, Ekebo 2250, 26890 Svalöv, Tel. +46 418 471305 Fax +46 418 471329
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	157 623
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	157 623

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name of the sub-project 2	**Identification of EST polymorphisms and candidate genes related to growth and wood properties in silver birch**
Project period	1.1.2003–31.12.2005
Organization in charge of research	University of Helsinki
Sub-project leader	Tapio Palva
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Dept. of Biological and Environmental Sci., Div. of Genetics, PO Box 56, FI-00014, Helsinki Phone +358-9-191 59600
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	168 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	168 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Markku K. Aalto, Ph.D. postdoc	M	University of Helsinki		University of Helsinki
Kalle Ojala, student	M	University of Helsinki		University of Helsinki

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	M.Sc.	M	Kalle Ojala, 1977, M.Sc.	University of Helsinki	Tapio Palva, University of Helsinki

The project is continuing until the end of year 2007. Thus the report describes the current status and briefly the work plans for the rest of the year 2007.

Abstract

IDEST is a multidisciplinary research project combining classical tree breeding with advanced molecular and genomic approaches to facilitate the development of new birch clones with improved growth and wood properties. The project relies on classical tree breeding, existing breeding material and a birch expressed sequence tag (EST) collection for the identification of candidate genes associated with the relevant properties. Identification of such candidate genes and EST polymorphisms will be instrumental in development of molecular markers that provide rapid and early identification of the desired individuals/progeny in breeding programs. The objective of this work is to elucidate the molecular basis of the natural variation in growth and wood quality in birch and utilize the obtained information to develop molecular markers for early selection of these traits in breeding programs.

Tiivistelmä

Idest on monitieteellinen projekti, joka yhdistää klassisen metsänjalostuksen ja modernin molekyyligenetiikan ja genomiikan koivukloonien löytämiseksi, joilla on paremmat kasvu- ja puuominaisuudet. Työ perustuu olemassaolevaan jalostusmateriaaliin sekä suureen EST (expressed sequence tag) kokoelmaan, joiden avulla voidaan tunnistaa haluttuihin ominaisuuksiin liittyviä kandidaattigeenejä. Näiden geenien tunnistaminen on edellytys molekyylimarkkereiden kehittämiseksi, joita voidaan jatkossa käyttää haluttujen yksilöiden/jälkeläistöjen valintaan jalostuksessa. Työn tavoite on on ensisijaisesti tunnistaa kasvu- ja puuominaisuuksien luonnollisen variaation molekyläärinen tausta koivulla ja käyttää tätä tietoa näiden ominaisuuksien tunnistukseen soveltuvien markkerien kehittämiseen.

Sammanfattning

Idest är ett multidisciplinärt project som kombinerar klassisk skogsförädling med modern molekylärgenetik och genomik för att identifiera nya björkkloner med bättre tillväxt och vedegenskaper. Projektet baserar på existerande förädlingsmaterial samt ett stort antal EST (expressed sequence tag) sekvenser för identifiering av kandidatgener associerade med relevanta egenskaper. Identifiering av sådana kandidatgener är en förutsättning för utveckling av molekylära markörer som kan utnyttjas för selektion av intressanta individer/avkommor i förädlingsprogram. Målsättningen med arbetet är att kartlägga den molekylära bakgrunden till den naturliga variationen i tillväxt- och vedkvalitetsegenskaper hos björk och att använda denna information för utveckling av molekylära markörer för dessa egenskaper.

1.1 Introduction

1.1.1 Background

In general, breeding of forest trees is based on a cyclic course, with crossing, testing and selection phases. After each cycle growth and quality traits are improved. This is a long-term process, especially for long living Scandinavian forest tree species. One cycle can take about 20–25 years depending on tree species, at what age the tree starts to flower and when the evaluation of the tests can be made. One way to shorten the breeding cycle is to develop early tests allowing diagnostic testing of young seedlings to determine, which individuals give the best yield in mature state.

In the beginning of 1990s methods for using molecular markers in early tests started to be developed. The basic idea is to find molecular markers that are located as close as possible to a certain segment of a chromosome that exerts a larger influence on a specific trait. In forest trees, there have been some reports of DNA marker-facilitated detection of loci putatively influencing variation in a range of traits. QTLs have been found e.g. for frost tolerance in an *Eucalyptus nitens* family, bud phenology control in Populus, age dependent wood density in radiata pine and physical and chemical wood properties in loblolly pine. Concerning the profitability of marker aided selection (MAS) it has been shown that significant genetic and financial gains are possible from selection using DNA markers.

The markers mostly used for quantitative trait loci analysis are RAPDs (random amplified polymorphic DNA), RFLPs (restriction fragment length polymorphisms), AFLPs (amplified fragment length polymorphisms) and SSRs (simple sequence

repeats). These markers are considered to be neutral and usually targeted to non-coding regions. Instead of using anonymous markers, the use of markers directly targeted to expressed genes would be beneficial. Due to the ongoing sequencing projects of expressed genes from woody plant tissues it is now possible to develop ESTP (expressed sequence tag polymorphism) markers for important traits, like growth and wood properties. When a clear connection between specific genes and the desired phenotypic trait is not readily available, the identity of the genes involved can be obtained by expression profiling of genes corresponding to isolated ESTs. Microarray analysis of thousands of ESTs, each corresponding to an expressed gene, defines genes with specific temporal or spatial expression patterns and aids in identification of those genes, which are involved in development of specific traits. This approach enables the analysis of the differences in the levels of expression of specific genes between individuals exhibiting different phenotypes for the desired trait. Thus, microarray analysis will help both to identify genes for ESTP analysis and to obtain target genes for analysis of correlation between expression and phenotype. Both ESTPs and candidate gene expression levels can then be used as markers for early selection of material from breeding programs.

1.1.2 Objectives

The overall aim of the research is:
To elucidate the molecular basis of the natural variation in wood quality in birch and utilize the obtained information to develop molecular markers for early selection for growth and wood property traits in breeding programs.

The specific objectives are:

1. To characterize regulons involved in growth and wood properties by gene expression profiling.
2. To identify genes whose expression level is correlating with desired growth and wood property traits.
3. To identify ESTPs and/or other (expression) polymorphic markers that correlate with growth and wood density for early selection of desired traits.

1.2 Results and discussion

The material for IDEST is a birch full sib family belonging to the ongoing tree improvement work at the Forest Research Institute of Sweden (Skogforsk). The parents of this full sib family are included in a study of wood and growth properties of birch and are divergent for these property traits. The family consists of 35 cloned individuals, totally 525 trees, planted 1999 in three locations in the south of Sweden. The family has been expanded by a controlled cross, made in spring 2003 between the parent trees. The seeds were sown in June 2003 in order to get a sufficient number of segregating individuals for the marker study. These controlled crosses (293 seedlings) were sowed in August 2003 and transplanted to a nursery test June 2004.

Characterization of genes and regulons involved in determination of growth and wood properties is central to this project. To this aim we have performed expression profiling of a subset of birch genes by utilizing a small microarray containing ~1300 selected cDNAs. For the array hybridization RNA was isolated from leaves and stems of parent trees. The stem tissues were collected twice during the growth period, in the middle of May 2003 and in the beginning of July 2003 for samples that were representative for both early and late wood formation. These samples represent the extremes in wood quality from the sib family. This analysis led to the identification of a small number of genes, whose expression was differentially regulated in the parental trees and during early and late wood formation. The genes showing differential expression include some obvious candidates e.g. genes involved in lignin biosynthesis but also some regulatory genes, which are of interest for further analysis (Table 1).

These expression data were verified for a subst of candidate genes by RNA gel blot hybridizations. Additionally we characterized genes for some potential growth regulators i.e. CO-like genes in more detail. This analysis has demonstrated that at least one of these, BpCOL2 controls both root and stem growth in birch (manuscript in preparation).

For progeny analysis we initially used the above genes, along with other candidate genes known or predicted to be of importance to wood formation and growth by a macroarray to identify genes that

Table 1. Partial listing of genes differentially expressed in parental trees trees with desired growth parameters (A) compared with the control parent (B).

Down in A1/B		
Acidic thaumatin-like protein (PR-5)	M90510	Arabidopsis
CONSTANS	A56133	Arabidopsis
Omega-3-desaturase (FAD8)	D14007	Arabidopsis
Glycosyl hydrolase family 14	NP_567523	Arabidopsis
PIN1-like auxin transport protein	AF190881	Populus
HMG-CoA reductase (HMG1)	JO4537	Arabidopsis
Alternative oxidase (AOX)	AJ319899	*V. unguiculata*
Down in A2/B		
Homeobox protein (ATHB6)	AF104900	Arabidopsis
Lipoxygenase (LOX1)	LO4637	Arabidopsis
Chalcone syntase (CHS)	M20308	Arabidopsis
Up in A1/B		
Fiber annexin		
Laccase		
Ag13 protein precursor		
Chloroplastic CuZn-superoxide dismutase	AJ278668	Populus
Ferulate-5-hydroxylase precursor		
Cysteine protease (CYSP)	AY039606	Arabidopsis
Caffeic acid 3-O-methyltransferase		
Up in A1/B		
Wound induced MAP kinase (WIPK)	D61377	*N. tabacum*
Mitochondrial phosphate translocator	Y08499	*B. pendula*
ABA induced antifreeze protein (KIN1)	AY062849	Arabidopsis

are likely to be involved in the desired traits and reveal expression differences between the parent trees and to correlate these patterns with growth properties in the progeny. For this, selected ESTs were printed on nylon membranes, which were hybridized with radiolabelled probes derived from mRNAs isolated from parent trees. However, due to the problems in reproducibility of the obtained results resulting from low signal intensity that would have hampered identification of regulatory genes in particular, we opted for another strategy and shifted the focus of gene identification to large scale gene expression profiling. This analysis is based on our large scale EST sequencing project that has now been completed. The project has delivered us a collection of birch genes (unigene set) that contains a representative set of genes active during different growth and stress related conditions. From totally 12 different libraries, representing genes expressed in various wood forming, leaf and flower tissues, in trees exposed to different stresses, and trees grown under different photoperiods, we have acquired 73 830 ESTs which, after clustering, provided us ESTs corresponding to 21 278 birch genes. Out of this unigene set, 8811 represent clusters and 12 467 are depicted as singletons. All the unigenes have been annotated and interestingly, 39% of them were assigned in the "function unknown" category. The sequence information from the unigene set has been utilized to design 12 000 gene specific synthetic oligonucleotides for generation of microarrays. All the 50 nucleotide long oligonucleotides are currently being synthesized by MGW and will, during spring 2007, be printed on glass slides for expression profiling.

Work to be done during 2007. Expression profiling using these microarrays will be optimized by comparing the parental trees, from which the RNAs for these studies has already been collected. Subsequently the analysis will be expanded to include RNAs isolated from the progeny of the previously performed crosses. We expect to have the microarray work finished during summer 2007 and data analyzed by the end of the year. The progeny of the second cross (from 2003) will be also ready to analyze during spring 2007 and will be included to verify the data from the initial progeny. This work will define the regulons whose expression correlate with the desired growth characteristics and pinpoint the potential regulatory genes controlling this expression. The most promising regulatory genes identified will be also analyzed for potential sequence and/or expression polymorphism. Potential gene markers showing polymorphism will be analyzed in the progenies by applying bulk segregant analysis. Co-segregation analysis of the polymorphisms and the growth and wood property traits will be done with the 35 clonally propagated as well as in the additional full sib family members.

1.3 Conclusions

The project will provide candidate gene markers for growth and wood property traits.

1.4a Capabilities generated by the project

1.4b Utilisation of results

Once candidate markers have been verified they can be directly utilized for progeny screening in tree breeding programs.

1.5 Publications and communication

a) Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

Puhakainen T, Li C, Boije-Malm M, Kangasjärvi J, Heino P, Palva ET (2004). Short-day potentiation of low temperature-induced gene expression of a C-repeat-binding factor-controlled gene during cold acclimation in silver birch. Plant Physiol. 136: 4299-4307.

Li, CY., Welling, A., Puhakainen, T., Viherä-Aarnio, A., Ernstsen, A., Junttila, O., Heino, P. & Palva, E.T. (2005) Differential responses of silver birch (*Betula pendula* Roth) ecotypes to short day photoperiod and low temperature. Tree Physiol. *25*, 1563-1569.

Welling, A. & Palva, E.T. (2006) Control of cold acclimation in trees. Phys Plantarum 127, 167-181.

Martz, F., Kiviniemi, S., Palva, E.T. & Sutinen, M.-L. (2006) Contribution Of Omega-3 Fatty Acid Desaturase And 3-Ketoacyl-ACP Synthase II (KASII) Genes In The Modulation Of Glycerolipid Fatty Acid Composition During Cold Acclimation In Birch Leaves. J. Exp. Bot. *57*, 897-909.

Kankainen, M., Brader, G., Törönen P., Palva, E.T. & Holm, L. (2006) Identifying functional gene sets from hierarchically clustered expression data: map of abiotic stress stimulated genes in *Arabidopsis thaliana*. Nucl Acids Res (doi: 10.1093/nar/gkl694).

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Aalto, M.K. & Palva, E.T. (2006) Control of growth and cold acclimation in silver birch. *in* Chen, T.H.H., Uemura, M. & Fujikawa, S. (eds.) Cold hardiness in plants. Molecular genetics, cell biology and physiology. CABI Publishing, Oxfordshire, UK. pp. 153-166.

Aalto, M.K., Heino, P. & Palva, E.T. (2006) Improving low temperature tolerance in plants. *in* Varshney, R. (ed.). Model plants, Crop improvement. CRC Press, Boca Raton, USA. pp. 247-290.

3. Articles in Finnish and Swedish journals with referee practice

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice

5. Scientific monographs

Ojala, K. (2007) Master's thesis: University of Helsinki.

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

b) Other dissemination

Such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results.

1.6 National and international cooperation

The presented research is performed in close collaboration between the Department of Biological and Environmental Sciences at the Faculty of Biosciences, University of Helsinki (Partner 1) and the Forest Research Institute of Sweden (Skogforsk, Ekebo) (Partner 2). At Department of Biological and Environmental Sciences Prof. Tapio Palva (research leader) and Docent Pekka Heino and at Skogforsk, Ekebo, Dr. Bo Karlsson and PhLic Maria Boije-Malm are in charge of the project, respectively. Additionally, collaboration has been started with PhD Tommy Mörling at the Wood fiber laboratory at the Swedish University of Agricultural Sciences in Umeå, Sweden. The EST collection has been generated in collaboration with other groups that are part of the Center of excellence in plant signal research. The expression profiling and data analysis is done in collaboration with the groups of Dr Petri Auvinen and Prof Liisa Holm at the Viikki Biocenter in Helsinki.

2 Wood and Wind (WAW)

FINAL REPORT

Name of the research project	**Wood and Wind**
Coordinator of the project	Björn Sundberg

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Wood and Wind, Sub-project 1**
Project period	1.1.2003–31.12.2006
Organization in charge of research	SLU
Sub-project leader	Prof. Björn Sundberg
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Umeå Plant Science Centre Department of Forest Genetics and Plant Physiology, 901 83 Umeå Tel. +46-90-7868382 Bjorn Sundberg@genfys.slu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	295 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	123 000
Other public funding	
Formas (National programme)	172 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Jonathan Love, Ph.D. student	M	Umeå Plant Science Center		Formas
Björn Sundberg, Prof.	M	Umeå Plant Science Center		Swedish Research Council

Total person-months of work conducted by the research team 73
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	M.Sc.	M	Jonathan Love	SLU, Swedish University of Agricultural Sciences	Björn Sundberg
2007	D.Sc.	M	Simon Björklund	SLU, Swedish University of Agricultural Sciences	Björn Sundberg

Name of the sub-project 2	Wood and Wind, Sub-project 2
Project period	1.1.2003–31.12.2005
Organization in charge of research	University of Helsinki
Sub-project leader	Prof. Jaakko Kangasjärvi
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Department of Biological and Environmental Sciences Plant Biology POB 65 (Viikinkaari 1) University of Helsinki FI-00014 Helsinki Tel: +358 9 191 59444 Fax: +358 9 191 59444 jaakko.kangasjarvi@helsinki.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	191 790 (+additional for FuncWood)
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland (1.1. 2003–31.12. 2005)	191 790
Other public funding Tekes-consortium FuncWood (1.12. 2005–31.12. 2007; reported separately) that continues the work initiated in WAW	340 030

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Jorma Vahala, Ph.D., scientist	M	University of Helsinki	WAW (2003-05); FuncWood (2006-07)	
Jaakko Kangasjärvi, Ph.D., Professor	M	University of Helsinki		

Total person-months of work conducted by the research team 36

person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 3	Wood and Wind, Sub-project 3
Project period	1.1.2003–31.12.2005
Organization in charge of research	Umeå University
Sub-project leader	Dr. Hannele Tuominen
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Department of Plant Physiology Umeå University 90187 Umeå Tel. +46 907869693 hannele.tuominen@plantphys.umu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	152 030
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	123 000
Other public funding	
Formas; project 230-2002-1674; salary for the main researcher	16 130
SSF; Graduate school in Genomics and Bioinformatics; salary for the Ph.D. student in the project	12 900

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Jane Geisler-Lee, Ph.D., post doc	F	Dept of Plant Physiol. Umeå University, Sweden	Canada	Formas
Edouard Pesquet, Ph.D., post doc	M	Dept of Plant Physiol. Umeå University, Sweden	France	Formas
Charleen Moreau, MSc, Ph.D. student	F	Dept of Plant Physiol. Umeå University, Sweden		SSF

Total person-months of work conducted by the research team 49
person-month = full-time work for at least 36 h/week, paid holidays included

Abstract

Ethylene is a gaseous two-carbon plant hormone that is an important signalling substance in plants, mediating a range of environmental stimuli into growth responses. We have demonstrated that ethylene is an important component of the signalling system inducing eccentric growth in response to wind and leaning of forest trees. We have further demonstrated that ethylene has a capacity to modify important traits such as wood production, fiber diameter and cell wall chemistry. We have demonstrated that this effect is mediated through the ethylene receptor; hence through a family of transcription factors denoted ethylene response factors (ERFs). We have mined the poplar genome to identify all poplar ERFs and used extensive PCR screening to find the key ERFs involved in wood modification. These genes are currently evaluated for commercial use through a transgenic approach.

Tiivistelmä

Etyleeni on kaasumainen, kaksihiilinen kasvihormoni, jonka tehtävä tärkeänä viestinvälittäjänä vaihtelee kasveissa ympäristövasteista kasvutekijöihin. Olemme osoittaneet etyleenin olevan merkittävä viestintäjärjestelmän osatekijä, joka käynnistää metsäpuissa epäkeskistä kasvua tuulen ja kallistuksen vaikutuksesta. Olemme edelleen osoittaneet, että etyleeni kykenee säätelemään sellaisia merkittäviä ominaisuuksia kuten esimerkiksi puun tuottoa, puukuitujen halkaisijaa ja soluseinien kemiallista koostumusta. Olemme näyttäneet toteen, että tämä vaikutus välittyy etyleeniä vastaanottavien reseptorien kautta, ja näin ollen edelleen transkriptio-geeniperheen kautta, joita kutsutaan etyleenivastetekijöiksi (engl. ethylene response factros, ERFs). Olemme etsineet poppelin genomista jokaisen ERF-geenin ja käyttäneet kattavaa PCR-seulontaa, jotta löytäisimme puun muodostusta säätelevät ERF-geenit. Hyväksikäyttäen geeninsiirtotekniikkaa, kyseiset geenit ovat parhaillaan tutkimuksen kohteena kaupallista sovellusta varten.

Sammanfattning

Etylen är ett växthormon och en viktig signalmolekyl i växter, framförallt är etylen viktigt för signalering av yttre miljö och stressfaktorer. Vi har visat att etylen är en signalkomponent vid induktion av excentrisk stamtillväxt hos skogsträd som ett resultat av vind eller mekaniskt orsakad lutning av stammen. Vi har även visat att etylen har en potential att modifiera ett antal andra viktiga vedegenskaper, till exempel fiberdiameter och fiberns kemiska egenskaper. Vi har visat att påverkan av dessa egenskaper signaleras via etylenreceptorn, och således via en familj av transkriptionsfaktorer (sk. etylenresponsfaktorer ERF). Vi har använt bioinformatik för att hitta alla ERFs i poppelgenomet och identifierat alla de ERFs som kan ha en roll i vedmodifiering. Det kommersiella värdet av dessa gener utvärderas för närvarande genom att studera deras effekter i transgena träd.

2.1 Introduction

2.1.1 Background and significance of the research

A major aim in forest management and tree breeding is to maximize the allocation of carbon that is fixed by photosynthesis, to the production of wood and fibers with valuable properties. In biological terms this means that we need to increase the activity of the vascular cambium, the wood forming meristem, and to optimize the process of wood fibre development. To achieve this goal in modern breeding, using molecular markers or a transgene approach, we need to identify the genes and understand the molecular mechanisms that underpin this biology.

Wind sway of tree stems is a major environmental cue in the forest ecosystem, stimulating cambial cell division (hence wood production) at the expense of height growth. Wind is also an important factor for fibre quality since it induces production of reaction wood with large modifications on fibre structure and cell wall chemistry. On the same theme, a static lean of tree stems stimulates cell division, and reaction wood formation, localized to the upper side of the stem. Both in wind sway and leaning it is believed that the wood forming tissues responds to signals generated by stem movement and the associated shift in gravity. The signalling system mediating this response is unknown. However, exogenous application of the plant hormone ethylene, a simple two carbon gas molecule, stimu-

late cambial growth, and ethylene biosyntheses is elevated within the upper side of bent tree stems due to an asymmetric inductor of the final enzyme responsible for ethylene biosyntheses, 1-aminocyclopropane-1-carboxylate (ACC) oxidase. Ethylene has therefore been hypothesized to have a role in mediating gravitational sensing into at least some aspect of reaction wood characteristics.

The approach taken in this project is to understand and dissect ethylene signalling resulting in increased cambial growth and modified fibers. This will provide novel knowledge in wood development and molecular tools to be used in tomorrow's tree breeding. Our research is based on the solid foundation of ethylene perception/signalling research in the model plant *Arabidopsis*, the genome programs on poplar (*Populus tremula* x P. *tremuloides*; hybrid aspen) and birch (*Betula pendula*) initiated in Sweden and Finland, platforms for fiber characterisation developed at UPSC and the expertise on plant hormones and wood biology among the partners.

2.1.2 Objectives

The objectives are to

- understand the function of endogenous ethylene in wood formation.
- identify the molecular components in the ethylene-dependent signalling pathway that is responsible in stimulating cambial growth and fiber modification.

2.2 Results and discussion

2.2.1 Facilitating tools established to study the ethylene response

Database mining and primer production for expression analysis

We mined altogether 217 poplar genes involved in ethylene biosynthesis and action from the recently sequenced genome of *Populus trichocarpa*. The number of especially ethylene-response-factor (ERF) proteins that directly regulate ethylene-targeted genes was found to be significantly higher in poplar than in *Arabidopsis* and rice genomes. This may directly reflect the need for more complex involvement of ethylene pathway and ethylene-dependent gene induction for perennial growth habit and xylogenesis. To explore the involvement of ethylene on xylogensis, we designed gene specific primers for poplar ACC synthase and oxidase gene families and for the whole poplar ERF family according to *P. trichocarpa* gene models and information from the Populus EST database (http://www.populus.db.umu.se/). For the correct product size and equal amplification efficiency between *P. trichocarpa* and hybrid aspen, the primer pairs for each gene were tested with PCR using genomic DNA from both species before running real-time quantitative PCR (qPCR).

Construction of ethylene insensitive poplar trees

To investigate the role of ethylene in wood formation, and to provide plants in which downstream ethylene signalling components can be identified, we produced ethylene insensitive trees by transgenic expression of the mutated ethylene receptor *AtETR1-1* from *Arabidopsis.* This mutated receptor will confer ethylene insensitivity due to its dominant negative nature. *AtETR1-1* was expressed with the constitutive CaMV35S promoter, and *hitherto* unpublished promoters more specific to different xylem and phloem stem tissues denoted LMX5 and LMP1. In total 15 35S lines, 9 LMX5 lines and 12 LMP1 lines were regenerated and screened for ethylene insensitivity.

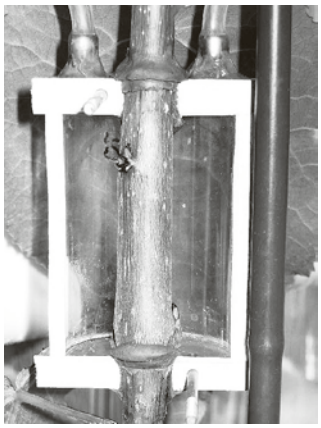
In vitro tree culture systems for rapid screening of ethylene responses

We developed a novel *in vitro* tree culture system, which allowed for the exogenous application of ACC (the metabolic precursor to ethylene) to solidified MS media at a standard height. A reference stem internode in which all wood was formed under the influence of ACC was used to quantify xylem growth and wood fiber morphology. A suitable ACC dosage giving optimal effects in wild type plants was established to be 100µM of ACC. We also established a method to apply 1-methylcyclopropene (MCP) (a gaseous compound known as the most specific and efficient inhibitor of ethylene responses) to *in vitro* cultured trees. Trees exposed to MCP during the period of ACC treatment would

therefore serve as a positive control for the ethylene insensitive trees. All transgenic lines were screened in the *in vitro* system for their insensitivity to ACC regarding ethylene inhibition of height growth and vessel diameter. We selected two lines representing each promoter construct that showed optimal ethylene insensitivity as judged from these traits; 35S 1E and 3A, LMX5 1 and 6, and LMP1 5 and 9. These lines were used in further work.

A cyvette system for experimentation on large trees

To avoid secondary effect that may be caused by ACC treatment to whole plants, a system for specific and continuous ethylene exposure to stem tissues was established by fixing sealed flow through chambers to stems of ca 2 m tall greenhouse grown tree. Treatment with a gas mixture of synthetic air, ethylene (2 ppm) and carbon dioxide (350 ppm) increased both xylem and phloem growth, and reduced vessel diameter and frequency. This experimental system was also required to produce large amount of ethylene treated material for subsequent analysis and tissue localisation of ethylene response factors.



A cell culture system for easy pharmacological experimentation

Studies into the hormonal control of wood formation are many times hindered by technical difficulties related to inaccessibility of the cambial and xylem tissues giving rise to the wood. Therefore we decided to establish a cell culture system as a novel tool to access the function of ethylene in xylogenesis. In this *in vitro* differentiation system, tracheary elements (TEs, ontologically comparable to the unit-forming xylem vessels) are derived from mesophyll cells of *Zinnia elegans* leaves that upon addition of the plant hormones auxin and cytokinin transdifferentiate into TEs. The *Zinnia in vitro* differentiation system gives amenability to application studies in a system with semi-synchronous differentiation of the TEs.

FT-IR microspectroscopy and mechanical testing

In order to investigate the effects of ethylene on chemical composition of cell walls *in situ*, cutting edge FT-IR microspectroscopy was performed on 20 mm thick sections from the wood. A focal plan array detector has been used, recording simultaneously 64 x 64 spectra (pixels) of an image, providing a maximum spatial resolution of about 5 mm. This opens up the possibility to extract chemical information from cell walls of specific cell types, i.e. fibers, vessels or rays. Spectra were sub-

jected to multivariate data analysis to identify IR bands that could be used for differentiating between lines and treatments and to determine the changes in chemical composition of fibre cell walls. Mechanical testing was done in collaboration with Prof. Lars Berglund's group at the Royal Institute of Technology (KTH), Stockholm. Using stems grown in our *in vitro* system, a mini materials tester model Minimat 2000 was then used to apply load, and record displacement and reaction force.

2.2.3 Ethylene's potential to modify wood and fibers

Cambial growth and wood development

ACC induced inhibition of height growth and stimulation of wood formation in wild type trees. The ethylene insensitive lines reduced or completely nullified both responses. The diameter of vessel elements was reduced by ACC treatment in wild type trees. This effect was not evident in any of the ethylene insensitive lines. A similar, but less obvious, effect was observed for fiber diameter. The effects of ACC treatment on the length of fiber and vessel elements was not consistent and did not allow any firm conclusions about a role for ethylene in affecting this trait.. The MCP treatments nullified the effects of ACC treatments for all traits observed, confirming that the ACC induced phenotypes were a result of ethylene action. The stimulation of xylem and phloem growth, and reduced vessel diameter and frequency observed after treatment of stem segments in the cyvette system were reduced in the ethylene insensitive line 1E. Taken together, our results demonstrate that applied ethylene inhibit height growth, stimulate cambial cell division and wood production and inhibit radial expansion of fibers and vessel elements, and that these responses are mediated through the ETR receptor.

Fiber chemistry and mechanics

In addition to ethylene's effects on radial growth, wood development and chemistry, ethylene may also influence the chemical and mechanical properties of wood. Initial Ft-IR analysis of fiber walls indicate that ethylene modify glycosidic links in the cell wall, as well as the amount of cellulose. The mechanical properties of wood are determined by a number of factors including density, cell wall chemistry and structure including microfibril angle. As such, mechanical testing data is an empirical measure encompassing these factors. Preliminary testing of tensile stress (σ) and Young's modulus (Y) suggests ACC treated stems are both stiffer and stronger. This work on wood chemistry and mechanics is currently being repeated with an optimized method.

Xylem cell differentiation in the Zinnia system

Ethylene (as ethephon) and its precursor ACC as well as inhibitors of ethylene biosynthesis and signalling were added to the cell culture in order to study the role of ethylene in xylem differentiation. The effect of the pharmacological treatments was followed by measuring ACC and ethylene levels with gas chromatography and gas chromatography mass spectrometry, by analysing TE differentiation under epifluorescent microscope, TE cell morphology with confocal microscopy, and gene expression by RT-PCR. The cell culture system revealed details about ethylene biosynthesis that have not been reported earlier. During TE differentiation, ACC was exported outside of the differentiating cells and the ACC oxidase activity was almost entirely restricted to the apoplastic space, suggesting that biosynthesis of ethylene takes place in the apoplast i.e. in the cell wall. Our results showed also that ethylene has a dual role during TE differentiation, influencing both the efficiency of TE differentiation and the proper lignification and cell death of TEs. The requirement of functional ethylene signalling in maturation (lignification and cell death) is contradictory to what is seen in intact plants, where defects in ethylene signalling do not seem to cause major effects in these processes. We propose that the *in vitro* system in its simplicity allows performance of experiments where you can precisely study effect of one compound on xylogenesis. In intact plants, complete blocking of ethylene signalling might be complemented by mechanisms taking place in surrounding cell types and tissues.

2.2.4 The function of endogenous ethylene in the tension wood response

The stimulation of cambial cell division by applied ethylene suggests that endogenous ethylene induced by gravitational stress mediates the increase of cambial growth in the tension wood response, resulting in eccentric stems. This hypothesis was tested by leaning control-, ethylene- insensitive- and MCP-treated lines in the *in vitro* system in which tension wood with all its typical characteristica was induced. Measuring xylem area at the TW and OW side in wild type trees induced a TW/OW ratio of about 2. All ethylene-insensitive- and MCP-treated trees had a significant decreased TW/OW ratio. This demonstrates a role for endogenous ethylene in stimulating cambial cell division in gravitational stress.

2.2.5 Identification of ethylene response genes mediating wood modification using real time qPCR and microarrays

The increase in transcript abundance of a certain gene is a common response to environmental cues. We are interested, which ERF genes are induced in active cambium, and thus involved in the stimulation of wood formation. We screened with highly sensitive real-time qPCR the whole poplar ERF gene family using gene specific primers and whole-stem material, which was treated with 2 ppm of ethylene for two weeks. When compared to controls, we identified 53 genes at least with 2-fold up-regulation, 43 genes at least with 2-fold down-regulation, and 78 genes with no induction. Approximately 30% of poplar ERF genes are present in Umeå EST collections and thus in microarray gene chips. Microarray experiment was conducted with *in vitro* -grown hybrid aspen material treated with ACC for 2, 5 and 10 hours. From this experiment, we identified seven ERF genes with a clear up-regulation. Two of these ERF genes showed similar result in response to both ethylene (with big trees) and ACC treatments (with *in vitro* trees). This indicates that some of the ERF genes might have considerably transient expression status, which we must further investigate. Additionally, because of the surprisingly high number of up-regulated ERF genes by ethylene treatment, we need to explore the ERF family in more detail. Especially to gain and confirm more knowledge about the cambium-specific ERF genes, a separately isolated cambial material treated with ethylene is currently under investigation.

2.3 Conclusions of major significance

- ACCOx has a role in controlling ethylene biosynthesis in vegetative shoots and wood development. This is a novel knowledge.
- Ethylene is an endogenous signal in gravitational sensing in woody stems and stimulate cell division in meristematic growth. This is a novel knowledge.
- We have identified all genes involved in ethylene biosynthesis and signaling in poplar.
- We have identified candidate ethylene signaling genes (ERFs) responsible for wood modification.

2.4a Capabilities generated by the project

M.Sc. Thesis. Jonathan Love. May 2007. Wood formation in response to leaning stress: ethylene's role.

Dr.Sc. Thesis. Sept. 2007. Simon Björklund. Hormonal control of wood devlopment (Preliminary title)

Dr.Sc. Thesis. Oct. 2007. Charleen Moreau. Programmed death of xylem elements (Preliminary title)

A database of ERF transcriptionfactors to be used for wood modification.

2.4b Utilisation of results

We have demonstrated that ethylene applied to developing wood can modify important traits such as wood production, fiber diameter and cell wall chemistry. We have demonstrated that this effect is mediated through the ethylene receptor, hence through ethylene response factors (ERFs). We have identified the key ERFs involved in wood formation. These genes will be evaluated for their capacities to improve tree growth and wood properties in a transgenic approach. Any successful application will be patented and commercialized through SweeTreeTechnologies (www.sweetree.com).

2.5 Publications and communication

a) Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

*Andersson-Gunnerås S, Hellgren J, Björklund S, Regan S, Moritz M, Sundberg B. 2003 Asymmetric expression of a poplar ACC oxidase controls ethylene production during gravitational induction of tension wood. *Plant Journal* 34: 339-349.

Gray-Mitsumune M, Abe H, Sundberg B, Mellerowicz EJ. 2004. Liquid-phase fluorescence in situ RT-PCR for high reolution and high throughput gene expression analysis in woody stem tissues. Plant Biology 6: 47-54.

Hellgren JM, Olofsson K, Sundberg B. Patterns of auxin distribution during gravtational induction of reaction wood in poplar. 2004. *Plant Physiology* 135: 212-220.

Gray-Mitsumune M, Mellerowicz, E, Abe H, Schrader J, Winzél A, Sterky F, Blomqvis K, McQueen-Mason S, Teeri T, Sundberg B. 2004. Secondary growth-specific expansin genes belong to the Subgroup A α-expansin gene family and exhibit conservation among different wood-forming species. *Plant Physiology* 135: 1552-1564.

Djerbi S, Aspeborg H, Nilsson P, Sundberg B, Mellerowicz E, Blomqvist K Teeri T. 2004. Identification and expression analysis of genes encoding putative cellulose synthases (CesA) in the hybrid aspen, *Populus tremula* (L.) x *P. tremuloides* (Michx.) . *Cellulose* 11 (3-4): 301-312

*Sterky, Bhalerao, Unneberg, Segerman, Nilsson, Brunner, Charbnonel-Campaa, Jonsson, Tandre, Strauss, Sundberg, Gustafsson, Uhlen, Bhalerao, Nilsson, Karlsson, Lundeberg, Jansson. 2004. A Populus EST resource for plant functional genomics. *Proc. Natl. Acad. Sci. USA* 101 (38): 13951-13956

Andersson, Keskitalo, Bhalerao, Sjödin, Sterky, Wissel, Tandre, Aspeborg, Moyle, Ohmiya, Bhalerao, Brunner, Gustafsson, Karlsson, Lundeberg, Nilsson, Sandberg, Strauss, Sundberg, Uhlen, Jansson, Nilsson1. 2004. A transcriptional timetable of autumn senescence. *Genome Biology* 5 (4): Art. No. R24

Aspeborg H, Schrader J, Coutinho PM, Stam M, Kallas Å, Djerbi S, Nilsson P, Denman S, Amini B, Sterky F, Master E, Sandberg G, Mellerowicz E, Sundberg B, Henrissat B, Teeri TT. 2005. Carbohydrate-active enzymes involved in the secondary cell wall biogenesis in hybrid aspen. *Plant Physiology* 137: 983-997.

Ruonala R, Rinne P, Baghour M, Moritz T, Tuominen H, Kangasjärvi J. 2006. Transition in the functioning of the shoot apical meristem in birch (Betula pendula) involve ethylene. Plant Journal. 46, 628-640.

*Moreau C, Aksenov N, Garcia M, Segerman B, Funk C, Nilsson P, Jansson S, Tuominen H 2005. A genomic approach to investigate developmental cell death in woody tissues of Populus trees. Genome Biology 6/4/R34.

Isrealsson M, Sundberg B, Moritz T. (2006)Tissue-specific localisation of GAs in wood-forming tissues in aspen. *Plant J* 44 (3): 494-504

*G. A. Tuskan, S. DiFazio, S. Jansson, J. Bohlmann, I. Grigoriev, U. Hellsten, N. Putnam, S. Ralph, S. Rombauts, A. Salamov, J. Schein, L. Sterck, A. Aerts, R. R. Bhalerao, R. P. Bhalerao, D. Blaudez, W. Boerjan, A. Brun, A. Brunner, V. Busov, M. Campbell, J. Carlson, M. Chalot, J. Chapman, G.-L. Chen, D. Cooper, P. M. Coutinho, J. Couturier, S. Covert, Q. Cronk, R. Cunningham, J. Davis, S. Degroeve, A. Déjardin, C. dePamphilis, J. Detter, B. Dirks, I. Dubchak, S. Duplessis, J. Ehlting, B. Ellis, K. Gendler, D. Goodstein, M. Gribskov, J. Grimwood, A. Groover, L. Gunter, B. Hamberger, B. Heinze, Y. Helariutta, B. Henrissat, D. Holligan, R. Holt, W. Huang, N. Islam-Faridi, S. Jones, M. Jones-Rhoades, R. Jorgensen, C. Joshi, J. Kangasjärvi, J. Karlsson, C. Kelleher, R. Kirkpatrick, M. Kirst, A. Kohler, U. Kalluri, F. Larimer, J. Leebens-Mack, J.-C. Leplé, P. Locascio, Y. Lou, S. Lucas, F. Martin, B. Montanini, C. Napoli, D. R. Nelson, C. Nelson, K. Nieminen, O. Nilsson, V. Pereda, G. Peter, R. Philippe, G. Pilate, A. Poliakov, J. Razumovskaya, P. Richardson, C. Rinaldi, K. Ritland, P. Rouzé, D. Ryaboy, J. Schmutz, J. Schrader, B. Segerman, H. Shin, A. Siddiqui, F. Sterky, A. Terry, C.-J. Tsai, E. Uberbacher, P. Unneberg, J. Vahala, K. Wall, S. Wessler, G. Yang, T. Yin, C. Douglas, M. Marra, G. Sandberg, Y. Van de Peer, D. Rokhsar (2006) The Genome of Black Cottonwood, *Populus trichocarpa* (Torr. & Gray). Science 313:1596- 1604.

*Andersson Gunnerås S., Mellerowicz E. J., Ohmiya Y., Nilsson P., Henrissat B., Love J., Moritz T. and Sundberg B. (2006) Making cellulose enriched tension wood in poplar: global analysis of transcripts and metabolites identifies biochemical and developmental regulators in secondary wall biosynthesis *Plant J* 45 (2): 144-165).

Jane Geisler-Lee, Matt Geisler, Pedro M. Coutinho, Bo Segerman, Nobuyuki Nishikubo, Junko Takahashi, Henrik Aspeborg, Soraya Djerbi, Emma Master, Sara Andersson-Gunnerås, Björn Sundberg, Stanislaw Karpinski, Tuula T. Teeri, Leszek A. Kleczkowski, Bernard Henrissat, and Ewa J. Mellerowicz. 2006. Poplar Carbohydrate-Active Enzymes (CAZymes). Gene identification and expression analyses Plant Physiol. 140: 940-962.

To be submitted:

Moreau C, Pesquet E, Muniz L, Sjödin A, Jansson S, Samuels L, Tuominen H. Development and programmed death of xylem fibers in Populus wood. Manuscript to be submitted.

Pesquet E, Moreau C, Tuominen H. Ethylene homeostasis and function during Zinnia elegans in vitro xylogenesis. Ms to be submitted.

Björklund S, Love J, Vahala J, Kangasjärvi J, Sundberg B. Ethylene stimulate cambial cell division in *Populus*. Ms to be submitted.

Vahala J, Björklund S, Love J, Kangasjärvi J, Sundberg B. Ethylene response factors mediating wood modification in *Populus*. Ms to be submitted.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice,

Pesquet E. and Tuominen H. 2007. Unraveling ethylene biosynthesis and role during tracheary element formation in Zinnia elegans. Proceedings in the 7th International Symposium on the Plant Hormone ETHYLENE, Pisa (Italy), June 2006.

3. Articles in Finnish and Swedish journals with referee practice

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice

5. Scientific monographs

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

b) Other dissemination

Meetings (Oral presentations)

Kangasjärvi. IUFRO tree biotechnology meeting in Umeå 9.-12.5.2003.

Sundberg. ”BioFuelRegion” med fokus på energiförsörjning och transportutveckling. Lycksele, Sweden, 2003. (invited)

Sundberg. Genetics and Improvement of Plant LaRochelle, France. 2003. INRA National meeting. (Plenary Lecture.)

Kangasjärvi. Eurosilva - COST meeting Krakow, 26.-27.7.2004.

Kangasjärvi. 12th New Phytologist symposium (Poplar genome release symposium), Tennesee USA 11.-15.9.2004.

Sundberg. Finnish-Swedish Wood Material Science Research Programme, Opening Seminar 6.4. 2004, Marina Congress Center, Helsinki. (invited)

Sundberg. Poplar symposiym May 2004. Göttingen, Germany. (Key note)

Sundberg. BIOTEC04. National congress of biotechnology, Oviedo ,Spain 2004. (Plenary lecture).

Sundberg. III SPPS-Ph.D. congress, 5-8th of August 2004, Sem Gjestegård, Norway, (invited).

Sundberg. Swedish Forest Biotechnology Conference. Stockholm Nov 2004. (invited)

J. Vahala, J. Love, S. Björklund, H. Tuominen, B. Sundberg, J. Kangasjärvi. COST-E28 WG1 Meeting, October 13-14 2005, Orleans, France.

Pesquet E., Moreau C., Muniz L. and Tuominen H. 1st UPRA meeting, Versailles (France), October 2005.

Kangasjärvi. Forest biotechnology workshop Helsinki 4.2.2005.

Kangasjärvi. Presentation of plant/tree biotechnology to Science journalists 1.4. 2005 (Helsinki)

Kangasjärvi. Finnish Forest Research Institute seminar 4.4.2005.

Kangasjärvi. Norwegian Functional Genomics workshop in Oslo, 6.9.2005.

Kangasjärvi. COST E28 meeting in Paris 19.-22.5. 2005.

Sundberg. Nordic Biofibre Conference, Stockholm 2005. (invited)

Sundberg. WURC International Seminar, Uppsala 2005. (invited)

Sundberg. Svenska kemisamfundet, Ny kemi för skogsindustrin, Stockholm 2005. (invited)

Sundberg. IUFRO Tree Biotechnology, Pretoria, RSA, 2005,

Sundberg. International Symposium on Wood Science and Technologies, Yokohama, Japan, 2005. (invited)

6th Pacific Regional Wood Anatomy Conference (PRWAC) Kyoto 2005. (invited)

Kangasjärvi. Forest biotechnology workshop 1.2.2006 (to forestry R&D professionals).

Sundberg. CSPP President's Symposium - Tree Physiology and Genomics. Joint Annual Meeting of the American Society of Plant Biologists and the Canadian Society of Plant Physiologists. Boston 2006. (plenary lecture)

Sundberg. Population Genetics and Genomics of Forest Trees: from Gene Function to Evolutionary Dynamics and Conservation. A joint conference of IUFRO Working Groups 2.04.01 (Population, ecological and conservation genetics) and 2.04.10 (Genomics), and Cost Action E-28 (Genosilva: European Forest Genomics Network). Madrid Spain 2006. (invited).

Sundberg. The Peter Wallenberg fundation symposium. Northern Woods Characteristic - Threats and Opportunities Stockholm 2007 (invited)

Media, industry and society

Kangasjärvi. Two short radio interviews about the Poplar genome sequencing.

Kangasjärvi. 45 minute radio program about tree genomics and tree biotechnology (after Poplar genome sequencing).

Kangasjärvi. Several newspaper stories about the Poplar genome sequencing.

Sundberg. Numerous interactions (ca 5-10 per year) with representatives from industry, media and society.

Articles popular science

Janson S, Nilsson O, Sundberg B. 2003 Genmodifierade träd i morgondagens skog. Genklippet? maten, miljön och den nya biologin. Formas Stockholm. pp. 81-92.

Sundberg B. 2006 Mer ved i träden. MiljöForskning 3:0614-16.

2.6 National and international cooperation

Sundberg main research network/cooperation is (in addition to this program) within several national and international networks: director and PI in FuncFiber (Formas funded excellence center in wood science, www.funcfiber.se), PI in Biomime (SSF funded excellence center in wood material science, www.biomime.org), PI in WURC (Vinnova funded excellence center in wood ultrastructure, www-wurc.slu.se), PI in EU funded FP6 programs EDEN (2002-2005) and CASPIC (2006-2009) with research focused on cell wall biosynthesis. Sundberg has also been the chair of IUFRO program TreeBiotechnology (2003-2005), and is a member of the European Scientific Advisory Board of Forest Technology Platform (www.forestplatform.org), and the scientific advisory board of SweeTreeTechnologies (www.swetree.com).

Kangasjärvi research group is a part of the Finnish Center of Excellence in plant signal research (2000-2011; before 2006 under the name Plant Molecular Biology and Forest Biotechnology Research Unit) that consists of eight research groups. He has had both within this Center of Excellence and outside it an extensive network of collaborations both nationally and internationally with both Universities and research institutes. He has been involved in two EU-funded consortia (TomStress and Establish), coordinated a Nordic Arabidopsis Network (2000-2005), has been the coordinator of the Finnish Project Program in Plant Genomics (2003-2006) and a member in the University of Helsinki Scientific Board (2007-09).

Tuominen has within the project had collaboration and also visited the two laboratories of professor H. Fukuda in Tokyo University and Dr. T. Demura in RIKEN/Yokohama to learn the /Zinnia elegans/ cell culture system. Outside of the project, Tuominen has active collaboration with professor M. Blazguez (Valencia Polytechnical Univ., Spain), Dr. Lacey Samuels (Univ. British Columbia, Vancouver, Canada) and Dr. Peter Bozhkov (SLU, Sweden). Tuominen is also PI in the collaborative project FuncFiber funded by Formas.

3 Value-chain analysis for forest management, timber purchasing and timber sale decisions (VACHA)

FINAL REPORT	
Name of the research project	**Value-chain analysis for forest management, timber purchasing and timber sale decisions**
Coordinator of the project	Professor Tuula Nuutinen

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**A forestry model for the national level analysis of forest management strategies in Finland**
Project period	1.1.2003–31.12.2005
Organization in charge of research	Finnish Forest Research Institute, Joensuu Research Centre
Sub-project leader	Professor Tuula Nuutinen
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Finnish Forest Research Institute, Joensuu Research Unit, Yliopistokatu 6, FI-80101 Joensuu, Tel. +358 10 211 3043, Fax +358 10 211 3113 tuula.nuutinen@metla.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	555 893
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	236 080
Ministry of Agriculture and Forestry	319 813

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Matti Kärkkäinen, Dr. (For.), prof.	M	University of Joensuu and the Centre of Expertise for Wood Technology and Forestry	Academy of Finland; University of Joensuu and the Centre of Expertise for Wood Technology and Forestry
Ulla Mattila, Dr. (For.), post doc	F	Finnish Forest Research Institute	Academy of Finland
Florian Berger, M.Sc, system analyst	M	Finnish Forest Research Institute	Academy of Finland
Hannu Hirvelä, M.Sc, research scientist	M	Finnish Forest Research Institute	
Kari Härkönen, M.Sc, research scientist	M	Finnish Forest Research Institute	
Olli Salminen, M.Sc. research scientist	M	Finnish Forest Research Institute	
Total person-months of work conducted by the research team 46 person-month = full-time work for at least 36 h/week, paid holidays included			

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name of the sub-project 2	**Models of wood properties for planning systems**
Project period	19.2.2003–31.10.2006
Organization in charge of research	Skogforsk
Sub-project leader	Dr. Lennart Moberg
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Skogforsk, Uppsala Science Park, SE-751 83 Uppsala, Sweden Tel. +46 18 18 85 00, Fax +46 18 18 86 00, Lennart.Moberg@skogforsk.se
URL of the project	http://www.woodwisdom.fi/en/
FUNDING	
Total sub-project budget in EUR	407 500
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	174 500
Other funding	
Skogforsk	233 000

RESEARCH TEAM

Name, degree, job title [1)]	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Lars Wilhelmsson, Ph.D., Program Leader	M	Skogforsk		Formas/ Skogforsk
Martin Ekstrand, M.Sc., Researcher	M	Skogforsk		Formas/ Skogforsk
Ingemar Eriksson, M.Sc., Researcher	M	Skogforsk		Formas/ Skogforsk

Total person-months of work conducted by the research team 46.5

2) person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name of the sub-project 3	**Timber purchasing and sale decision models in accordance with alternative selection of wood assortments**
Project period	1.4.2003–31.12. 2006
Organization in charge of research	Finnish Forest Research Institute, Joensuu Research Centre
Sub-project leader	Professor Erkki Verkasalo
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Finnish Forest Research Institute, Joensuu Research Unit, Yliopistokatu 6, FI-80101 Joensuu, Tel. +358 10 211 3020 Fax +358 10 211 3001 erkki.verkasalo@metla.fi
URL of the project	http://www.metla.fi/hanke/3357/index-en.htm http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	617 279
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Ministry of Agriculture and Forestry	121 344
Other funding	495 395

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Erkki Verkasalo, Dr (For.) Scientific and administrative leader	M		Finnish Forest Research Institute	
Tapio Wall, M.Sc. (For.), Operational leader and researcher	M		Finnish Forest Research Institute	
Jukka Malinen, Dr (For.), Main researcher	M		Finnish Forest Research Institute	
Harri Kilpeläinen, M.Sc. (For.), Programming	M		Finnish Forest Research Institute	
Total person-months of work conducted by the research team 46 person-month = full-time work for at least 36 h/week, paid holidays included				

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2005	M.Sc.	M	Teppo Piira	University of Joensuu	Harri Kilpeläinen, Tapio Wall, Jukka Malinen, Finnish Forest Research Institute

Name of the sub-project 4	**Influence of environmental factors, forest structure and silvicultural practices on Scots pine, Norway spruce and birch properties**
Project period	1.1.2003–31.12.2005
Organization in charge of research	University of Joensuu, Faculty of Forestry
Sub-project leader	Professor Heli Peltola
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	University of Joensuu, Faculty of Forestry, Yliopistokatu 7, FI-80101 Joensuu Tel. +358 13 251 3639 Fax +358 13 251 4444 heli.peltola@joensuu.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	172 840
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	172 840

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Heli Peltola, Dr (Agr. and For.), leader of sub-project 4	F	University of Joensuu, Faculty of Forestry		University of Joensuu, Faculty of Forestry
Antti Kilpeläinen, M.Sc. / Dr (Agr. and For.), PhD student (1/2003-3/2005) and PostDoc (4/2005-12/2005)	M	University of Joensuu, Faculty of Forestry, Graduate school for Forest Sciences		Graduate school for Forest Sciences/ Academy of Finland
Hannu Väisänen, M.Sc., computer scientist	M	University of Joensuu, Faculty of Forestry		Academy of Finland
Veli-Pekka Ikonen, M.Sc. computer scientist	M	University of Joensuu, Faculty of Forestry		Academy of Finland
Jarmo Pennala, research amanuensis	M	University of Joensuu, Faculty of Forestry		Academy of Finland/ University of Joensuu, Faculty of Forestry

Total person-months of work conducted by the research team: 54 funded by Academy of Finland (in addition, 28 person-months covered by the Graduate school for Forest Sciences and 14 by the University of Joensuu)
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2005	Dr (Agr. and For.)	M	Antti Kilpeläinen, 1977, M.Sc. 2001	University of Joensuu	Dr Heli Peltola and Prof. Seppo Kellomäki, University of Joensuu, Faculty of Forestry

Abstract

A multi-disciplinary Finnish-Swedish research team has developed and tested models on wood and timber properties applicable for planning the utilization of forest resources. In sub-project 1, a forestry model was developed based on the MELA software of the Finnish Forest Research Institute (Metla). A new method was incorporated into the MELA software for calculating total felling potential and reserves. Calculations were made using the national forest inventory (NFI) data to determine how the changes in wood use, forest management strategies, and climate affect the future forest resources and felling potential. The risks and impacts of forest damage such as butt rot and pine twisting rust were modelled for the purpose of analysing forest management strategies. To assist national forestry analysis, global scenarios of wood use and wood paying capabilities in different fields of forest and wood product industries were also explored. In sub-project 2, a family of models was developed by the Forestry Research Institute of Sweden (Skogforsk) as a compatible extension to the Heureka software of the Swedish University of Agricultural Sciences (SLU). These models for wood properties can be used when designing optimal delivery of timber from the forest to the end-users. In sub-project 3, the impacts of alternative assignments of timber assortments, log dimension and quality criteria (i.e. bucking instructions) on the stumpage value of stands were investigated from the points of view of forest owners and buyers. Simultaneously, sensitivity analyses were performed to determine the effects of stand factors and assortment unit prices, as well as product prices, by-product credits, log utilization ratio, and manufacturing costs in wood product industries on the stumpage value. To predict the recoveries of conventional and special timber assortments, and, further, the value of the timber, different assortment recovery models based on field measurement or airborne laser scanning were constructed. For training and teaching purposes, empirical model stands provided with the timber recovery and sales value information were established in two provincial areas. In sub-project 4, the effects of elevated CO_2 and temperature on the growth dynamics of 20–25 years old Scots pine with implications on wood properties were studied. In addition, data measured from Scots pine, Norway spruce and silver birch were used in statistical models where radial growth distribution on stem was modelled in addition to wood properties (wood density, percentage of early wood, fibre length), which in turn were modelled in relation to radial growth and tree ring age. These wood property models were also used in calculations with a physiological growth and yield model to study the impact of silvicultural procedures on tree growth and various wood properties. The wood property models for Scots pine and Norway spruce were also compared with the models developed by sub-project 2. In co-operation with sub-projects 1, 2 and 4 the Swedish models were applied in the MELA analysis on the present and future forest resources and felling potential in Northern Karelia. In co-operation between sub-projects 1 and 3, the model chain in the MELA system and the bucking simulation of stems were compared when estimating timber yields. According to experiences there is potential for multi-national modelling and model sharing between Finland and Sweden if the independent variables are defined generally enough. Incorporated into forest planning software (MELA in Finland and Heureka in Sweden), the models will provide extensive benefits for practical forestry in both countries when planning forest management, wood procurement and timber trade.

Tiivistelmä

Monitieteinen suomalais-ruotsalainen tutkimusryhmä kehitti ja testasi malleja, joiden avulla voidaan kuvata puun käyttöominaisuuksia erilaisissa metsätalouden suunnittelutehtävissä. Osahankkeessa 1 kehitettiin Metsäntutkimuslaitoksen (Metla) MELA-ohjelmistoon perustuvaa metsätalousmallia. MELA-ohjelmistossa otettiin käyttöön uusi menetelmä hakkuumahdon ja -reservin laskemiseksi. Uudistetulla ohjelmistolla ja valtakunnan metsien inventoinnnin (VMI) aineistolla tehtiin laskelmia puun käytön, metsänkäsittelystrategioiden ja ilmaston muutoksen vaikutuksista tuleviin metsävaroihin ja hakkuumahdollisuuksiin. Met-

sänkäsittelystrategioiden analysointia varten mallinnettiin myös metsätuhojen riskiä ja vaikutuksia. Kansallisia metsälaskelmia varten selvitettiin myös teollisuuden käyttöpuun globaalia kysyntää ja puustamaksukykyä metsä- ja puutuoteteollisuuden eri aloilla. Osahankkeessa 2 kehitettiin puunhankinnan suunnitteluun Ruotsin Maatalousyliopiston (SLU) ja Metsäntutkimuslaitoksen (SkogForsk) yhteisen Heureka-ohjelmiston kanssa yhteensopiva puun ominaisuuksia kuvaava malliperhe, jonka avulla voidaan kohdentaa metsistä hakattava puu loppukäytön kannalta optimaalisesti. Osahankkeessa 3 tutkittiin puukaupan strategiseen suunnittelun tueksi vaihtoehtoisten puutavaralajimäärittelyjen eli katkontaohjeiden vaikutuksia leimikoiden arvoon erikseen puunmyyjien ja ostajien kannalta. Samalla tehtiin herkkyysanalyyseja leimikkotekijöiden sekä puutavaralajien yksikköhintojen ja puutuoteteollisuuden tuotehintojen, sivutuotehyvitysten, raaka-aineen käyttösuhteen ja valmistuskustannusten vaikutuksista leimikoiden arvoon. Perus- ja erikoispuutavaralajien kertymien ennustamiseksi ja tätä kautta puuraaka-aineen taloudellisen arvon selvittämiseksi tehtiin puutavaralajikertymämalleja, jotka pohjautuivat joko maastomittauksiin tai laser-keilaimella kerättyyn inventointiaineistoon. Koulutus- ja opetuskäyttöä varten perustettiin kahteen maakuntaan sarjat empiirisiä esimerkkileimikoita, joiden puutavaralajikertymät ja myyntiarvot laskettiin tämän hankkeen vaihtoehtoisilla puutavaralajeilla ja niiden yhdistelmillä. Osahankkeessa 4 tutkittiin kohotetun hiilidioksidin ja lämpötilan vaikutuksia 20–25 vuotiaan männyn kasvuun ja puuaineen ominaisuuksiin. Lisäksi mallinnettiin männyistä, kuusista ja koivuista mitattuja aineistoja hyödyntäen sekä puun sädekasvun jakautumista rungolla että eri puuaineen ominaisuuksia (puuaineen tiheys, kevätpuuprosentti, kuidun pituus) suhteessa puun sädekasvuun ja luston ikään. Näitä puuaineen ominaisuusmalleja käytettiin fysiologisella kasvu- ja tuotosmallilla tehdyissä laskelmissa, joissa tutkittiin metsänhoidon vaikutuksia puiden kasvuun ja eri puuaineen ominaisuuksiin. Osahankkeen 4 suomalaisia puuaineen ominaisuusmalleja verrattiin osahankkeessa 2 kehitettyihin ruotsalaisiin malleihin. Jälkimmäisiä malleja testattiin osahankkeiden 1, 2 ja 4 yhteistyönä myös Pohjois-Karjalan nykyisiä ja tulevia puu- ja kuituvaroja koskevissa MELA-laskelmissa. Osahankkeiden 1 ja 3 yhteistyönä verrattiin MELA-ohjelmistossa käytössä olevaan malliketjuun ja osahankkeessa 3 käytettyyn runkojen simuloituun pölkytykseen perustuvia menetelmiä puutavaralajikertymän arvioinnissa. Tulosten perusteella monikansallinen mallinnus ja mallien soveltaminen eri oloihin on mahdollista, jos malleissa käytetyt selittäjät on määritelty riittävän yleisesti. Osana suunnitteluohjelmistoja (MELA Suomessa ja Heureka Ruotsissa) mallit tulevat jatkossa palvelemaan entistä laajemmin molempien maiden käytännön metsätaloutta metsien käsittelyn, puunhankinnan ja puukaupan suunnittelussa.

Sammanfattning

En tvärvetenskaplig grupp av finska och svenska forskare utvecklade och testade vedegenskapsmodeller som skall kunna användas för planering av virkesutnyttjande. Inom delprojekt 1 utvecklades en skoglig modell som är baserad på det finska MELA-systemet (Metla). En ny metod integrerades i MELA-systemet för att beräkna avverkningspotential och tillgångar. Beräkningar genomfördes med den nya mjukvaran (software) baserat på den finska riksskogstaxeringen för att uppskatta effekter av förändringar i virkesutnyttjande, med skötselstrategier som definierats antingen genom avverkningsmöjligheter eller skötselåtgärder, och klimat. Risken för och påverkan av skogsskador modellerades för att analysera skötselstrategier. För att stödja nationella analyser av optimalt utnyttjande av skogsresursen utvecklades globala scenarier av virkesbehov och betalningsförmåga hos olika skogsindustrier. Inom delprojekt 2 utvecklades ett system av modeller och algoritmer som kan användas till att uppskatta det nuvarande och framtida produktutbytet från ett skogsinnehav. Ett mål var att ta hänsyn till behoven hos de planeringssystem som finns inom ramen för Heureka vid Sveriges lantbruksuniversitet så att de ska kunna användas praktiskt i framtiden. Ett annat var att i samarbete med finska forskare jämföra modellerna med de modeller som utvecklas för MELA-systemet. Inom delprojekt 3 studerades och utvecklades modeller för strategiska

studier av virkesmarknader. Effekter på rotnettot av alternativa kombinationer av olika rundvirkessortiment och deras dimensioner och kvalitetskriterier, d.v.s. kapningsinstruktioner, samt utnyttjandegraden av virkespotentialeten i olika bestånd studerades ur både skogsägarens och virkesköparens perspektiv. Samtidigt utfördes känslighetsanalyser för att skatta effekter av olika beståndsfaktorer, priser på sortiment och träprodukter samt priser på biprodukter, utnyttjandegraden av virket och tillverkningskostnader hos träindustrier på beståndsvärden. Modeller utvecklades för att uppskatta potentiella uttag av olika grund- och specialsortiment, och därigenom virkesvärdet hos olika bestånd, baserade antingen på fälmätningar i skogen eller laserscanning från flygmaskinen. För undervisning etablerades två regionala serier av empiriska modellbestånd i södra och östra Finland, där virkesutbytet och försäljningsvärdet beräknades baserade på projektets alternativa sortiment och deras olika kombinationer. Inom delprojekt 4 studerades effekter av höjd CO_2 och temperatur på tillväxten och vedegenskaper hos tall. Till sist användes mätdata för tall, gran och björk i statistiska modeller där radiell tillväxt modellerades tillsammans med vedegenskaper (densitet, vårvedsandel och fiberlängd) i relation till radiell tillväxt och antal årsringar från märgen. Dessa egenskapsmodeller användes även i beräkningar kopplade till fysiologiska tillväxtmodeller för att studera effekter av skötselåtgärder på tillväxt och vedegenskaper. Egenskapsmodellerna för gran och tall jämfördes också med svenska modeller av delprojekt 2. De senare testades i samarbete mellan delprojekt 1, 2 och 4 i MELA-beräkningar av nuvarande och framtida skogstillgångar i Norra Karelen. Dessutom jämfördes metoder för att skatta sortimentsutbyten med modellsystemet i MELA och programvara för apteringssimulering i samarbete mellan delprojekt 1 och 3. Möjligheterna att samordna modellutvecklingen inom de båda länderna ser bra ut. Som en del av skogliga planeringssystem (MELA i Finland och Heureka i Sverige) kommer modellerna som utvecklades i projektet att ge omfattande nya möjligeter för det praktiska skogsbruket i de båda länderna genom effektivare planering av skogsskötsel, virkesförsörjning och virkesmarknad.

3.1 Introduction

3.1.1 Background

The effects of silvicultural practices on the growth of trees and the consequent stem and wood properties operate through interactions between biological processes (i.e. height growth, radial growth of the stem, crown development) and environmental conditions (e.g. temperature, precipitation, nutrients and light). Furthermore, the material properties of wood are also affected by forest damages, such as butt-rot. Consequently, variations in different wood properties influence both the quality and quantity of pulp and paper and the sawn timber products for which the material is used. Therefore, there is need for deeper understanding how environmental factors, forest structure and silvicultural practice, and also forest damage, affect tree growth with implications on the material properties of stem and wood.

To be able to incorporate the end-use requirements into the forestry models and planning systems, methods are needed to translate the industrial needs into meaningful measures and definitions of wood raw material, compatible with forestry modelling and planning systems. The information on the definition and relative values of various wood assortments, and their development trends in the future, is required to make wood growing decisions that reflect the profitability of the future forest industries.

3.1.2 Objectives

The consortium (figure 1) aimed to provide information on the variation of wood properties and end-user requirements for the forestry planning systems such as MELA in Finland and Heureka in Sweden. In addition, the joint Finnish-Swedish team collaborated in the further development and validation of the knowledge and models for wood properties in order to analyse how trees in different environmental conditions react to silvicultural methods.

In sub-project 1, a model was developed for the analysis of forest management strategies based on the Finnish MELA system and national forest inventory (NFI) data to provide information on the

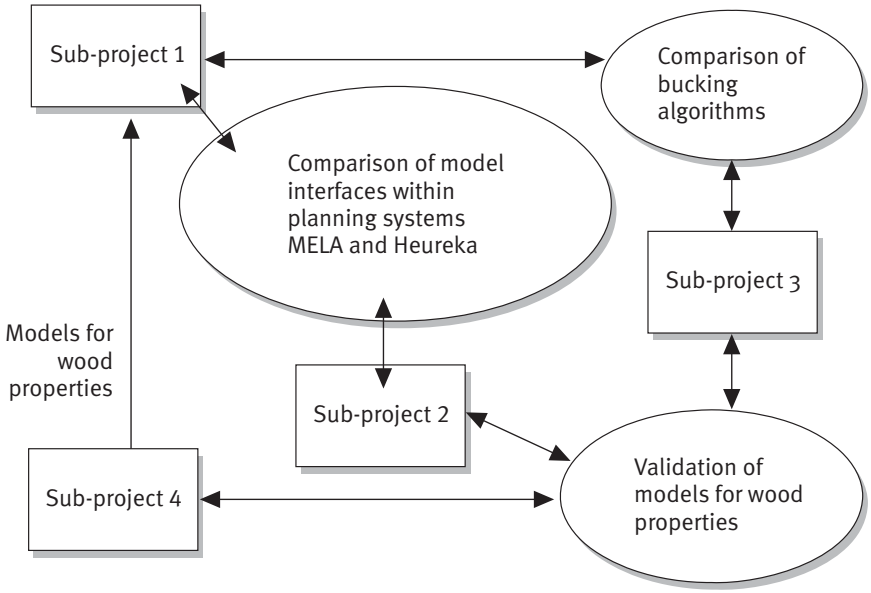

Figure 1. Interfaces between sub-projects.

future wood and fibre resources of Finnish forests under different management strategies, and to derive optimal forest management strategies reflecting different scenarios of wood use and wood-paying capability of forest industry. In addition, sub-project 1 aimed at developing models for the occurrence and consequence of forest damage, such as butt-rot, to incorporate the consequent quality losses connected to various management alternatives into the analysis.

In sub-project 2, the objective was to develop a system of models and algorithms which can be used to describe the industrial potential from the present and future forest resource in terms of wood and fiber properties, in the context of forest management planning systems. The performance of these models was compared in a cross-validation study in collaboration with Finnish partners.

In sub-project 3, the objective was to provide information on how the characteristics of the stand, dimensions and quality factors of timber assortments as well as principles and objectives of bucking affect the sales value of a stand. Results will support a novel planning approach for timber purchasing and sales applications as a part of value chain analysis, and obviously clarify the confused situations between forest owners and forest industry amidst in cut-to-length method based timber markets.

In sub-project 4, influence of environmental factors, forest structure and silvicultural practices on wood properties was studied and models constructed for the wood properties in order to analyse how trees in different environmental conditions will react to silvicultural methods.

3.2 Results and discussion

In sub-project 1, a forestry model was developed based on the MELA software of the Finnish Forest Research Institute (Metla). The project started with an international review of forestry models (Nuutinen 2003) and continued with the development of a method to compare silvicultural strategies (Nuutinen et al. 2006a). In addition, a new method was incorporated into the MELA software for calculating total felling potential and reserves (Nuutinen & Kettunen 2005, Redsven et al. 2005). Calculations were made using the new software version and the national forest inventory (NFI) data to determine the future forest resources and cutting possibilities (figure 2; Nuutinen 2005a,c,

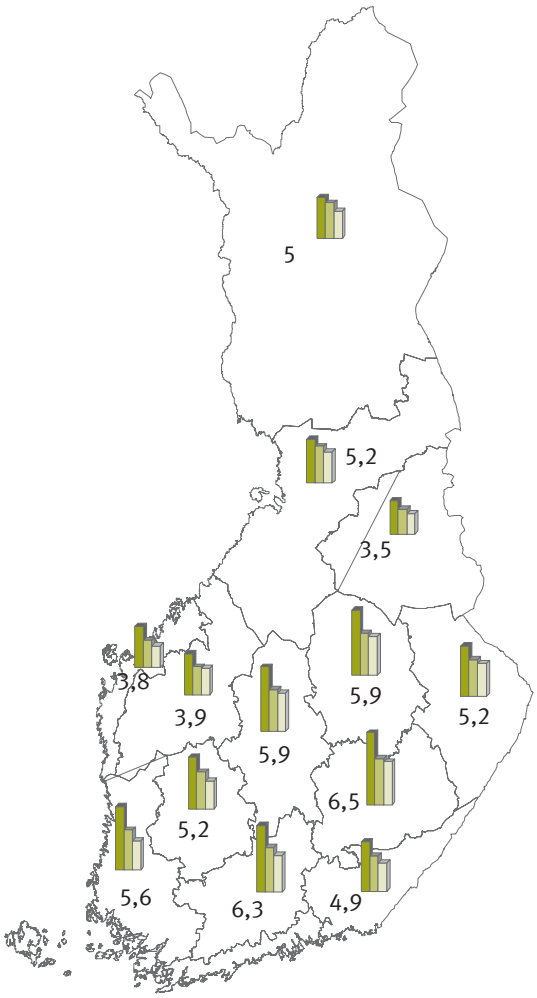

Figure 2. Regional comparison of three cutting strategies (Nuutinen et al. 2005c).

d,e) and how the changes in wood use (Nuutinen et al. 2004a, Nuutinen et al. 2005j), forest management strategies (Nuutinen et al. 2005b), and climate (Nuutinen et al. 2006b) affect the future forest resources and felling potential. The models were developed for the occurrence and consequence of forest damage (e.g. *Heterobasidion* root and butt rot damage to spruce stands and spruce trees and pine twisting rust damage in Scots pine stands) to incorporate into the analysis the consequent quality losses connected to various management alternatives (Mattila 2005, Mattila & Nuutinen 2007). According to the studies, root and butt rot damage was more common on fertile sites and in such stands where special or selective cuttings had been carried out, and the occurrence of aspens and site fertility were the most important factors increasing the probability that pine twisting rust damage will occur in a stand. To assist national analysis of optimal forest resources, the global trends of the demand and value of different wood assortments were studied, and wood paying capability of mechanical and chemical wood industry branches analysed based on the simulation of production processes (Kärkkäinen 2003a,b; 2004; 2005a,b).

In sub-project 2, a family of models was developed by the Forestry Research Institute of Sweden (Skogforsk) as a compatible extension to the Heureka software of the Swedish University of Agricultural Sciences (SLU). These models describe wood properties such as density, heartwood content, latewood content, juvenile wood content,

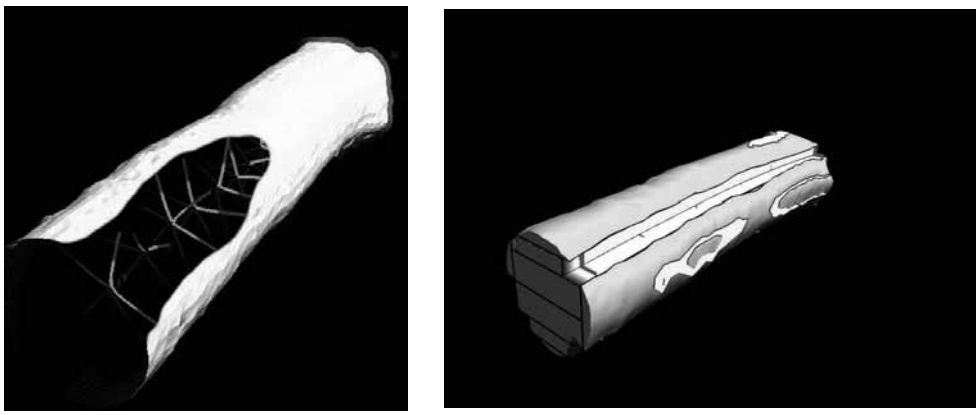

Figure 3. Examples of the use of intra-tree models.

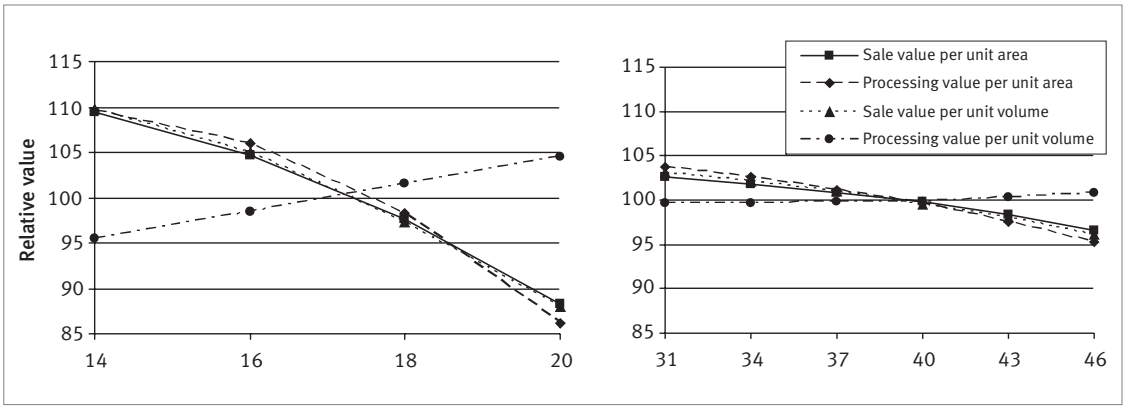

Figure 4. The impacts of minimum log diameter (14 cm, 16 cm, 18 cm and 20 cm) and minimum saw log length (31 dm, 34 dm, 37 dm, 40 dm, 43 dm and 46 dm) on the average sales and processing value in Scots pine stands when saw logs and pulpwood were harvested (Malinen et al. 2007c).

knot characteristics, moisture content and fibre dimensions (figure 3; Ekenstedt et al. 2003; Moberg & Wilhelmsson 2003; Moberg 2004; Moberg & Nordmark 2004a,b; Moberg 2005; Moberg 2006a) and can be used when designing optimal delivery of timber from the forest to the end-users. In addition, the use of a radar-based remote sensing method and laser-based technology together with field measurements for data acquisition were evaluated (Ekstrand & Walter 2006, Moberg 2006b, Moberg & Nordmark 2006a, Moberg & Nordmark 2006b). The results indicated that both the radar- and laser-based methods performed fairly well for large trees, but it was difficult to detect small trees (under a DBH of about 20 cm) because of the low contrast to background noise in the images.

In sub-project 3, the variation in the log recoveries as well as in the sales and processing values (Piira 2005, Piira et al. 2007, Malinen et al. 2007c,d) were calculated when targetting to alternative timber assortments and dimensions by using field mesurement data from 124 Scots pine or Norway spruce dominated final fellings and 17 thinnings in Scots pine stands, which was used in bucking simulation. Maximizing saw log percentage and including special assortments meets the interests of forest owners and those of wood industries when there is a shortage of logs in the market. However, when the users have no log shortage, the value recovery from timber can be increased by increasing minimum allowable log diameter and length, thus, increasing the average size of the logs (figure 4). A variety of sensitivity analyses were performed to determine the responses to the sales and processing values per stand hectare and per unit volume of timber; the factors tested included different stand properties and unit prices of timber assortments, as well as product prices, by-product credits, log utilization ratios, and manufacturing costs in wood product industries.

Critical stand characteristics and bucking features were analyzed and models predicting assortment recoveries were created (Malinen et al. 2007b,d). These models were based on the assumption that the typical stand characteristics depicting tree stock volume are available (figure 5). The predictor variables included variables depicting size distribution of the trees, quality of the trees and used bucking regimes. However, pre-harvest measurements are sometimes considered too expensive and laborious (see e.g. Malinen 2004), and the general interest is towards new remote sensing technology, such as air-borne laser scanning (ALS). For the prediction of the crown ratio, saw log recovery and log length-diameter distribution the models based on the ALS data were constructed. The use of ALS in the pre-harvest measurement was studied in co-operation with the forest mensuration group in the University of Joensuu, Faculty of Forestry (Packalén et al. 2005,

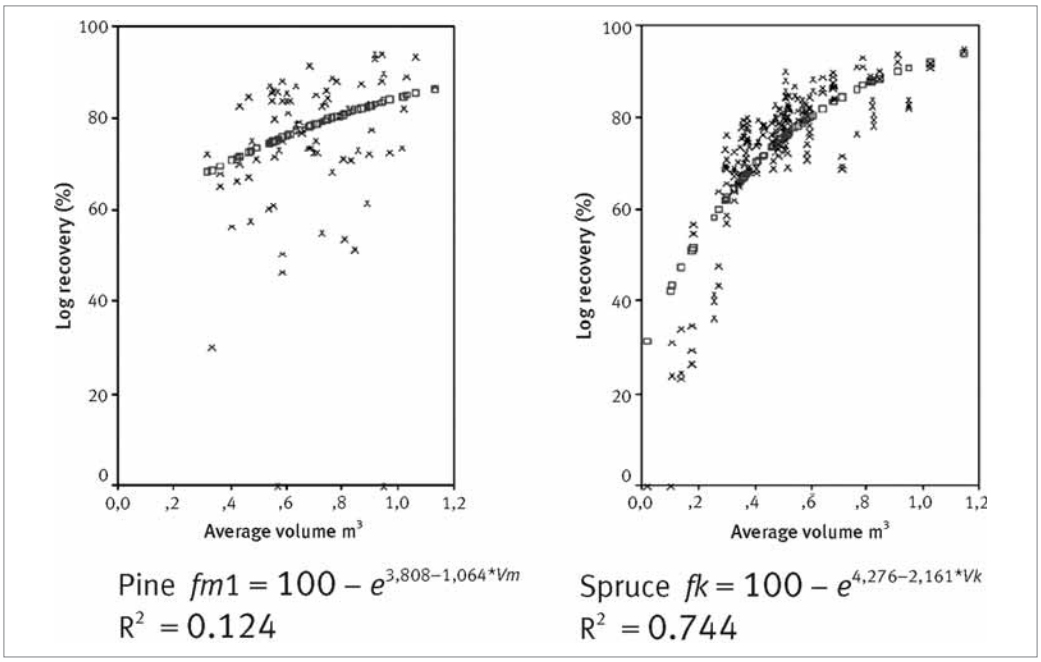

Figure 5. The impacts of average stem volume on the saw log recovery (%) in the final fellings of Scots pine and Norway spruce stands, when saw logs and pulpwood were harvested and the sales value was maximized in the simulation (Piira et al. 2007).

Maltamo et al. 2006a, Maltamo et al. 2006b, Peuhkurinen et al. 2007, Korhonen et al 2007).

For training and teaching purposes, empirical model stands were established in two provincial areas (northern Karelia / Nurmes, south-western Finland / Paimio). Here, the timber yields and sales values of individual stems and entire stand were calculated based on this project's alternative timber assortments and their combinations.

In sub-project 4, the chamber study of sub-project 4 provided information on how the year around, all day, elevation in CO_2 and temperature alone or concurrently affect the growth dynamics of 20-25 years old Scots pine with implications on both physical and chemical properties of wood (Kilpeläinen 2005, Kilpeläinen et al. 2003, 2005, 2006, 2007). In addition, data measured from Scots pine, Norway spruce and silver birch were used in statistical models where radial growth distribution on stem was modelled in addition to wood properties (wood density, percentage of early wood, fibre length), which were modelled in relation to radial growth and tree ring age (see Ikonen et al. 2006, Peltola et al. 2007; figure 6). These wood property models were also used in calculations with a physiological growth and yield model to study the impact of silvicultural procedures on tree growth and various wood properties.

In co-operation between the sub-projects 1, 2 and 4, the ring-based wood property models (by sub-project 4) in Scots pine and Norway spruce were also compared with the Swedish disc-based property models (by sub-project 2) (Peltola et al. 2006). The latter models were also applied in the MELA analysis made on present and future timber and pulpwood resources in northern Karelia by sub-project 1 in co-operation with sub-projects 2 and 4 (figure 7; Nuutinen et al. 2007).

In addition, methods for estimating yields of timber assortments either with the chain of models in MELA software or bucking simulation software of stems were compared in co-operation between sub-projects 1 and 3 (figure 8; Malinen et al 2007a).

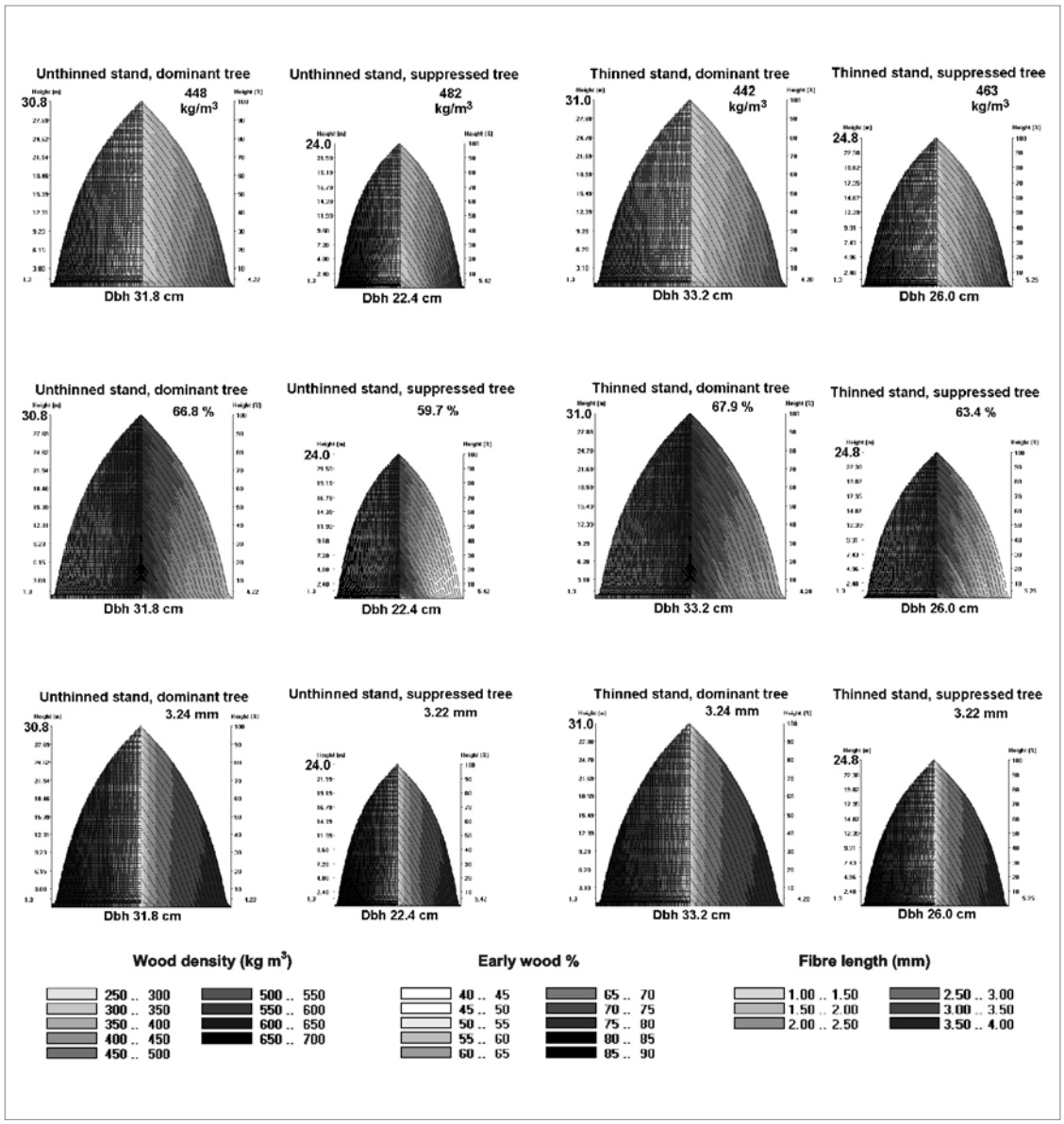

Figure 6. Examples of computations by the process-based growth and yield model (FinnFor) with wood properties models for air dry wood density (above), early wood % (middle) and fibre length (below) in dominant and suppressed Scots pine trees grown in unthinned and thinned stands over 90 years rotation. These stand were planted with 2500 seedlings/ha and in thinned stand two thinnings were done during the rotation (i.e. first thinning from below was done in 40 years old stand with 1000 stems ha^{-1} left after thinning and second one in 70 years old stand with 500 stems ha^{-1} left after thinning) (Peltola et al. 2006).

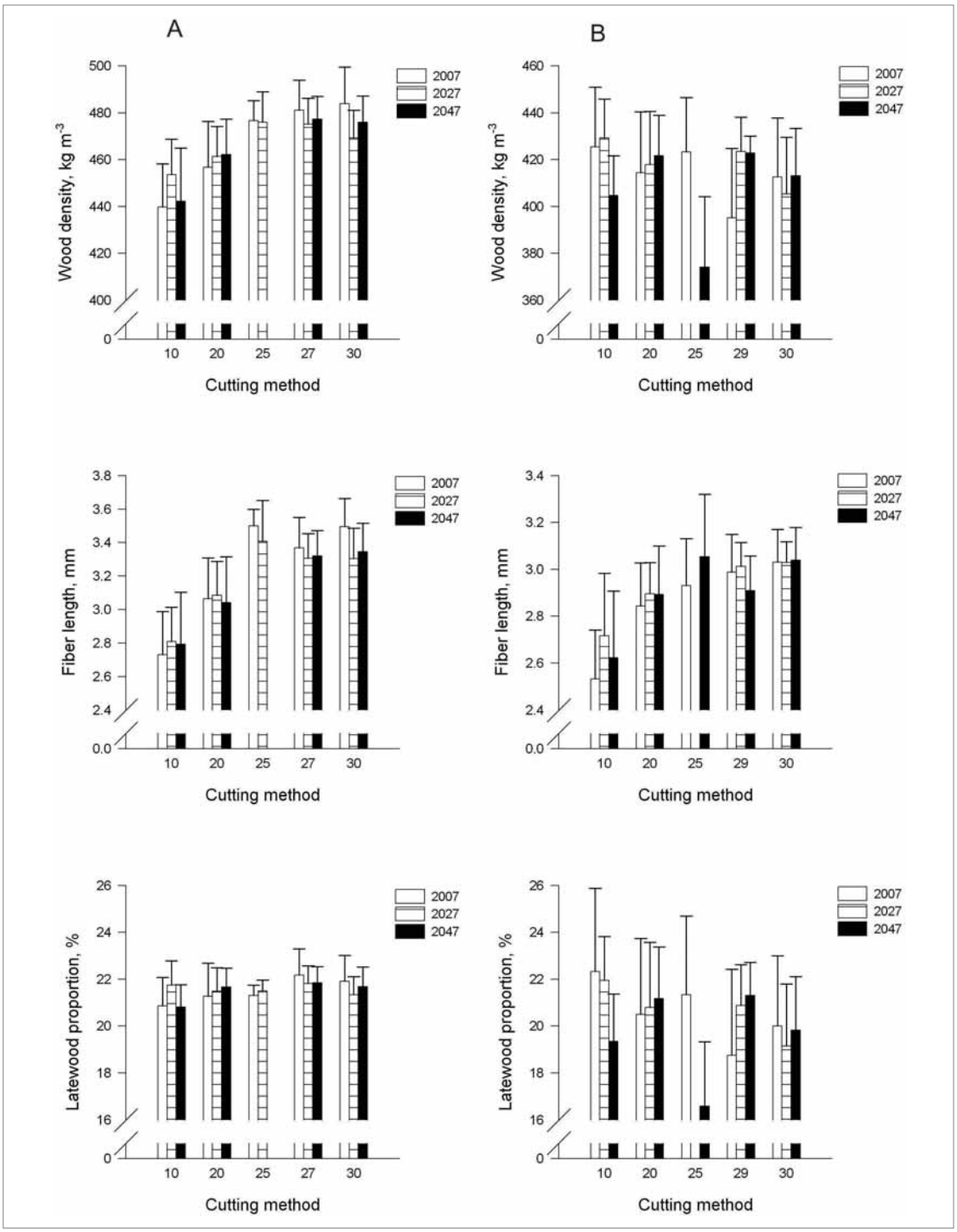

Figure 7. Wood properties of the harvested Scots pine (A) and Norway spruce trees (B) in different cutting methods, 10= (first) thinning, 20=basal area thinning, 25=over story removal, 27=seed tree cutting (pine), 29=shelterwood cutting (spruce), 30=clear cutting. (Nuutinen et al. 2007)

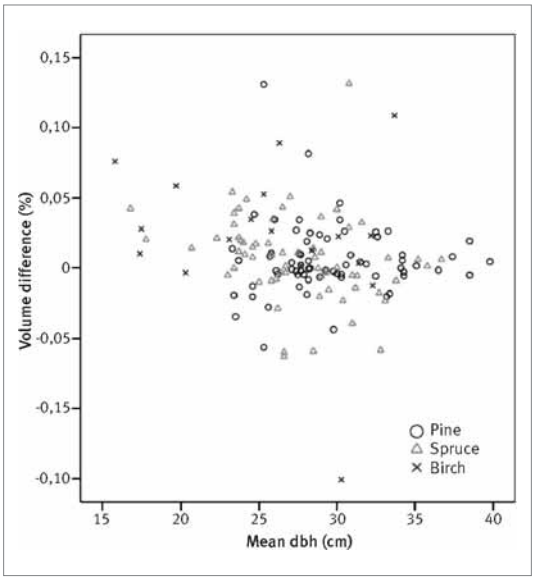

Figure 8. The differences between model-based approaches and bucking simulation in the volume recoveries for pine, spruce, and birch. (Malinen et al. 2007a)

3.3 Conclusions

A multi-disciplinary Finnish-Swedish research team has developed and tested models on wood and timber properties applicable for planning the utilization of forest resources. For the effective integration of the models with the planning systems, emphasis was laid on the proper interfacing of models with data acquisition techniques, planning routines and information systems.

Wood property models being developed in sub-project 2 through extensive use of mixed model theory applied for the conventional tree measurements make it possible to predict properties with a high intra-tree resolution. The proper interfacing of models with new data acquisition methods is a key issue in order to obtain reliable results in different applications. The preliminary evaluation and independent cross-validation of the models carried out together with sub-projects 1, 2 and 4 using datasets from Finland indicates that it will be possible to implement models in planning systems and in different environments, and to apply these in order to predict the industrial potential of the present and future Swedish and Finnish forest resources.

The results of the sub-project 3 demonstrate the effects of varying bucking objectives and enable comparison of the bids of potential wood buyers with different selections of timber assortments and different dimensions and grades. In addition, the results on the variation in the processing value illustrate the reasons for the different wood product industries to individually adjust the minimum log length and/or diameter according to the end-products and markets. The results add to the understanding of value formation both from the forest owners' and wood buyers' viewpoints, improve the overall knowledge of the effects of bucking instructions and contribute to making conclusions on the strategic effects of alternative selections of timber assortments on the income flow within the value chain of wood procurement and timber trade. However, the results cannot be used to directly compare the profitability of wood processing for different end-uses, because the costs of timber harvesting, transport and storage or marketing and distribution costs of wood products were not considered. Neither can the log recovery, sales value or processing value be predicted in individual stands in timber trade. The behaviour of timber harvester driver could not be taken into account, either.

The chamber study of sub-project 4 is the first attempt to understand the effect of year around, all day, elevation in CO_2 and temperature alone or concurrently on growth of Scots pine older than young seedlings in juvenile phase with implications on the properties of wood. However, the extrapolation of these findings directly for mature trees may still be difficult, because the wood properties of these Scots pines differ from those of mature trees. Information of this kind is both important in validation of physiological growth model (FinnFor) and urgently needed in the forest industry. The further modelling work carried out in sub-project 4 related to the distribution of growth and consequent wood properties along the stem of Scots pine, Norway spruce and silver birch offer means to study how environmental factors, forest structure and silvicultural practices affect tree

growth with implications on the material properties of stem and wood (and their distribution along the stems) (see Peltola et al. 2006). In this context, the co-operation especially with sub-project 2 was crucial in regard to validation of the ring-based models for wood properties.

The integration of the Swedish disc-based wood property models into the MELA model (by sub-project 1 in co-operation with sub-projects 2 and 4), opens up new possibilities to analyse current and future wood and fibre resources to support, for example, the strategic investment decisions of forest industry. In addition to strategic analyses, the models serve in daily wood procurement decisions. According to tests by sub-projects 1, 2 and 3, the model chains are compatible enough with the input data of new remote sensing technology (both radar and ALS) regarded cost-efficient tools to replace field measurements in pre-harvest inventory.

3.4a Capabilities generated by the project

A multi-disciplinary Finnish-Swedish research team has developed and tested models on wood and timber properties applicable for planning the utilization of forest resources.

3.4b Utilisation of results

Both the Finnish MELA system and the Swedish Heureka system provide platforms for the dissemination and exploitation of the modeling results from the different sub-projects. Through these systems the models will provide extensive benefits for practical forestry in both countries in the planning of forest management, wood procurement and timber trade. For example, the information on the future wood and fibre resources of forests under different management strategies will support in the investment decisions of forest industry and the national level analysis of optimal forest management strategies reflecting different scenarios of wood use and wood-paying capability of forest industry will provide information for the adjustment of forest management to correspond the profitability of forest industry.

The results of the sub-project 3, especially on the variation in the sales value according to the bucking objectives, minimum log diameter and length, have been submitted to, and used by the forest owner associations and forest enterprises in their development work. The experimental model stands established in the project in order to demonstrate the effect of different bucking regimes in different stands contribute to the education forest owners, wood buyers and timber harvester drivers.

During the project, modelling approaches and their compatibility was analysed. According to the results there is potential for multi-national modelling and model sharing between Finland and Sweden if the independent variables are defined generally enough. The results from this co-operation provide an excellent basis to establish a formal collaboration between the developers and users of the MELA and Heureka systems.

3.5 Publications and communication

a) Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

Ikonen, V-P., Kellomäki, S., Väisänen, H., Peltola, H. 2006. Modelling the distribution of diameter growth along the stem in Scots pine. Trees 20:391-402.

Kilpeläinen, A., Peltola, H., Ryyppö, A., Sauvala, K., Laitinen, K. and Kellomäki, S. 2003. Wood properties of Scots pines (*Pinus sylvestris*) grown at elevated temperature and carbon dioxide concentration. Tree Physiology 23:889-897.

*Kilpeläinen, A., Peltola, H., Ryyppö, A., and Kellomäki, S. 2005. Scots pine responses to elevated temperature and carbon dioxide concentration: growth and wood properties. Tree Physiology 25:75-83.

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Kilpeläinen, A., Zubizarreta, A., Luostarinen, K., Peltola, H., Kellomäki, S. 2007. Effects of elevated temperature and CO_2 concentration on the anatomical xylem characteristics of Scots pines (*Pinus sylvestris* L.). Tree Physiology. In print.

*Malinen, J., Kilpeläinen, H., Piira, T., Redsven, V., Wall, T. & Nuutinen, T. 2007a. Comparing model-based approaches with bucking simulation based approach in the prediction of timber assortment recovery. Revised for Forestry.

Maltamo, M., Hyyppä, J. & Malinen, J. 2006a. A comparative study of the use of laser scanner data and field measurements in the prediction of crown height in boreal forests. Scandinavian Journal of Forest Research 21:231-238.

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*Moberg, L. & Nordmark, U. 2006a. Predicting lumber volume and grade recovery from Scots pine stems using tree models and sawmill conversion simulation. Forest Products Journal 56(4): 68-74.

Nuutinen, T.. Anola-Pukkila, A., Lempinen, R., Redsven, V., Siitonen, M. 2006A. Balancing between set-aside and forest management activities of stands using integrated stand and forest level optimization. Allgemeine Forst- und Jagdzeitung 177 ¾: 71-76.

Nuutinen, T., Matala, J., Hirvelä, H., Härkönen, K., Ojansuu, R., Peltola, H., Väisänen, H., Kellomäki, S. 2006B. Regionally optimized forest management under changing climate. Climatic Change 79(3-4):315-333.

Peltola, H., Kilpeläinen A., Sauvala K., Räisänen T. and Ikonen V.-P. Effects of the early thinning regime and the tree status in a stand on the radial growth of Scots pine with implications for wood properties. 2007. Silva Fennica, in review process.

Submitted

Peuhkurinen, J., Maltamo, M., Malinen, J., Pitkänen, J. & Packalen, P. 2007. Pre-harvest measurement of marked stand using airborne laser scanning. Submitted to Forest Science.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

*Malinen, J., Kilpeläinen, H., Wall, T. & Verkasalo, E. 2007c. Variation in the value recovery when bucking to alternative timber assortments and log dimensions. Forestry Studies / Metsanduslikud uurimused 45: 89-100.

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3. Articles in Finnish and Swedish journals with referee practice

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Piira, T., Kilpeläinen, H, Malinen, J., Wall, T. & Verkasalo, E. 2007. [Variation in timber recovery and sales value in marked stands by using different bucking models] Leimikon puutavaralajikertymän ja myyntiarvon vaihtelu erilaisilla katkontaohjeilla. Accepted to Metsätieteen aikakauskirja. (In Finnish)

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice

5. Scientific monographs

Kilpeläinen, A. 2005. Growth and wood properties of Scots pines (*Pinus sylvestris* L.) at the elevated temperature and carbon dioxide concentration. Ph.D. thesis (Agr. and For.), Research Notes, University of Joensuu, Faculty of Forestry (summary with 4 scientific papers).

Piira, T. 2005. [Impact of the bucking constraints on log recovery and sales value] Katkontaohjeiden vaikutus leimikon puutavaralajikertymään ja myyntiarvoon. M.Sc. Thesis, University of Joensuu, Faculty of Forestry. 52p. (In Finnish)

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series

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Ekstrand, M. & Walter, F. 2006. Carabas production line – individual trees. The Forestry Research Institute of Sweden, Uppsala, Work report No. 615.

Kärkkäinen, M. 2004. Sawmill simulators in silvicultural and technical research. In: Forest ecosystems in the changing world. Silva Carelica 44:33-52. (In Finnish)

Kärkkäinen, M. 2005a. Kuvaako sanomalehtipaperin kulutus demokratian toimivuutta? In: Tyynelä, T. & Pelkonen, P. (Eds.). Metsien kokonaisarvon jäljillä - In search of a total value of forests. Professori Olli Saastamoinen 60 vuotta. Silva Carelica 50:161-166. 6 p. (In Finnish)

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Malinen, J., Kilpeläinen, H., Piira, T., Wall, T. & Verkasalo, E. 2007d. Variation in the sales and processing value of timber stand by its timber quality when targeting for alternative end-products in shortwood harvesting. In: Nepveu, G. (ed.). Proceedings of IUFRO WP S5.01-04 Fifth Workshop "Connection between Forest Resources and Wood Quality: Modelling Approaches and Simulation Software". November 20-27, 2005, Waiheke Island Resort, Aucland, New Zealand. (in print)

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Moberg, L. 2006b. Precision i planeringen styr framtida utfall. [Precise planning determines future results.] I Bruzelius, B. (Red.): Mer guld i grön skog. Miljöforskning Nr. 3 2006, Formas, Stockholm. pp. 17-18. (In Swedish.)

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Nuutinen, T. & Kettunen, L. (toim.) 2005. MELA 2005 ja -nettisovellukset. Metlan työraportteja 15. 65 p. (In Finnish)

Nuutinen, T., Hirvelä, H. & Salminen, O. 2005b. Etelä-Suomen metsien kehitys - vuosille 2001-2005 tehtyjen alueellisten metsäohjelmien vaikutusanalyysi. Metlan työraportteja 12. 22 p. (In Finnish)

Nuutinen, T., Hirvelä, H. & Salminen, O. 2005c. Alueelliset hakkuumahdollisuudet Suomessa. Metlan työraportteja 13. 73 p. (In Finnish)

Nuutinen, T., Hirvelä, H. & Salminen, O. 2005d. Kestävistä hakkuumahdollisuuksista hyödynnetään hieman yli 80 prosenttia. In: Peltola, A. (Eds.). Metsätilastollinen vuosikirja 2005 - Skogsstatistisk årsbok - Finnish Statistical Yearbook of Forestry. SVT Maa-, metsä- ja kalatalous 2005:45: 155.

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Software". November 20-27, 2005, Waiheke Island Resort, Aucland, New Zealand. In print.

Verkasalo, E. 2005. [Grading and price determination of timber] Puutavaran laadutus ja arvon määrittäminen. In: Heräjärvi, H. & Hakkila, P. (eds.). Metsän ja puun asialla - Professori Matti Kärkkäinen 60 vuotta. Silva Carelica 51: 74-87. (In Finnish)

7. Manuscripts in preparation

Korhonen, L., Peuhkurinen, J., Malinen, J., Suvanto, A., Maltamo, M., Packalén, P. & Kangas, J. 2007. Airborne laser scanning in estimation of saw log volume on clear cut stands. Manuscript.

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Malinen, J. 2006. [From stump to end-user – bucking of stem as pre-processing decission] Kannolta käyttäjälle – rungon katkonta esijalostusprosessina. Puutiede tänään- seminaari, Joensuu 23.5. 2006. Metla Joensuu 25 vuotta -juhlaseminaarisarja. Oral presentation (Finnish abstract, 1p)

Malinen, J., Kilpeläinen, H., Piira, T., Wall, T & Verkasalo E. 2005a. [Value formation of the marked stand – Sales value] Leimikon arvon muodostus - myyntiarvo. PKM-tutkimusohjelman tutkimuspäivä 4.10.2005, Lahti, Sibeliustalo. Metsäntutkimuslaitos, Joensuu. Oral presentation (Finnish abstract, 1 p.).

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Nuutinen, T. 2004. Skoglig planering - Finska erfarenheter. Proceedings from ”Skogskonferensen” held in Uppsala, Sweden on 30 Nov. – 1 Dec. 2004, The Swedish University of Agricultural Sciences, Uppsala, Sweden. p. 4. (In Swedish).

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3.6 National and international cooperation

In collaboration between Skogforsk (sub-project 2), University of Joensuu (sub-project 4) and Metla (sub-project 1) wood property models developed in Sweden were tested against an independent dataset collected in Finland, and vice-versa (Peltola et al. 2006).

The project collaborated with the Forest Management Planning research programme of the Finnish Forest Research Institute and Heureka research programme co-ordinated by the Swedish Agricultural University. In sub-project 1, partners included the Finnish Environment Institute and Metsäteho. In sub-project 4, the work related to the modelling of tree growth with implications on the properties of Scots pine, Norway spruce and birch was carried out at the Faculty of Forestry, University of Joensuu in a co-operation with the Centre of Excellence for Forest Ecology and Management (Project no. 64308), led by Academy Prof. Seppo Kellomäki, under the Finnish Centre of Excellence Programme (2000-2005). The work carried out in regard to the modelling of the distribution of diameter growth along the stem of Scots pine was also related to the International Project "Compression wood in conifers: the characterisation of its formation and its relevance to timber quality" (Compression Wood, funded by European Commission, 2001-2004), led by Prof. Barry Gardiner, Forestry Commission, UK. The scientific expertise of the Centre of Expertise for Wood Technology and Forestry working under the Joensuu Science Park was also available for the project.

In addition, continuous contacts were kept with the industry, forest managers and relevant research teams in order to guarantee the applicability of the results. The role of an advisory group consisting of the representatives of forest owners and forest industry was to participate in the definition of relevant management strategies and relevant scenarios for the end-use of wood. In addition, the members of advisory group represented the potential users of the results. The advisory group consisted of 7 members.

Advisory group

Torleif Carlsson, Manager, Stora Enso
Juha Hakkarainen, Research manager, The Central Union of Agricultural Producers and Forest Owners
Jukka Ranua, Manager, M-Real
Lennart Rådström, Research director, Skogforsk
Jukka-Pekka Ranta, Director, Finnish Sawmills
Tuula Nuutinen, Professor, Finnish Forest Research Institute
Leena Paavilainen, Programme director (until 31.5.2006), Wood Material Research Program

4 Impact of forest management and climate on tree structure and wood quality (IMWO)

FINAL REPORT	
Name of the research project	**Impact of forest management and climate on tree structure and wood quality**
Coordinator of the project	Pekka Saranpää

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**Impact of management and climate on partitioning of biomass in Norway spruce**
Project period	1.7.2003–31.12.2006
Organization in charge of research	Swedish University of Agricultural Sciences (SLU)
Sub-project leader	Professor Sune Linder
Contact information of the sub-project leader	SLU, Southern Swedish Forest Research Centre, P.O. Box 49, SE-230 53 Alnarp, Sweden Tel. +46 (0)40 415 162 Fax +46 (0)40 462 325 sune.linder@ess.slu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	251 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	131 000
SLU	120 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sune Linder, Ph.D., Prof.	M	SLU, Southern Swedish Forest Research Centre, Alnarp		SLU, Swedish University of Agricultural Sciences
Johan Bergh, Ph.D., Docent	M	SLU, Southern Swedish Forest Research Centre, Alnarp		SLU

Tomas Lundmark, Ph.D., Docent	M	SLU, Vindeln Experimental Forests, Vindeln	SLU
Michael Freeman, Ph.D.	M	SLU, Dept. of Ecology and Environmental Research, Uppsala	SLU
Thomas Severinsson, M.Sc.	M	Ph.D.-student, SLU, Southern Swedish Forest Research Centre, Alnarp	Formas

Total person-months of work conducted by the research team 60
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	Fil. Lic.	M	Sivertsson, Thomas, 1972	SLU, Alnarp	Sune Linder, Urban Nilsson, SLU, Alnarp

Name of the sub-project 2	**Effects of silvicultural treatments on wood-quality in Norway spruce plantations**
Project period	1.7.2003–1.7.2007
Organization in charge of research	Swedish University of Agricultural Sciences (SLU)
Sub-project leader	Professor Urban Nilsson
Contact information of the sub-project leader	SLU, Southern Swedish Forest Research Centre, P.O. Box 49, SE-230 53 Alnarp, Sweden Tel. +46 (0)40 415 193 Fax +46 (0)40 462 325 urban.nilsson@ess.slu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	300 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	300 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Urban Nilsson, Ph.D., Prof.	M	SLU, Southern Swedish Forest Research Centre, Alnarp		SLU

Oriana Pfister, M.Sc.	F	Ph.D.-Student, SLU, Southern Swedish Forest Research Centre, Alnarp	Formas

Total person-months of work conducted by the research team 52
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	Ph.D.	F	Pfister, Oriana, 1971	SLU, Alnarp	Urban Nilsson, Eric Agestam, SLU, Alnarp, Göran Öhlander, Växjö

Name of the sub-project 3	**Effect of forest management and climate on wood structure and fibre properties**
Project period	1.1.2003–31.12.2005
Organization in charge of research	Finnish Forest Research Institute (Metla)
Sub-project leader	Docent Pekka Saranpää
Public financing organizations	Ministry of Agriculture and Forestry, Finnish Forest Research Institute
Contact information of the sub-project leader	Finnish Forest Research Institute, Vantaa Research Centre, Jokiniemenkuja 1, P.O. Box 18, FI-01301 Vantaa Tel. +358 10 211 2340 Fax +358 10 211 2203 Pekka.Saranpaa@metla.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	554 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Ministry of Agriculture and Forestry	254 000
Metla	300 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Pekka Saranpää, Ph.D.,docent	M	Metla, Vantaa		Metla
Riikka Piispanen, Ph.D., Researcher	F	Metla, Vantaa		MoF

Harri Mäkinen, Ph.D., Researcher	M	Metla, Vantaa	Metla
Erkki Pesonen, Lic., Researcher	M	Metla, Vantaa	Metla
Tuula Jaakkola, M.Sc., Reseacher	F	Ph.D.-student, Univ. of Helsinki	SLTS
Marko Peura, M.Sc., Researcher	M	Ph.D.-student, Univ. of Helsinki	HY
Sanni Raiskila, M.Sc., Researcher	F	Ph.D.-student, Univ. of Helsinki	AoF, Tekes
Matti Sarén, Ph.D., Researcher	M	Ph.D.-student, Univ. of Helsinki	SLTS
Minna Pulkkinen, M.Sc., Researcher	F	Ph.D.-student, Univ. of Joensuu	MoF
Mikael Agopov, Research assistant	M	Undergraduate student, Univ. of Helsinki	MoF
Satu Lehto, Research assistant	F	Undergraduate student, Univ. of Helsinki	MoF
Tiina Koponen, M.Sc., Researcher	F	Ph.D.-student, Univ. of Helsinki	UH
Total person-months of work conducted by the research team 180 person-month = full-time work for at least 36 h/week, paid holidays included			

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2004	Ph.D.	F	Piispanen, Riikka, 1964	University of Helsinki	Pekka Saranpää, Metla, Kurt Fagerstedt, University of Helsinki
2004	M.Sc.	M	Agopov, Mikael, 1978	University of Helsinki	Erkki Pesonen, Metla
2006	Ph.D.	M	Sarén, Matti, 1976	Universities of Helsinki and Oulu	Ritva Serimaa, University of Helsinki, Pekka Saranpää, Erkki Pesonen, Metla
2007	Ph.D.	M	Peura, Marko, 1977	University of Helsinki	Ritva Serimaa, University of Helsinki
2005	M.Sc.	F	Lehto, Satu, 1977	University of Helsinki	Riikka Piispanen, Pekka Saranpää, Metla
2005	M.Sc.	F	Koponen, Tiina, 1981	University of Helsinki	Ritva Serimaa, Edward Haeggström, Timo Karppinen, University of Helsinki, Pekka Saranpää, Metla

Name of the sub-project 4	Impact of forest management and climate on wood chemistry
Project period	1.1.2003–31.12.2005
Organization in charge of research	Finnish Forest Research Institute
Sub-project leader	Doc. Elina Vapaavuori
Contact information of the sub-project leader	Finnish Forest Research Institute, Suonenjoki Research Station, Juntintie 154, FI-77600 Suonenjoki, Tel. +358 10 211 4888 Fax +358 10 211 4801 Elina.Vapaavuori@metla.fi
URL of the project	http://www.metla.fi/pp/EVap/ http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	500 140
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	200 140
Metla	300 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Vapaavuori Elina Ph.D., Docent	F	Metla Suonenjoki		Metla
Kaakinen Seija Ph.D., Docent	F	Metla Suonenjoki		AoF
Kostiainen Katri MSc, Researcher	F	Metla Suonenjoki		SLTS, AoF
Iivonen Sari Ph.D., Researcher	F	Metla Suonenjoki		AoF
Jalkanen Hanna Research assistant	F	Undergrad.student University of Jyväskylä		AoF
Warsta Elina Research assistant	F	Undergrad. student Techn. Univ. Helsinki		AoF
Pohjanen Johanna Research assistant	F	Undergrad. student University of Oulu		AoF

Total person-months of work conducted by the research team 155
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2005	M.Sc.	F	Jalkanen, Hanna, 1979	University of Jyväskylä	Elina Vapaavuori, Seija Kaakinen, Metla
2005	DI	F	Warsta, Elina, 1981	Technical University of Helsinki	Seija Kaakinen, Pekka Saranpää, Elina Vapaavuori, Metla
2006	M.Sc.	F	Pohjanen, Johanna, 1978	University of Oulu	Elina Vapaavuori, Seija Kaakinen, Metla
2007	Ph.D.	F	Kostiainen, Katri, 1977	University of Oulu	Seija Kaakinen, Pekka Saranpää, Elina Vapaavuori, Metla, Hely Häggman, University of Oulu

Abstract

The project is a consortium between Vantaa and Suonenjoki Research Units of the Finnish Forest Research Institute (Metla) and SLU, Southern Swedish Forest Research Centre in Alnarp. This collaboration investigated the impact of forest management and climate on tree growth and wood properties of the boreal forest trees, Norway spruce, birch, aspen, and maple.

Thinning programs with few and heavy thinnings produce large sized logs with the possibility to saw planks far away from the centre of the log, which gives better form stability. In contrast, many, light thinnings give logs with smaller knot-size, especially for top-logs. Supplementary planting of gaps reduces the size of the edge-trees and improves wood quality.

Fertilisation affects wood properties by decreasing wood density and, depending on the type of fertilisation or growing site, may also decrease fibre length. Without improved nutrient availability one cannot expect a "CO_2 fertilisation effect" in the coniferous boreal forests.

Effects of elevated CO_2 and elevated O_3 on wood structure and chemistry depended on tree species and the duration of experiment. Our results show that wood properties of Norway spruce are slightly altered under elevated CO_2 and temperature.

Tiivistelmä

Projekti on Metsäntutkimuslaitoksen (Metla) Vantaan ja Suonenjoen yksiköiden ja Ruotsin maatalousyliopiston (SLU) Alnarpin Etelä-Ruotsin Metsäntutkimuskeskuksen välinen konsortio. Konsortion tutkimusaiheena oli metsänkäsittelyn ja ilmaston vaikutus boreaalisten metsäpuiden (kuusi, koivu, haapa ja vaahtera) kasvuun ja puun ominaisuuksiin.

Harvennusohjelmissa voimakas harvennus suurentaa tukkikokoa. Tämä mahdollistaa lankkujen sahauksen kauempaa ytimestä, mikä parantaa muotopysyvyyttä. Lievemmissä harvennuksissa erityisesti latvatukkien oksankoko pienenee. Aukkojen täydennysistutus pienentää reunapuiden kokoa ja parantaa näin puun laatua.

Lannoitus vaikuttaa puun ominaisuuksiin alentamalla puuaineen tiheyttä. Lannoitus voi myös lyhentää kuidun pituutta riippuen lannoituskäsittelystä ja kasvupaikasta. Kohoava hiilidioksidipitoisuus ei todennäköisesti kiihota puiden kasvua boreaalisessa havumetsässä ilman lannoitusta.

Kohotetun hiilidioksidin ja otsonin vaikutukset puun rakenteeseen ja kemiaan riippuvat puulajista ja altistuksen kestosta. Tuloksemme osoittavat että kohonnut hiilidioksidipitoisuus ja lämpötila muuttavat vai hieman kuusen puun rakennetta ja kemiallista koostumusta.

Sammanfattning

Projektet är ett samarbete mellan Vantaa och Suonenjoki forskningstationer, Skogsforskninginstitutet (Metla)- Finland och Sveriges lantbruksuniversitet (SLU), Institutionen för sydsvensk skogsvetenskap i Alnarp. Samarbetet har utforskat inverkan av skogsskötsel och klimat på trädtillväxt och vedegenskaper i boreala skogar av gran, björk, asp och lönn.

Få och hårda gallringar producerar stora stockar. Det ger möjlighet att såga plankor långt ifrån märg. De här plankorna får högre formstabilitet än plankor som sågats nära märgen. Fördelen med många och lätta gallringar är att de producerar stockar med små kvistar, särskilt i toppstockarna. Hjälpplantering i luckiga föryngringar minskar trädstorleken för kantträden och förbättrar därmed vedkvaliteten.

Gödsling påverkar vedens egenskaper. Veddensiteten minskar och beroende av typ av gödsling och bonitet kan även fiberlängden minska. Ingen ”CO_2- gödslingseffekt” kan förväntas i de boreala skogarna om näringstillgången inte samtidigt förbättras genom gödsling.

Effekter av hög CO_2 och hög O_3 på vedstruktur och vedens kemiska sammansättning är beroende av trädslag och försökens längd. Resultaten visar att vedegenskaper i gran förändras marginellt vid hög CO_2 och hög temperatur.

4.1 Introduction

4.1.1 Background

A possible future trend in the Nordic countries is diversified forestry, where wood production is mainly done in plantations that are managed according to intensive silvicultural practices (e.g. fertilisation, intensive regeneration, end-product oriented thinning programmes) whereas other areas are set aside for nature conservation. Today, increased growth rate of individual trees is thought to be mainly negative for pulpwood and timber quality, but this may not be true for all end-users of the material.

4.1.2 Objectives

Hypotheses of the consortium are:

1. Growth rate affects the wood and fibre properties of Norway spruce in a similar manner despite factors that determine the growth rate (e.g. fertilisation, thinning).
2. In the future, climate change will influence the growth patterns and growth rate of forest trees which will have an impact on wood and fibre properties.

4.2 Results and discussion

Effects of climate and nutrition on partitioning of biomass

The coarse-roots from the 15 trees that were harvested in Asa in October 2003 were excavated during the summer 2004. The analysis of the data has, however, been delayed since the Ph.D. student in charge, Thomas Severinsson, was on leave without pay during 2005. The final analysis is now on its way and the data will be combined with the similar material from Flakaliden to test if it is possible to derive generic allometric relationships for Norway spruce. The results from Flakaliden have shown that nutrient availability did not affect the relative partitioning of biomass between aboveground and belowground structures (Fig. 1; Iivonen et al. 2006). There is, however, an effect of fertilisation in terms of a reduction in soil respiration and an increase in accumulation of soil carbon (cf. Freeman et al. 2005; Högberg et al. 2005; Olsson et al. 2005).

The thinning that was carried out at Flakaliden during winter 2003/2004 was planned together with sub-project 2 and contained two thinning regimes, 30 and 60% of stand basal area. This reduced the leaf area index (LAI) in the strongest thinning treatment (60%) to the same level as in the non-fertilised control and irrigated stands. This gives a unique opportunity to study the effect of nutrient availability on growth and biomass allocation, at initially similar LAIs. The initial response in terms of tree growth (basal area and height) and LAI development was measured in October 2005, but another couple of years are needed before a fi-

nal analysis of the response to the treatments can be evaluated.

The analyses of the effects of elevated air temperature and/or [CO_2], in the Whole-Tree Chamber (WTC) experiments have continued and shown that there was no effect of elevated [CO_2] on phenology or growth (Slaney et al. 2007), which means that without improved nutrient availability one cannot expect a "CO_2-fertilisation effect" in the coniferous boreal forests. There was, however, a significant increase in soil respiration in WTCs with elevated [CO_2] (Comstedt et al. 2007). Elevated temperature resulted in earlier bud break, but did not affect the final shoot length (Slaney et al. 2007). The results were also used in testing a number of ecophysiological models of bud burst, which clearly indicated that none of the tested models can accurately predict bud burst of Norway spruce in a future warmer climate (Hänninen et al. 2007). The results from the manipulation experiments at Flakaliden have over the last year also been used in modelling projects to estimate the production potential of Norway spruce in Sweden (Bergh et al. 2005), the carbon balance of coniferous forests (Medlyn et al. 2005), and the likely impact of climate change on the carbon balance of the boreal forests (Eliasson et al. 2005; Pepper et al. 2007).

Effects of silviculture on wood quality

Preliminary results from experiment 1 indicate that supplementary planting will not increase yield in the stand. However, supplementary planting may be a worth-while silvicultural method if wood-quality of trees at the edge of gaps is significantly improved. Our data show that supplementary planting reduces the size of the edge-trees and the effect on wood-quality is substantial.

Results from experiment 2 showed that the stands in the densest spacing produce a higher volume. Trees from dense spaced stands have smaller thickest branch compared to trees of the same size from wider spacing (Figure 1). No differences have been found between spacings in regard to the presence of spike knots and other defects. The experiment also showed that the quality of the stands from low-density plantations can be improved by thinning from above in the first thinning which removes large, bad-quality trees. Thinning from above will also increase net income from the first thinning since large trees are removed which will decrease the cost for harvest and skidding. Results and analysis from this experiment has been reported in an article that has been sent for possible publication in Scandinavian Journal of Forest Science.

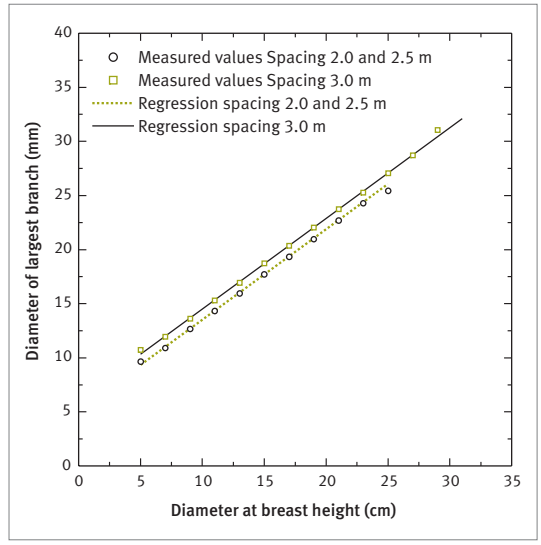

Figure 1. Diameter of largest branch in three whorls around breast height for trees with various diameters at breast height. In the figure, a regression model is indicated which has diameter at breast height and an indicator variable for 3.0 m spacing as independent variables.

In experiment 3, the effects of thinning strategies on wood quality are investigated. Logs from four different thinning experiment has been transported to a saw-mill for test-sawing. The sawn material has been characterized with respect to form-stability and other quality measures such as knot-size and compression wood. Preliminary analysis shows that the thinning program has indeed a large impact on wood-quality. Thinning programs with few and heavy thinnings produce large sized logs with the possibility to extract planks far away from the centre of the log which gives better form stability. In contrast, many, light thinnings give logs with smaller knot-size, especially for top-logs. This experiment will be analyzed during the spring of 2007.

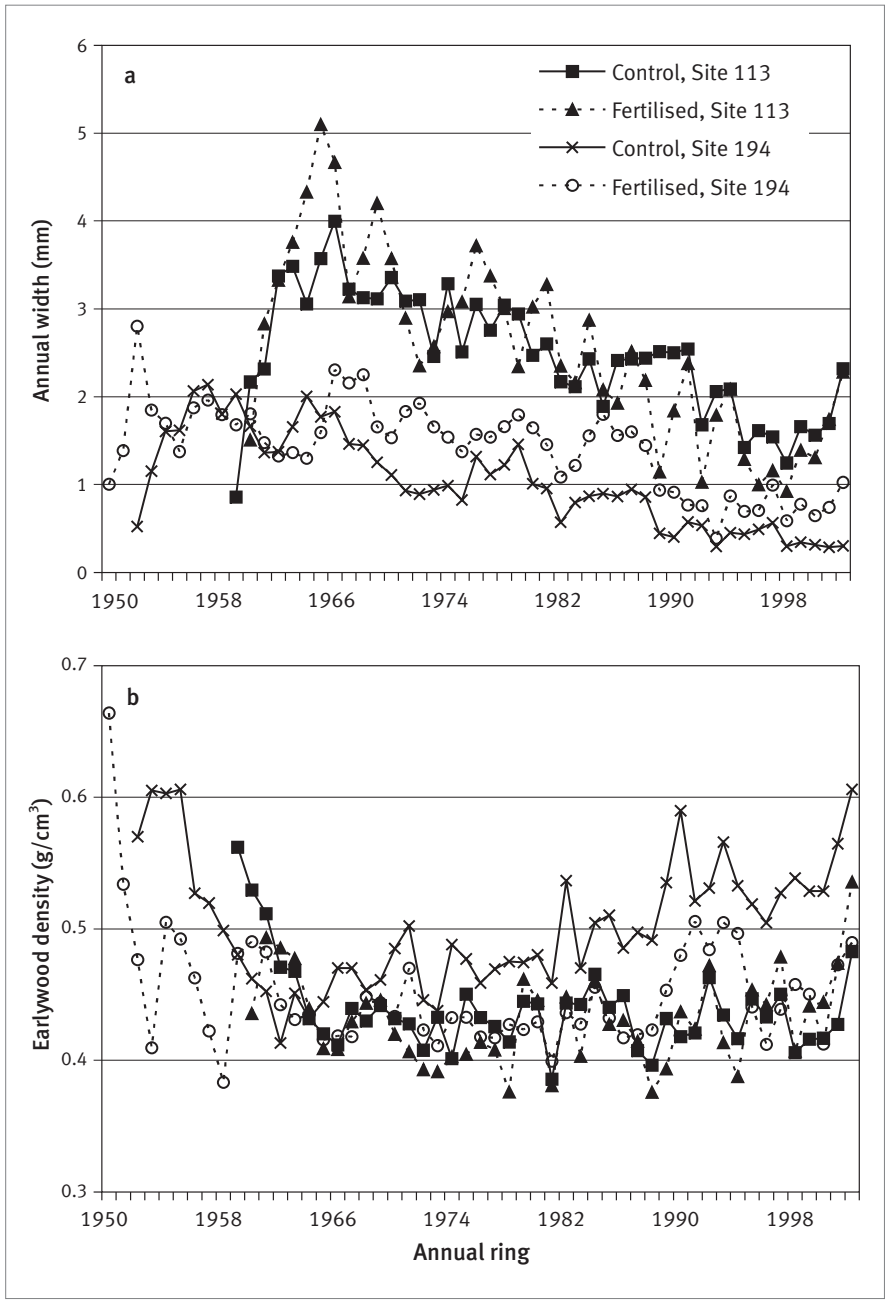

Figure 2. Annual ring width (a) and earlywood density (b) in Norway spruce from southern Finland (Heinola 113) and northern Finland (Kemijärvi 194). Solid N-fertilisation at five year intervals began in Heinola and Kemijärvi in 1961 and 1965, respectively.

Wood structure and fibre properties

Effect of fertilisation on fibre properties

In the nutrient optimisation experiment in Asa fibre length, annual ring width, and lignin concentration were studied were examined jointly with sub-projects 1,2 and 4. FTIR-spectras were used to estimate the concentration of lignin ring by ring with the help of principal component analysis based on Norway spruce samples of another study with known Klason lignin and soluble lignin concentrations (Raiskila et al. 2007). The diameter growth of the trees was doubled in fertilised trees, which also had wider annual rings than the untreated control trees ($p < 0.01$), except the last five years of the experiment when the annual ring width of the IL-trees was smaller due to increased volume of the IL-trees. The IL-trees had the similar fibre length than the untreated control trees, even though trees had increased their volume production significantly. However, the IL-trees had a slightly larger earlywood lignin concentration than the untreated controls. When compared to the previous results of the nutrient optimisation experiment in Flakaliden (Anttonen et al. 2002, Mäkinen et al. 2002), one would have expected that faster growth would have a more distinct effect on earlywood fibre length and lignin concentration than was actually found in this study.

Kemijärvi and Heinola experiments

Fertilisation increased significantly annual ring width in Kemijärvi ($p < 0.01$), but had no effect in Heinola (Figure 2a). Annual ring width was larger in Heinola than in Kemijärvi, although towards the end of the experiment growth rate decreased to the same level on both experimental sites (Fig. 2a). Fertilisation decreased slightly earlywood density ($p < 0.05$), latewood density (data not shown) as well as tracheid length ($p < 0.05$) in Kemijärvi, while in Heinola no effects of fertilisation on wood properties were seen (Fig. 2b). Tracheids were longer in the southern experimental site, Heinola, than in the northern experimental site, Kemijärvi (Fig. 3). The results for wood properties were consistent in relation to the growth rate of the trees at both experimental sites.

A new rapid method was developed to study tracheid transverse radial diameter in Norway spruce wood. The method is based on laser diffraction and requires a round coherent laser beam projected with the help of beam adjusting lenses

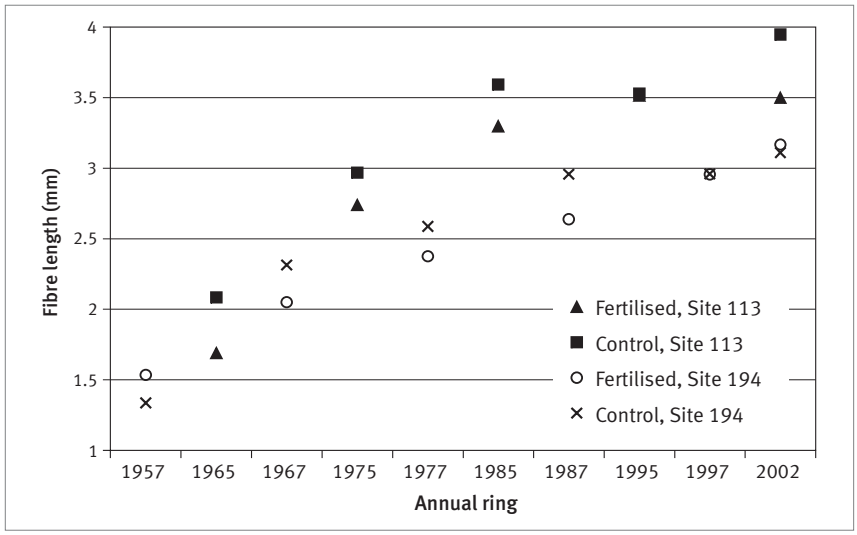

Figure 3. Fibre length at ten and five year intervals in Norway spruce from southern Finland (Heinola 113) and northern Finland (Kemijärvi 194). Solid N-fertilisation at five year intervals began in Heinola and Kemijärvi in 1961 and 1965, respectively.

through the transverse wood section (14-µm thick) and captured with the CCD-camera connected to PC-equipment (Sarén et al. 2007). The laser diffraction based method was verified with conventional microscopy of parallel samples selected from Kemijärvi and Heinola fertilisation experiment.

Effects of fertilisation and thinning on biomass and wood chemistry

Fertilisation effects on biomass

In collaboration with sub-project 3, two nitrogen fertilisation experiments, one in Kemijärvi (northern Finland) and the other in Heinola (southern Finland) were used to study the fertilisation effects on biomass production and allocation. Fertilisation increased biomass only in northern Finland but not in southern Finland, probably due to high site fertility and nutrient imbalance (Kaakinen et al., submitted). Fertilisation and location (North vs. South) affected the ratio of biomass to stem sapwood area at crown base. Fertilisation increased foliage mass with more than 300 kg m-2 of sapwood. Due to northern location the foliage mass decreased by 800 kg m-2 of sapwood. The estimates for root and branch masses per unit sapwood area were 700 kg and 1000 kg less, respectively, for Kemijärvi than Heinola. Fertilisation and location did not influence the ratios of foliage, root and branch biomass either to stem biomass or to stem surface area in Norway spruce. Further, foliage, branch and root biomasses regressed on stem surface area had constant slopes (Kaakinen et al. 2007, submitted manuscript) irrespective of fertilisation and location. This suggests that biomass estimates depend similarly on stem surface area, although the environmental conditions vary markedly. This result could offer a new and useful tool in biomass estimation of Norway spruce.

In collaboration with sub-project 1, trees from the Flakaliden nutrient optimisation experiment were examined for biomass and allocation. The results (Iivonen et al. 2006) showed that the liquid fertilisation treatment (IL) increased biomass markedly, with approximately 2.5-times more biomass than the control trees, but did not change biomass allocation between the tree compartments. Total dry mass of dominant IL-trees was 77.9 kg (± 13.5 kg) and that of control trees 30.2 kg (± 1.8 kg). The results clearly show that there is a large potential for carbon sequestration and to increase biomass production of Norway spruce. The growth increase due to nutrient optimisation in Flakaliden was considerably higher than the increase that was obtained after repeated nitrogen fertilisations in Kemijärvi. This indicates that not only N, but also other nutrient elements are required to approach the yield potential of Norway spruce.

Fertilisation effects on wood chemistry

Data from the above-described Kemijärvi and Heinola experiments (collaboration with sub-project 3) show that the effect of nitrogen fertilisation on wood chemistry was relatively small but differed between locations (Kaakinen et al. 2007). The cellulose and carbon concentrations were higher in Heinola than in Kemijärvi while the concentrations of extractives, uronic acids, starch, acid-soluble lignin and nitrogen were higher in Kemijärvi than in Heinola . In Kemijärvi, N-fertilisation induced almost a 1.5-fold increase in annual radial increment and an increase in concentration of nitrogen in stem wood. In Heinola, N-fertilisation did not affect annual radial increment, but the concentration of extractives was increased in stem wood. Distance from the pith affected wood chemistry. The concentrations of acid-soluble lignin, nitrogen, extractives and soluble sugars increased with the distance from the pith, .ie. from juvenile to mature wood. On the contrary, gravimetric lignin and uronic acids decreased along with distance from the pith. In addition in Heinola, cellulose increased and carbon decreased with the distance from the pith, while such effect was not significant in Kemijärvi.

Materials from the nutrient optimisation experiment (IL) in Asa were examined in collaboration with sub-projects 1, 2 and 3. The IL-treatment induced an increase in the concentration of nitrogen in wood (Fig. 4c) and consequently, decreased the ratio of carbon to nitrogen (Fig. 4g).The IL-treatment decreased the starch concentration in wood (Fig. 4j).The IL-treatment increased the gravimetric lignin concentration at the height of 1.3 m, and also at the height of 6 m the increase was almost significant (Fig 4b) In addition, carbon concentrations increased in IL-trees at heights of 1.3 m, 4 m and 6 m (Fig 4e).

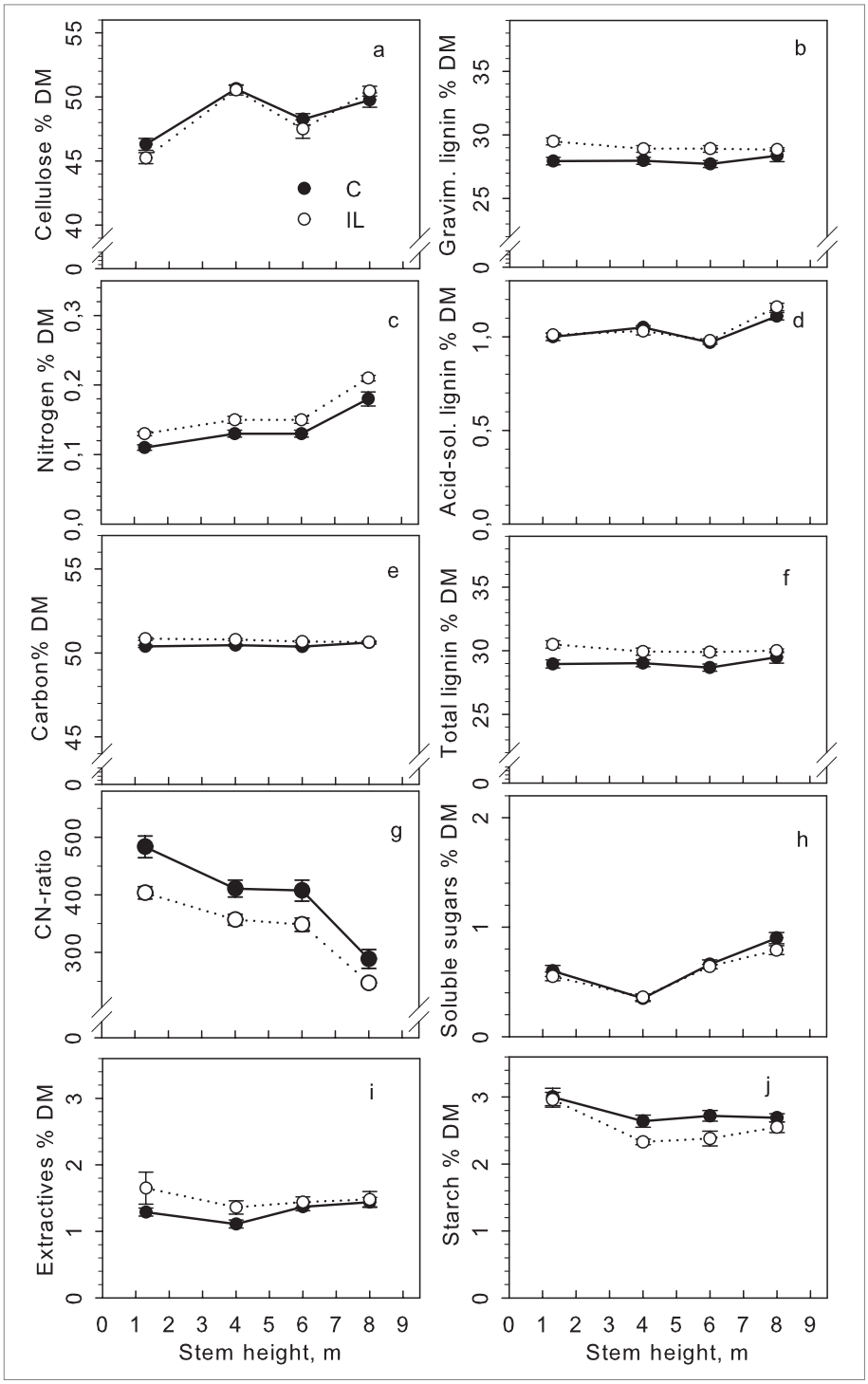

Figure 4. Concentrations of wood chemical components (% DW) and the C/N- ratio as a function of stem height in Norway spruce from Asa nutrient optimisation experiment in Sweden. Values are means ± SE of nine trees in each treatment: control (C) and nutrient optimisation (IL).

Fertilisation and thinning effects on wood chemistry

The effect of thinning and fertilisation on Norway spruce wood properties was studied in Finnish (Suonenjoki and Parikkala) and Swedish (Herrevadskloster) experiments (collaboration with sub-projects 2 and 3). Preliminary results from the Suonenjoki experiment (data not shown) suggest that the gravimetric lignin concentrations were not affected by the treatments. This is different from the nutrient optimisation treatment where a striking increase in growth was connected with an increase in gravimetric lignin concentration of stem wood. Analyses of results from Parikkala and Herrevadskloster experiments are under progress.

Results of climate change studies

The wood materials for the climate change studies were obtained from exposure studies in Rhinelander (USA, collaboration), Suonenjoki (Finland, sub-project4) and Flakaliden (Sweden, sub-project 1) (Tables 1-2). Data from the Rhinelander study (aspen clones, paper birch) showed that the effect of genetic variation on wood chemistry and structure was significant in aspen. After five years of exposure to elevated CO_2 and O_3 (Tables 2-3), the responses of wood properties were changed especially in stem wood chemistry compared to results after three years of fumigation (Kaakinen et al. 2004). Exposure to elevated CO_2 increased stem diameter and decreased uronic acids (in hemi-

Table 1. Effects of carbon dioxide and ozone on chemistry of wood in trembling aspen and paper birch after 5-year exposure, and in silver birch after 3-year exposure, and effects of carbon dioxide and temperature (T) on chemistry of wood in Norway spruce after 3-year exposure. Symbols indicate ↑ increase, ↓ decrease, ± no change, due to the treatments.

Experiment	Species	Cellulose	Klason lignin	Uronic acids	Extractives	Soluble sugars	Starch	Nitrogen	Cellulose/ lignin
FACE 2002	Aspen	±	±	CO_2↓	O_3↓	±	±	±	±
	Paper birch	±	±	±	CO_2↑ O_3↑	±	CO_2↓ O_3↑	±	±
OTC 2001	Silver birch	CO_2↓	CO_2↓	±	CO_2↑	±	CO_2↑	O_3↓ (cl 4)	±
Flakaliden 2004	Norway spruce	±	±	±	T↓	T↓	±	±	±

Table 2. Effects of carbon dioxide and ozone on structure of wood in trembling aspen after 5-year exposure, and in silver birch after 3-year exposure, and effects of carbon dioxide and temperature (T) on structure of wood in Norway spruce after 3-year exposure. Symbols indicate ↑ increase, ↓ decrease, ± no change, due to the treatments, n.a. not analysed, ew earlywood, lw latewood. .

Experiment	Species	Radial growth	Annual ring width	Fibre lumen diameter	Vessel lumen diameter	Vessel %	Cell wall %	Cell wall thickness	Wood density
FACE 2002	Aspen	CO_2↑	n.a.	±	O_3↓	±	±	n.a.	n.a.
OTC 2001	Silver birch	n.a.	CO_2↑	n.a.	±	O_3↓ (cl 80)	O_3↑ (cl 80)	n.a.	n.a.
Flakaliden 2004	Norway spruce	n.a.	±	CO_2↑ (lw)			n.a.	T↑ CO_2↓(ew)	T↑

cellulose) in aspen. In paper birch, an increase in extractives and a decrease of starch were found. Under elevated O_3 exposure, decrease of extractives and vessel lumen diameter were found in aspen, while in paper birch, concentrations of extractives and starch were increased. No changes were found in total lignin in either of the species, which is in contrast to the results of the three-year-exposure (Kaakinen et al. 2004).

Data from Suonenjoki study with 10-year-old silver birch showed that elevated CO_2, elevated O_3 and genetic factors affected the wood characteristics (Tables 2-3) (Kostiainen et al. 2006). Elevated CO_2 elicited increases in extractives and starch and decreases in cellulose and lignin. Elevated O_3 decreased nitrogen in one clone (clone 4). There were no significant interactions with ozone and carbon dioxide in stem wood chemistry. Elevated CO_2 increased wood formation, seen as increased annual ring width. Elevated ozone decreased vessel percentage and increased cell wall percentage in one clone (clone 80). Stem wood structure varied between the two clones. Clone 4 had longer fibres, greater vessel lumen diameter and vessel percentage than clone 80, while in clone 80 the cell wall percentage was greater.

Responses of 40-year-old Norway spruce to elevated temperature and CO_2 were examined in Flakaliden study. Elevated temperature decreased concentrations of extractives and soluble sugars while the main cell wall components (lignin and cellulose) were not affected. Elevated CO_2 had no effect on stem wood chemistry while our previous data on Norway spruce (Kostiainen et al. 2004) showed changes in concentrations of soluble sugars and N under CO_2 exposure. Both elevated temperature and CO_2 affected wood structure. Elevated temperature increased wood density and cell wall thickness. Elevated CO2 decreased cell wall thickness in earlywood and increased tracheid lumen diameter in latewood.

4.3 Conclusions

Growth of Norway spruce in boreal forests is limited by nutrient acquisition. Stem wood diameter growth can be increased markedly with fertilisation, the nutrient optimisation treatments giving greatest growth increments. Nitrogen fertilisation alone may lead to nutrient imbalance with no growth increments (Heinola experiment). The nutrient optimisation data suggest that fertilisation has no effect on the relative partitioning of biomass between aboveground and belowground structures. Fertilisation affects wood structure by decreasing wood density and, depending on the type of fertilisation or growing site, may also decrease fibre length. Optimal fertilisation increases lignin concentration of wood, but this is not seen with nitrogen fertilisation only. Wood properties are also affected by the site, with distinct differences in wood chemical composition between the northern and southern locations.

Data from the climate change studies show that in deciduous trees (aspen, birch), elevated CO_2 increases stem diameter growth, and depending on species, it also affects wood properties, mainly stem wood chemistry. Elevated O_3 affects mainly wood structure with increasing cell wall percentage and decreasing vessel percentage. From the Whole-Tree Chamber experiments in Flakaliden it can be concluded that without improved nutrient availability one cannot expect a "CO_2-fertilisation effect" in the coniferous boreal forests.

In Norway spruce, elevated CO_2 induces changes in anatomical and physical properties of wood. Under elevated temperature treatment, with temperatures projected to year 2100, the concentrations of extractives and soluble sugars decrease and denser wood is produced with parallel changes in wood anatomy. Our results show that wood material properties are slightly altered under elevated CO_2 and temperature.

4.4a Capabilities generated by the project

The consortium served as a capacity-building platform for two post doc researchers, three Ph.D. students and five M.Sc. students. Altogether 22 referred scientific publications and 19 manuscripts/other publications were produced by the research team. New methodological approaches were developd for measurement of lignin (FTIR method) and tracheid transverse radial diameter (laser diffraction method).

4.4b Utilisation of results

Results have been presented in several national international seminars. A Wood Wisdom Material Science Programme -seminar was organised in November 30, 2006 in Helsinki (Puun ominaisuudet ja käyttömahdollisuudet tulevaisuudessa) and two excursions about Intensive Forest Management in Flakaliden in June 19, 2006 and in Asa June 21, 2006 (Intensivodling – är det lösningen?) for foresters and decision makers. Advisory group has played a central role in transferring the results into practice.

4.5 Publications and communication

a) Scientific publications

1. Articles in international scientific journals with referee practice

Bergh J, Linder S, Bergström J 2005: Potential production for Norway spruce in Sweden. *Forest Ecology and Management* 204: 1-10.

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Hänninen H, Slaney M, Linder S 2007: Dormancy release of Norway spruce under climatic warming: Testing ecophysiological models of bud burst with a whole-tree chamber experiment. *Tree Physiology* (Submitted)

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Kaakinen S, Piispanen R, Raiskila S, Lehto S, Pohjanen J, Nilsson U, Linder S, Saranpää P, Vapaavuori E 2007: The effect of long-term nutrient optimisation on Norway spruce (Picea abies [L.] Karst.) wood chemistry and fibre length. Manuscript to be submitted to *Can. J. For. Res.* 8/2007.

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Peura M, Müller, M., Serimaa, R., Vainio. U., Sarén, M., Saranpää, P., Burghammer, M. 2005. Structural studies of single wood cell walls by synchrotron X-ray microdiffraction and polarised light microscopy. *Nuclear Instruments and Methods in Physics Research B* 238: 16-20.

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3. Articles in Finnish/Swedish journals with referee practice

4. Articles in Finnish/Swedish scientific compilation works and Finnish/Swedish scientific conference proceedings with referee practice

5. Scientific monographs

Jalkanen H 2005: Effects of elevated CO_2 and O_3 concentrations on wood chemistry of silver birch (*Betula pendula* Roth). University of Jyväskylä, Department of Environmental Science. Master's thesis, 48 p.

Lehto S 2005. Lannoituksen vaikutus kuusen trakeidin pituuteen ja ligniinipitoisuuteen (in finnish). University of Helsinki, Department of Forest Resource Management. Master's thesis, 67 p.

Pohjanen J 2006. Ravinneoptimoinnin vaikutus kuusen (*Picea abies*) runkopuun kemialliseen koostumukseen (in finnish). University of Oulu. Department of Biology. Master's thesis, 52 p.

Warsta E 2005: Effects of elevated carbon dioxide and ozone concentrations on wood sstructure and chemistry of Trembling aspen (*Populus tremuloides*). Helsinki University of Technology, Department of Forest Products Technology. Master's thesis, 72 p.

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Freeman M, Morén A-S, Strömgren M, Linder S 2005: Chapter 3: Climate change impacts on forests in Europe: Biological impact mechanisms. In: Kellomäki S, Leinonen S (eds) *Management of European Forests Under Changing Climatic Conditions*. Research Notes 163, University of Joensuu, Forest Faculty, pp. 46-115.

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b) Publishing and dissemination of information outside the scientific community

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4.6 National and international cooperation

Advisory group

Friberg, Ragnar, StoraEnso
Vehniäinen, Annikki, KCL
Lehto, Jouko, UPM-Kymmene
Lundén, Jan-Åke, Södra Skogsägarna
Marntel, Tor, VMR
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Exchange of material and methods, joint papers.

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Raman-microscopy, a joint paper.

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X-ray diffraction studies, ultrasonic testing, joint papers.

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Exchange of materials, joint papers.

5 Specific wood and timber properties, competitive ability and advanced conversion of Nordic Scots pine in mechanical wood processing (SPWT)

FINAL REPORT	
Name of the research project	**Specific wood and timber properties, competitive ability and advanced conversion of Nordic Scots pine in mechanical wood processing**
Coordinator of the project	Prof. Erkki Verkasalo

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**Competitive properties of Scots pine from Nordic countries in mechanical wood processing for advanced joinery, interior and furniture products**
Project period	1.1.2003–31.12.2006
Organization in charge of research	Finnish Forest Research Institute, Joensuu Research Centre
Sub-project leader	Prof. Erkki Verkasalo
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URL of the project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	884 898
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	167 550
Finnish Ministry of Agriculture and Forestry	15 060
Other public funding, please specify:	
Graduate School of Forest Sciences (supporting funding)	32 288
ERASMUS Trainee Programme	3 000
Other public funding, please specify:	
Own funding, Metla	667 000

RESEARCH TEAM

Name, degree, job title [1)]	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Erkki Verkasalo, D.Sc. (For.), professor, co-coordinator of consortium, sub-project leader	M	Finnish Forest Research Institute, Joensuu Research Unit		Finnish Forest Research Institute
Mika Grekin (former: Riekkinen), M.Sc. (For.), research scientist, Ph.D. student,	M	Finnish Forest Research Institute (1.1.2003-31.12.2005) University of Joensuu, Graduate School of Forest Sciences (1.1.2006-31.12.2008)		Academy of Finland, Graduate School of Forest Sciences
Seppo Nevalainen, M.Sc. (For.), research scientist	M	Finnish Forest Research Institute, Joensuu Research Unit		Finnish Forest Research Institute
Thibaud Surini, B.Sc. (Eng.), research assistant	M	Finnish Forest Research Institute, Joensuu Research Unit	France, Ecole Superieure du Bois, Nantes	ERASMUS Trainee Programme
Total person-months of work conducted by the research team			71.3	
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Antti Lukkarinen, M.Sc. (For.), research scientist	M	Finnish Forest Research Institute, Rovaniemi Research Unit		T&E Centre for Lapland, Finland (collaboration funding)
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DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2008	D.Sc.	M	Mika Grekin, 1976	University of Joensuu	Erkki Verkasalo, Finnish Forest Research Institute Matti Kärkkäinen, University of Joensuu

Name of the sub-project 2	**Competitive properties of Scots pine from Nordic countries in mechanical wood processing for advanced structural products**
Project period	1.1.2003–31.12.2006
Organization in charge of research	SLU The Swedish University of Agriculture Sciences, Department of Forest Products and Markets
Sub-project leader	Prof., Dean Mats Nylinder
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URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	206 200
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Ministry of Agriculture and Forestry	80 000
Other public funding	
Setra Group Ltd., Sweden	45 000
Own funding, SLU	81 200

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Total person-months of work conducted by the research team			32.5	

Name of the sub-project 3	**Potential of specific wood and timber properties of Scots pine in marketing planning of Nordic wood industry companies**
Project period	1.1.2003–31.12.2006
Organization in charge of research	SLU The Swedish University of Agriculture Sciences, Department of Forest Products and Markets
Sub-project leader	Assoc. Prof. Anders Roos
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URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	364 095
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Ministry of Agriculture and Forestry	265 150
Other public funding	
Business Know-How Research Programme (Academy of Finland, supporting funding)	17 352
Graduate School of Forest Sciences / University of Helsinki (supporting funding)	22 593
Other funding, please specify:	
Wood Focus Ltd., Finland	50 000
Foundation "Puumiesten ammattikasvatussäätiö"	9 000
Own funding, SLU	30 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Heikki Juslin, D.Sc. (For.), professor, scientific supervisor,	M	University of Helsinki, Department of Forest Economics	University of Helsinki
Roger Naylor, M.Sc. (For.), research scientist, PhD student	M	University of Helsinki, Department of Forest Economics (1.2.2003-30.11.2004)	Ministry of Agriculture and Forestry, Wood Focus Ltd.
Pasi Paldanius, M.Sc. (For.), research scientist, Ph.D. student	M	University of Helsinki, Department of Forest Economics (1.12.2004-31.12.2005)	Ministry of Agriculture and Forestry, Wood Focus Ltd.
Lei Wang, M.Sc. (For.), research scientist, Ph.D. student	M	University of Helsinki, Department of Forest Economics (1.1.-31.12.2006)	Business Know-How Research Programme (Academy of Finland,)
Antti Virtanen, M.Sc. (For.), research scientist, Ph.D. student	M	University of Helsinki, Department of Forest Economics (1.3.-31.12.2006)	Graduate School of Forest Sciences
Total person-months of work conducted by the research team	99.5		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2009	D.Sc.	M	Matti Stendahl, 1975	SLU The Swedish University of Agriculture Sciences	Assoc. prof. Anders Roos, SLU
2010	D.Sc.	M	Antti Virtanen, 1975	University of Helsinki	Prof. Heikki Juslin, University of Helsinki
2020	D.Sc.	M	Lei Wang, 1976	University of Helsinki	Prof. Heikki Juslin, University of Helsinki

Abstract

The primary aim of the project was to increase and systematize the knowledge of Nordic Scots pine wood and timber on the potential for improved utilization of specific wood and timber properties in the acknowledged segments of joinery, interior, and furniture products and in the growing segments of structural products to be used in marketing planning, in the first hand, but also in product development and raw material sourcing. . The final goal was a synthesis of the development and success factors in marketing planning,, product development and raw material competence in Nordic saw mill industry processing pine timber.

In sub-project 1, new data were produced on the wood properties related to visual impression, easiness for tooling and service, life expectancy and durability (weather, wear and decay resistance) of wood products, and on the affecting physical characteristics of wood (e.g., annual ring width, density, heartwood location and scope). Variation in the properties was statistically modelled at sub-geographic, within-stand and between-tree and within-tree levels, incl. site and tree stock effects and the interactions.

In sub-project 2, new data were produced on the tree, log and wood properties related to strength and stiffness, in particular, as well as form stability. Analyses of the speed of sound technique and the swept resonance technique were performed to study the basics for sorting trees and saw logs for strength (stiffness) and form stability (tendency to distortion); both techniques provide the dynamic modulus of elasticity (MOE). Destructive bending tests were performed on the flitch sample, with and without defects, to determine the effects of the defects in the prediction of MOE and to compare the flexural properties between the sub-regions.

In sub-project 3, the concept of the interface between the company and customer in pine product business was studied and developed. More specifically, the customer and market information required for new marketing designs was studied and defined emphasizing close customer contacts and superior properties of Nordic pine. Further, models for implementing customer-oriented production were studied, with particular regard to supply chain management, innovation and product development. A qualitative study on product development in the Nordic pinewood industry was conducted, and a quantitative survey investigation was carried out. Market and industry studies produced hypotheses and ideas for the above-mentioned studies. The hypotheses and ideas were tested empirically by customer survey both in the UK and Germany and an industry study was done on pine sawmills in Finland and Sweden.

Tiivistelmä

Konsortion päätavoite oli lisätä ja systematisoida tietoja pohjoismaisen männyn puutavarasta ja puuaineksesta koskien mahdollisuuksia entistä laajempaan ja kannattavampaan hyödyntämiseen perinteisesti tärkeissä rakennuspuusepän-, sisustus- ja huonekalutuotteiden segmenteissä (osahanke 1) ja merkitykseltään kasvavassa rakennustuotteiden segmentissä), ja liittää tulokset erityisesti mäntytuotteiden markkinoinnin suunnitteluun mutta myös tuote- ja teknologiakehitykseen, ja sopivien puuraaka-ainelähteiden tunnistamiseen ja arviointiin. Hankkeen lopputavoitteena oli esittää synteesi markkinoinnin suunnittelun ja tuote- ja teknologiakehityksen ja raaka-aineosaamisen menestys- ja kehitystekijöistä pohjoismaisessa mäntyä jalostavassa sahateollisuudessa.

Osahankkeessa 1 tuotettiin uutta tietoa männyn puuaineksen ominaisuuksista koskien puutuotteiden visuaalisia ominaisuuksia (esteettistä ilmettä), työstettävyyttä, asennettavuutta ja helppohoitoisuutta (ml. käyttömukavuus), odotettavissa olevaa pitkäaikaiskestävyyttä (sään-, kulutuksen- ja lahonkestävyys), ja taustalla vaikuttavia puuaineen fysikaalisia ominaisuuksia (esim. vuosilustorakenne, puuaineen tiheys, sydänpuun sijainti ja laajuus). Ominaisuuksille laadittiin deterministisiä tilastotieteellisiä ennustemalleja, joissa otettiin huomioon alueellinen sekä metsiköiden sisäinen ja runkojen välinen ja sisäinen vaihtelu, ja lisäksi taustalla vaikuttavat kasvupaikka- ja puustotekijät ja erilaiset ristivaikutukset.

Osahankkeessa 2 tuotettiin uutta tietoa männyn puu-, tukki- ja puuaineominaisuuksista koskien erityisesti puumateriaalin lujuutta ja jäykkyyttä, mutta myös muotopysyvyyttä. Rasitusaaltojen eli

äänen nopeuden mittaukseen ja rasitusaaltojen resonanssitaajuuden mittaukseen perustuvia tekniikoita tutkittiin puille ja tukeille sovellettuina lujuuden (jäykkyyden) ja muotopysyvyyden mukaisen lajittelun kehittämisen perusteiksi, molemmat tekniikat perustuvat dynaamisen kimmokertoimen laskentaan mittaustulosten perusteella. Lyhyiden saheiden otoksesta mitattiin taivutuslujuus ja -kimmokerroin rikkovalla menetelmällä sekä erilaiset viat, lähinnä oksat sisältäneistä että virheettömistä testauskappaleista vikojen vaikutusten määrittämiseksi ja taivutusominaisuuksien vertailemiseksi alueiden välillä. T

Osahankkeessa 3 tutkittiin mäntytuotteita valmistavan puutuoteyrityksen ja asiakkaan rajapintaa ja kehitettiin siihen perustuvaa konseptia. Erityisesti tutkittiin ja määriteltiin uusien markkinointiratkaisujen vaatimaa asiakas- ja markkinainformaatiota, painottaen läheisten asiakasyhteyksien ja pohjoismaisen männyn hyvien ominaisuuksien hyödyntämistä. Lisäksi tutkittiin asiakaslähtöisen tuotannon käyttöön ottamisen malleja, koskien erityisesti arvoketjujen hallintaa, innovaatiostrategioita ja -toimintaa ja tuotekehitystä. Pohjoismaisen mäntyteollisuuden tuotekehityksen tilasta tehtiin kvalitatiivinen haastattelututkimus, ja tämän jälkeen syvemmälle viety kvantitatiivinen kyselytutkimus. Markkina- ja teollisuustutkimukset tuottivat hypoteeseja ja ideoita, joita testattiin sekä asiakashaastattelututkimuksissa Iso-Britanniassa ja Saksassa että sahayrityksiä koskeneissa tutkimuksissa Suomessa ja Ruotsissa.

Sammanfattning

Projektets primära mål var att öka och systematisera kunskaper om nordisk furu och förutsättningarna för en förbättrad användning baserad på furans specifika egenskaper för både respekterade produkter i snickerier, inredning och möbler, och det växande segmentet som konstruktionsvirke. Dessa resultat kombinerades med en forskning särskilt om marknadsplanering men också om produktutveckling och rundvirkets anskaffning. I sin syntes kom projektet att urskilja intressanta utvecklingsinriktningar och framgångsfaktorer för den nordiska furuindustrins marknadsföring samt produktutveckling och råvarukompetens.

I delprojekt 1 utvecklades ny information om olika virkesegenskaper hos träprodukter som visuellt utseende, lämplighet för bearbetning och underhåll (inkl. upplevelse av komfort), livscykelegenskaper och beständighet (avseende väderpåverkan, slitning och nedbrytning), samt beträffande centrala virkesegenskaper (t.ex. årsringsbredd, densitet, kärnvedens omfattning och utsträckning). Variationerna modellerades statistiskt (deterministiskt) på träd- och beståndsnivå, och för de olika geografiska regionerna med beaktande av ståndorts- och interaktionseffekter.

I delprojekt 2 genererades nya uppgifter om träd-, stock- och virkesegenskaperna styvhet och styrka, men även formstabilitet. Akustiska metoder (“speed of sound technique” och “swept resonance technique”) användes för att studera möjligheterna att tillämpa metoderna för att sortera träd och stockar med avseende på styrka, styvhet och formstabilitet. Bägge metoderna kan även användas för att förutspå en dynamisk elasticitetsmodul (MOE). Destruktiva böjningstester utfördes på korta bräder från svenska och finska stockar, med och utan virkesfel (närmast kvistar), för att bestämma inverkan av virkesfel i modellering av MOE och jämföra böjningsegenskaper mellan delregionerna. Torkning analyserades för att skapa grundförutsättningar för en automatisk sortering på sågverken.

I delprojekt 3 studerades en helhet av marknadsaspekter kring den nordiska furan. Inom delprojektets ram insamlades information om kunder och marknader med syftet att formulera en förbättrad marknadsföring av furuprodukter. En tonvikt lades vid tätare kundkontakter och en betoning av furans konkurrensfördelar för produktegenskaper. Dessutom formulerades modeller för att införa en mer kundorienterad produktion, med speciell hänsyn till förädlingskedjans förutsättningar, innovation och produktutveckling. Inom ramen för innovationsstudier genomfördes en kvalitativ studie om produktutveckling, åtföljd av en kvantitativ enkätstudie. För att testa hypoteser och ideér kring marknadsbehov och modeller för marknadsföring genomfördes empiriska marknadsstudier i Storbritannien och Tyskland och industristudier hos sågverksindustrier i Finland och Sverige.

5.1 Introduction

5.1.1 Background

Nordic sawmill industry manufacturing pine products has to position its activities and products into the current and future market segments and implement new innovation and marketing strategies to be able to compete in the international market and re-gain, or increase, the market share compared to alternative wood species and substituting materials. Especially, the specialization in further-processed, value-added products requires a better knowledge of the critical properties that should be further developed. Also the competing ability of the prevalent properties, and the key arguments to be used by the industries to target on the end customers should be taken into account. Superior wood properties are worthy in marketing only when they can be linked to preferences and images of, and commercial value to, the customers.

5.1.2 Objectives

In this project, wecombined the expertise in wood science, forest products, and forest product marketing, and linked the technological and marketing approaches. The primary aim was to increase and systematize the knowledge of Nordic Scots pine wood and timber on the potential for improved utilization of specific wood and timber properties in the acknowledged segments of joinery, interior, and furniture products (sub-project 1) and in the growing segments of structural products (sub-project 2), to be used in marketing planning, in the first hand, but also in product development and raw material sourcing (project 3). The final goal was a synthesis of the development and success factors in marketing planning,, product development and raw material competence in Nordic saw mill industry processing pine timber.

5.2 Results and discussion

In sub-project 1 new data were produced on the wood properties related to visual impression, easiness for tooling and service, life expectancy and durability (weather, wear and decay resistance), and on the affecting physical characteristics of wood (e.g., annual ring width, density, heartwood location and scope). Variation in the properties was statistically modelled at sub-geographic, within-stand and within-tree levels, incl. site and tree stock effects and the interactions. To illustrate the background of wood variation, the dimensions and external quality of the standing timber stock were compared between the sub-regions. Also, literature review was done on Nordic Scots pine versus seven competing softwood species in joinery and structural product segments, and versus the most important substituting non-wood materials in three end-use segments (windows, exterior cladding, structural products).

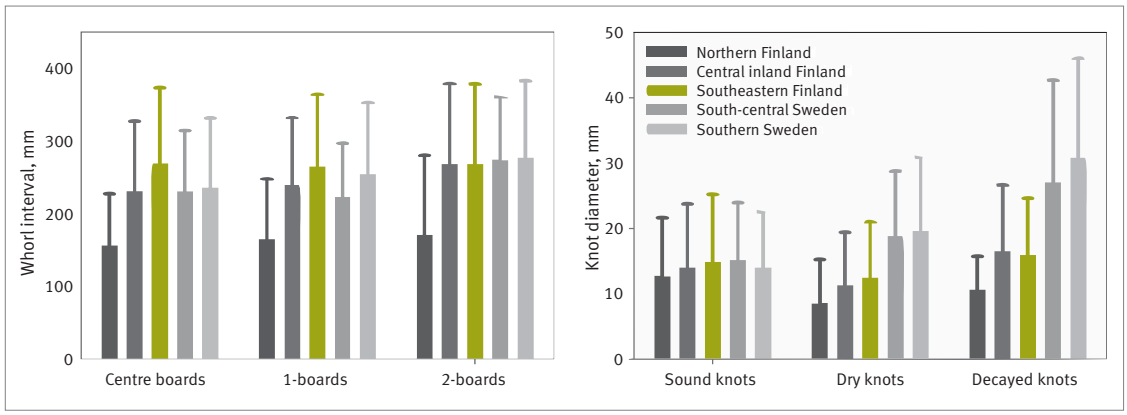


Figure 1. Selected knottiness properties in sawn timber billets of Scots pine in the five sub-regions, averages and standard deviations: whorl intervals on the surface face of through-trough sawn boards from the centre board outwards, mm (left); diameter of the thickest knot on the surface face of through-trough sawn boards by knot type (right). By Mika Grekin, METLA.

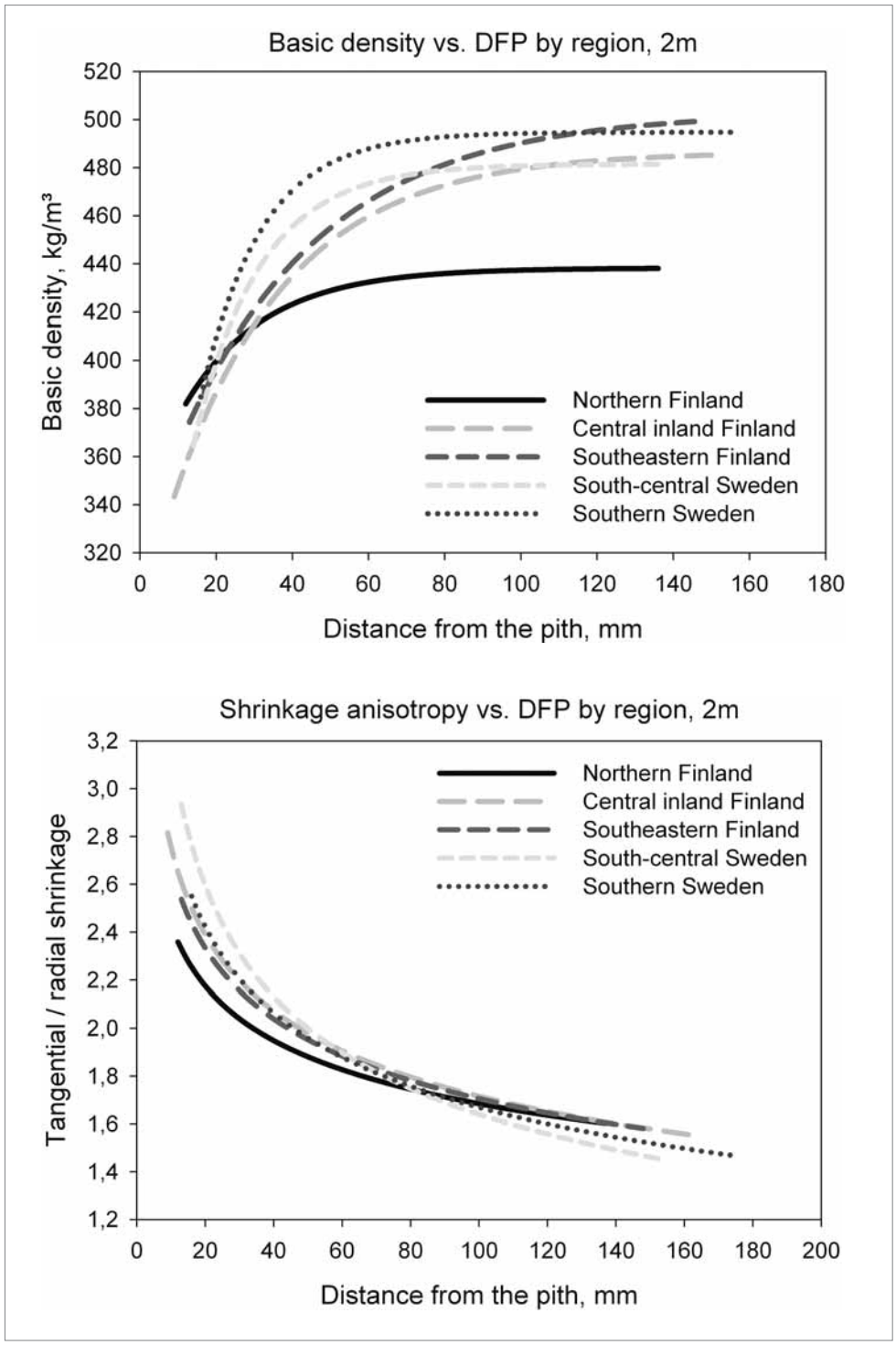


Figure 2. Selected physical properties in sawn timber billets of butt log in Scots pine in the five sub-regions: basic density (above); shrinkage anisotropy, tangential vs. radial (below), both from the centre board outwards. By Mika Grekin, METLA.

The short growth period in northern Finland and Sweden and the between-year variation in growing conditions crucially affect the growth of quality development in trees. The more northern the location, the smaller the tree dimensions and the more the external defects affect the internal wood quality. In the north, both height and diameter increment are often less than half of that in southern Swedish and Finnish, resulting in considerably smaller stems and logs, shorter sections of clear wood between the whorls (indicating lower potential for finger-jointing and gluing applications), and higher number of knots per unit height and larger percentage of dead knots of all knots, but also smaller knots (esp. dry and decayed knots), larger knot angle (more vertical),and higher heartwood percentage at given tree dimensions (Fig. 1).

In further processing of wood, the homogeneity of density and the related properties is of great importance. Due to its relatively even density, smaller difference between tangential and radial shrinkage, narrow growth rings, smaller grain deviation, and high heartwood percentage, sawn timber of northern pine is well suitable for end-uses where good ability for tooling, accuracy in product dimensions and good form stability is needed (e.g. windows, doors, decking, garden timber) (Fig. 2). This is more pronounced thanks to the small diameter and percentage of juvenile wood core.

In sub-project 2, new data were produced on the tree, log and wood properties related to strength and stiffness, in particular, as well as form stability. Analyses of the speed of sound technique and the swept resonance technique were performed to study the basics for sorting trees and saw logs for strength (stiffness) and form stability (tendency to distortion); both techniques provide the dynamic modulus of elasticity (MOE). Destructive bending tests were performed on the flitch sample, with and without defects, to determine the effects of the defects in the prediction of MOE and to compare the flexural properties between the sub-regions. Water uptake and distortion in wetting of short flitches was studied on a sub-sample of centre yield. In addition, relationships of MOE with density, strength, temperature, moisture content, knot size and swelling deformation were analyzed, aiming to the basics for developing automatic sorting equipment for saw mills.

The first sub-study showed the large variation in MOE in Scots pine at tree, log, and within-tree levels (at differing stem height & radial distance from pith). From a given stand there will be trees that yield logs with low MOE that give poor structural

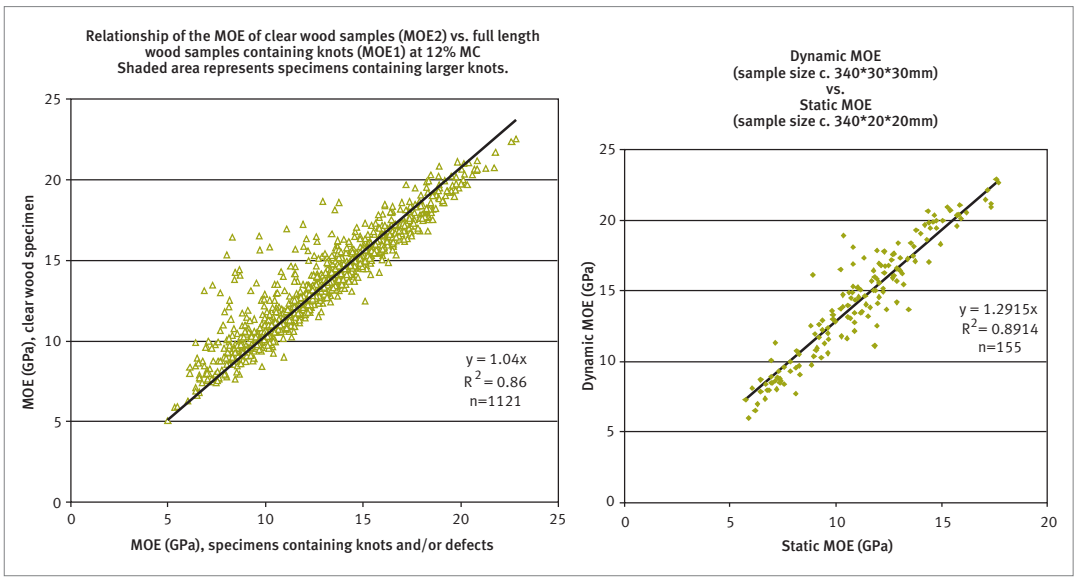


Figure 3. Dynamic MOE measured on original flitches with defects vs. flitches re-cut into clear wood samples (left) and agreement between dynamic MOE vs. static MOE representing the entire range in dynamic MOE (right). By Håkan Lindström, SLU.

lumber, whereas other trees in the same stand will yield high MOE logs that will give excellent structural lumber. According to the derived models, trees are more likely to have high MOE if they are (having): a) non-dominant in the stand, b) tall in relation to diameter (have low taper), c) high onset of visible dead knots (due to crown competition), d) large stem volume (mature older trees), e) circular stem cross-section. Still, there seems to be plenty of exceptions from the outline above which is probably due to genetic differences and complex interactions that go beyond the scope of this study.

In the second sub-study, using ca. 1120 wood specimens cut from the sample trees at three stem positions, both the MOE and the wood density increased from pith outwards. With the reference to other studies, the almost two-fold variation in MOE at given wood density could be largely explained by the difference in microfibril angle. A validation study of the relationship between dynamic and static MOE using 155 wood samples showed a good agreement ($r^2 = 0.89$) between dynamic vs. static MOE (Fig. 3). The results on both the predicted dynamic MOE and the tested static MOE indicated systematic variations with differing regions (from north to south) and within-tree location. The decreasing strength and stiffness from the south to the north could be mainly traced to the density differences, but, for knot-including wood, this difference was slightly reduced by the reverse difference in the crucial properties of knottiness.

In sub-project 3, the concept of the interface between the company and customer was studied and developed (), and strategic models were suggested for implementing customer-oriented production, with regard to innovation and product development, through a better understanding of the innovation process (). More specifically, the customer and market information required for new marketing designs was studied and defined, emphasizing close customer contacts and the role of the properties of Nordic pine. Models for implementing customer-oriented production were studied, with particular regard to supply chain management, innovation and product development. A qualitative study on product development in the Nordic pinewood industry was conducted, and a quantitative survey investigation was carried out. Market and industry studies produced hypotheses and ideas for the above-mentioned studies. The hypotheses and ideas were tested empirically by customer surveys both in the UK and Germany, and an industry study on pine sawmills in Finland and Sweden.

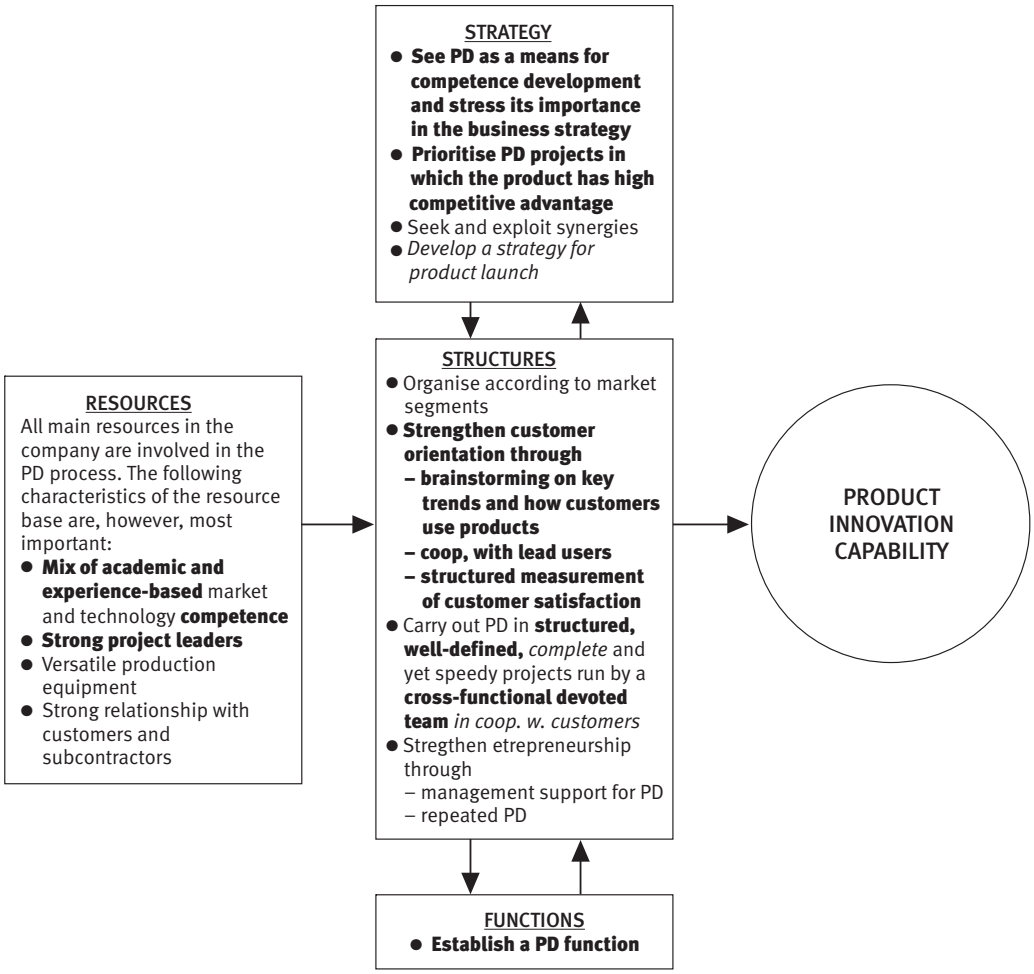
As the synthesis of all sub-studies, the following twelve propositions for future timber business are suggested:

1. Value-based philosophy has an important role in the sawmilling business in the future. Nordic companies need to develop a vocabulary of value into their common business. The best value approach focuses on the customers' needs.
2. Nordic companies support the development of integrated teams, and supply chain, to achieve maximum value and optimum performance, leading to the changes in the timber business and value adding being the objective.
3. Nordic industry increases its awareness and performance in social, environmental, and economical responsibilities.
4. Product development and innovation are essentials, and they have to be customer driven.
5. Product development is done through the integrated teams. Nordic companies should provide efficient cost information to the team members.
6. Industrial customers increasingly demand for environmentally friendly timber products. The sawmilling sector will require more sustainable design guides.
7. The future of timber marketing is about distribution restructuring and supply chain management.
8. Customer orientation leads to timber supply chain integration, and to integrated marketing communication.
9. Timber customers expect that sawmills do business with them, and understand and meet their objectives. Nordic companies build long term "business partnership" with their clients in order to add value to them. Relationship marketing is the future for timber business.
10. Nordic companies increase the presence of local availability in their business, and increase the level of their services.
11. E-Business is an exciting new environment in which to do business. Nordic companies take full advantage of these opportunities and benefits to develop their business.
12. New forms of business model enter the timber business, consisting of new forms of supply channels, partnerships and responsiveness.

Common views and opinions about product development were sought after among medium and large pine producing sawmilling companies in Sweden and Finland in the qualitative study. The high complexity of product innovation in the Nordic pine industry turned out obvious. This was mainly blamed on the divergent material flow resulting from the heterogeneous Nordic pine wood. Key factors of successful product innovation that were most frequently mentioned by the informants were:

- Strong customer relationships
- Existence of a market-oriented, innovative, 'go-ahead' company culture
- Strong support from senior management for innovation work
- Slearly defined, dynamic and fast development projects executed by a product development team with extensive and diverse knowledge
- High relatedness between current and required resources and capabilities

Insufficient knowledge about the wood material, lack of product standards, and adverse industry structure were pointed out as key hampering factors for product innovation. Typically, the solution of these challenges was stated to call for industry-wide collaboration.



Bold: **Highest priority (quantitatively validated qualitative finding)**
Normal: Medium priority (qualitative finding)
Italic: *Lowest priority (qualitative finding that could not be quantitatively validated)*

Figure 4. A business model for product innovation in wood industry. By Matti Stendahl, SLU.

According to the quantitative study, innovation is more common among larger companies and companies having further-processing activity, and slightly more common among pine-focused than spruce-focused SBUs. More innovation is also associated with the existence of product development function, customer-orientation, and, especially, the focus being on creating, adopting and implementing new products. The share of university educated personnel was higher in innovating SBUs than non-innovating SBUs; the share of exports, however, did not differ between them.

High sharpness of product concept definition, team devotion and structure and formality of projects made the difference between successful and less successful product development projects. The best third of projects that were rated high in complexity showed superiority in product advantage, strength of leadership and team cross-functionality, as well as the best third of the projects that were relatively high in product newness to SBU in sharpness of product concept definition. The top five rated obstacles for product development were: 1) product development not prioritised in the daily operations, 2) product development considered difficult and costly, 3) few ideas for new products, 4) low knowledge of customer needs, 5) raw material suppliers not open to change.

The factors associated with high innovation activity and successful product development are summarised in a business model for product innovation in the wood industry (Fig. 4). This model can be used as a guide for companies in the wood industry that want to increase their innovation activity and make their product development more effective. The data in the background will be further analysed during 2007 and 2008.

5.3 Conclusions

Technological studies of sub-projects 1 and 2 showed the large variability in Scots pine wood, which may confuse the wood processors, end-users, designers, architects and other specifiers. However, the variability provides also a rich basis for the diversity of product development. The large between-region variation in many wood properties within Nordic countries calls for focusing on specified end-uses in the product development and marketing argumentation, and embarking on selected sources of wood raw material for the intended uses. The largest potential seems to be in utilizing the high strength and stiffness, good and rather homogeneous stability and, to some extent, good durability of Nordic pine, not to forget the traditional advantages of visual beauty and good tooling ability. Linking to the expectations of the customers revealed in the qualitative and quantitative market studies, functionality, durability and safety performance during the service life, and overall environmental performance belong to the important marketing arguments related to wood and wood products. The species in itself does not seem significant, but the customers appreciate the species that they know well for the performance, availability and overall benefits vs. purchase price. Advanced construction sector seems to be the new large-scale option for Nordic pine.

The large variability in the key properties of Nordic pine calls for modern, efficient and accurate technologies in sorting raw materials and primary products, and for development of standards for measurements and products. Statistical modelling of the properties within and between trees, and between stands and regions can be used as a tool in the development of products and measurement and grading technologies, and in different benchmarking analysis. There are good possibilities to develop strategic models that could be used to select trees and logs through different grading systems for structural wood products, in particular. The derived models, using limited number of descriptive site and tree growth variables have, however, limited accuracy. This is believed to be a result of large genetic variation that cannot be accounted for by the models. Such models may be further extended and refined for the use in strategic decision support systems in forest management and timber procurement.

The results of market and industry studies provided conclusions for:

- Foresight, or points of view of future pine industry structure: a) new customer benefits to provide, b) the core competencies to be built or acquired to offer those benefits, c) proposals to reconfigure the customer interface to deliver those benefits
- Strategic marketing planning of Nordic pine wood industry companies - new marketing designs: a) new business opportunities and mod-

els, b) new marketing tools for Nordic pine wood industry, c) value propositions to meet the needs and preferences of the customers

The industry studies indicated that the enhancement of the innovation activity needs a good understanding of the complexity of the concept. However there are several important management measures available to improve and speed up innovation and product development in the company.

Based on the results, the following market-based drivers (demands) for business development may be concluded for sawmilling/wood industries:

- Sustainability: a) responsible production and consumption, b) environmental responsibility, c) social and ethical responsibility, d) economic responsibility
- New types of products: a) eco-products, b) system products, c) components and semi-finished products, d) special and custom-made sawn wood
- New types of customer interface and relationships: a) availability, b) information, c) customer service

Product development is driven by market changes, process needs and dissatisfaction with current situation, has a significant impact on competence development, and can be improved by: a) support from management for an entrepreneurial market orientation; b) establishment of a x-func team including academic and experience-based knowledge; c) well-defined and speedy projects; d) access to versatile process technology; and e) a strategy to launch. There clearly are drivers in the export markets that call for development of sawn timber marketing. The main point in marketing is not the species, and the only point is not the product; however, better understanding of the strengths and weaknesses, their variation and controlling options (clarified in this project) contributes to positioning into the markets and segmentation into product and customer groups. The entire value proposition (raw materials, manufacture, products, service, information, logistics) needs development and improvement.

There is still much to do in r&d on market-oriented, integrated product development, e.g. to apply and integrate methods of consumer research and product planning, separately on BtoB and BtoC basis , and cover entire value chains between forest and end-user. Further benchmarking efforts and analysis of operational and competition environments are needed for production and marketing of Nordic pine industries, respectively. Future projects should aim to more automated methods to measure and grade wood and log properties for structural, but also visual end-uses.

5.4a Capabilities generated by the project

The project contributed to create added value in the following terms as industrial and scientific achievements as well as current and future's r&d collaboration platforms:

- Multi-disciplinary approach widened the scope and contributed to prioritise the aims of the studies
- Possibilities to compare and benchmark future research strategies
- Insight of the product development activity in the industry, its content and driving forces, success factors and obstacles
- Better understanding and positioning of Nordic pine from different regions against competing species and non-wood materials
- Data banks on wood and timber properties available for further r&d work (modelling, etc.)
- Four doctoral theses, one in wood science and three in forest product marketing, were started in the project, they will be ready in 2008-10, thus, adding to the Nordic r&d capacity within the disciplines.

5.4b Utilisation of results

The results of the project will provide extensive benefits for the strategic development of wood product industries in both countries in the planning, allocation and sourcing of log procurement, organising and focusing product and technology development, planning marketing designs and contents of marketing information and formulating investment strategies, based on the raw material basis and potential for positioning and segmentation in the end-product market. The advantages and disadvantages of Nordic Scots pine can now be weighed and considered in the before-mentioned activities, especially in the further development of business-to-business trade of pine products. These results also indicate the optimal forest management strategies and activities of Scots pine to pro-

fessional forest owners and forestry planning officials at the regional and stand levels, e,g, the potential of timber growing for high quality to improve the profitability of forestry.

Understanding the concept and alternatives of customer interfaces, partnerships and value chains in pine industry business, the role of the different specifiers as well as the potential and complexity of innovation process contribute to further develop the business strategies of the wood industries. Consequently, the strategic models suggested for implementing customer-orientation, with regard to innovation and product development may be tailored to the benefits of individual wood industry companies.

During the project, specified ideas were raised and implemented among the participating companies to apply wood technological modelling and NDT measurement of trees and logs in the product and technology development, using the data banks collected and organised within the project. Marketing tool packages are also under development in the main product segments of Nordic pine, at the levels of regions and individual companies. New r&d contacts were established between the research institutions in Finland and Sweden, as well, for, hopefully, fruitful project combinations also in the future.

5.5 Publications and communication

a) Scientific publications

The most important publicatoins are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

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* Stendahl, M., Roos, A. & Hugosson M. 2007. Product development in the Swedish and Finnish sawmilling Industry – a qualitative study of managerial perceptions. Journal of Forest Products Business Research. Accepted.

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* Lindström, H. 2005. The effects of growth condition variables on the modulus of elasticity in Nordic Scots pine. Scandinavian Journal of Forestry. Submitted.

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2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

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6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series

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5.6 National and international cooperation

Continuous contacts were kept with the Finnish and Swedish wood industries, forest managers, and relevant research teams in order to guarantee the applicability of the results. The project collaborated for the dissemination in Finland with several regional development programmes of the wood product sector, Wood Focus Ltd. and the Finnish Centre of Expertise for Wood Products (PuuOSKE). Scientific collaboration was done with the related projects of VTT and Helsinki University of Technology (sub-projects 1 and 2) and the Norwegian University of Agriculture (sub-project 3).

Advisory group

Mikael Eliasson, Setra Group)

Matti Heikurainen, Finnish Ministry of Agriculture and Forestry

Ismo Heinonen, Vapo Timber Ltd., 2003-04

Jaakko Lehto, UPM Wood Products Oy

Leena Paavilainen, Wood Material Science and Engineering Research Programme 2003-05, Finnish Forest Research Institute 2006

Timo Pöljö, Finnforest Ltd.

Jouko Silén, chairman; Stora Enso Timber Oy Ltd.

Heikki Juslin, *Mats Nylinder, Anders Roos* and *Erkki Verkasalo* participated as the representatives of the project.

Commonly to the project, sub-project 1 organised the work of an invited expert group to map the current knowledge and set the priorities of r&d issues and key marketing arguments, in particular. Thus, the researchers were provided with necessary knowledge and ideas for higher relevance of the project, and new development ideas for products, processing methods and treatment of Nordic pine as well as advanced business models were aimed to. The group had a role in the dissemination of the project and the previous research knowledge. During 2004-06, four expert group meetings were organised, and their results were reported as summarising papers.

Researchers of all sub-projects presented papers in several scientific international conferences, such as XXII IUFRO World Congress in Australia (2005, four participants), IUFRO WP S5.01.04 Fifth Workshop "Connection between forest resources and wood quality: modelling approaches and simulation software" in New Zealand (2005, three participants), three workshops of COST Action E44 Workshop (2004-2006, six participants, in total), 5th International Symposium of Wood Structure and Properties '06 in Slovakia (2006, two participants) and COST Action E35 Training School on Surface Characterization (2006, one participant). Researchers of sub-project 3 are actively participating the activity of the new COST Action E51 and established research collaboration with Oregon State University, that also conducts research on marketing and innovation in the wood industries. Researcher trainees from Ecole Superieure du Bois, Nantes (France) and Catalan Wood and Furniture Insititute, Solsona (Spain), two of them, worked in the sub-project 1 during 2005.

6 Straight and durable timber (STDUT)

FINAL REPORT	
Name of the research project	**Straight and durable timber**
Coordinator of the project	Anders Grönlund

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**Straight**
Project period	1.3.2003–30.6.2006
Organization in charge of research	Luleå University of Technology, Skellefteå Campus
Sub-project leader	Anders Grönlund
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SKERIA 3; SE-931 87 Skellefteå, Sweden Tel. +46 910 585307 anders.gronlund@ltu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	300 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	260 000
Other public funding	
Luleå University of Technology	40 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Anders Grönlund	M	LTU		Formas
Stig Grundberg	M	SSP-Trätek/LTU		Formas
Johan Oja	M	SP-Trätek/LTU		Formas
Micael Öhman	M	LTU		Formas
Mats Ekevad	M	LTU		Formas

Total person-months of work conducted by the research team 23,9
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	D.Sc.	M	Mats Ekevad	LTU	Anders Grönlund

Name of the sub-project 2	Durable
Project period	1.3.2003–30.6.2006
Organization in charge of research	VTT
Sub-project leader	Arto Usenius
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Materials and Building P.O.Box 1000 FI-02044 VTT, Finland Tel. +358 20 722 5540 Fax +358 20 722 6251 arto.usenius@vtt.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	265 671
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	164 177
Other public funding	
VTT	35 000
Other funding	
Finnish companies	66 494

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, raduate school	For a visitor: rganization & ountry of origin	Funder
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Isabel Pinto Seppä, D. Sc. (Tech), Research Scientist	F			
Pieti Marjavaara, M.Sc., Research Scientist		VTT		
Jorma Fröblom, M.Sc. (Tech), Senior Research Scientist	M	VTT		

Antti Heikkilä, M.Sc. (Tech), Research Scientist	M	VTT
Tiecheng Song, Lic.Sc. (Tech), Research Scientist	M	VTT
Eero Halonen, Technician	M	VTT
Total person-months of work conducted by the research team 23,2 person-month = full-time work for at least 36 h/week, paid holidays included		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Abstract

Two important disadvantages with wood are that it can warp and rot. The result of these negative properties is that wood products have lost market shares in applications where wood traditionally has been the natural choice of material.

The basic idea of this project is to make it possible to avoid the negative properties of wood and utilise the good properties in a systematic way. To achieve this, fast and accurate methods for automatic measurement and detection of different wood features have to be developed. This project has been focused on measurement and detection of spiral grain, compression wood and pine heartwood. Some results of the project are:

- The spiral grain gradient is an important parameter for the size of twist
- Twist can be predicted fairly well from measurements of spiral grain on the mantel surface of the log
- Twist can also be predicted fairly well from measurement of spiral grain on board surfaces in transverse feed.
- It is possible to calculate the size of needed pretwist during drying in order to avoid twist.
- The outer shape of the logs is a fairly good predictor for compression wood which gives possibilities to avoid crook and bow on the sawn boards.
- There are several possible methods for measurement of heartwood content in logs and boards. Following sensors have been used in the investigations: x-ray, RGB-camera, IR-camera and laser systems.

The project also resulted in a sawing simulator software capable to take into account heartwood content as a product feature simultaneously with other type of sawn timber product. This is for predicting profitability of producing heartwood products.

Tiivistelmä

Puu voi tietyissä olosuhteissa kieroutua ja lahota. Nämä negatiiviset ominaisuudet ovat johtaneet siihen, että puu on menettänyt markkinaosuuttaan käyttökohteissa, joissa aikaisemmin puun valinta oli itsestäänselvyys.

- Projektin perusajatuksena oli systemaattisesti luoda sellaista tietoa, jolla voidaan poistaa puun huonoja ominaisuuksia ja hyödyntää täysimääräisesti hyvät ominaisuudet. Tämä edellyttää nopeiden ja luotettavien puun ominaisuuksien mittausmenetelmien kehittämistä. Projekti suuntautuu vinosyisyyden, lylyn ja männyn sydänpuun mittaamiseen. Seuraavassa joitakin tärkeitä tutkimustuloksia:
- Vinosyisyyden säteensuuntaisella gradientilla on suuri vaikutus sahatavaran kieroutumiseen

- Sahatavaran kieroutuminen voidaan luotettavasti ennustaa mittaamalla tukin pinnalta vinosyisyys
- Sahatavaran kieroutuminen voidaan myös ennustaa mittaamalla vinosyisyys kappaleen sydänlappeelta
- On mahdollista laskea kuivauksessa tarvittava vastavääntö kieroutumisen välttämiseksi
- Tukkien ulkomuoto on hyvä indikaattori ennustamaan lylyn määrää, jotta voidaan välttää sahatavaran lape- ja syrjävääryyttä
- Erilaisilla menetelmillä voidaan mitata sydänpuun osuutta tukeissa ja lankuissa. Seuraavia menetelmiä tutkittiin: röntgen, värikamera, IR-kamera ja laser mittari.

Projektin tuloksena syntyi myös sahauksen simulointiohjelmisto, joka laskee tukeissa ja tuotteissa olevan sydänpuuosuuden. Tuotteet voidaan lajitella samanaikaisesti myös muilla lajittelusäännöillä, jolloin saadaan selville sydänpuutuotteiden valmistuksen kannattavuus.

Sammanfattning

Trä kan under vissa förhållanden deformeras (skevt, krokigt) och ruttna. Dessa negativa egenskaper har medfört att trä har förlorat marknadsandelar i vissa applikationer där trä tidigare har varit det självklara materialvalet.

Den grundläggande idén med detta projekt har varit att på ett systematiskt sätt försöka undanröja träets dåliga egenskaper och att nyttja de goda egenskaperna fullt ut. För att klara detta krävs att snabba och tillförlitliga metoder för mätning av olika träegenskaper utvecklas. Detta projekt har inriktats mot mätning av växtvridenhet, tjurved och furukärnved. Några viktiga projektresultat är:

- Växtvridenhetens gradient i radiell led har stor inverkan på plankornas skevhet.
- Plankornas skevhet kan på ett tillförlitligt sätt predikteras baserat på uppmätning av växtvridenheten på stockarnas mantelyta.
- Plankornas skevhet kan också predikteras baserat på uppmätning av växtvridenheten på plankornas splintsida.
- Det är möjligt att beräkna erforderlig motvridning under torkning för att undvika skevt virke.
- Stockarnas yttre form är en god indikator för mängden tjurved vilket gör det möjligt att minska flatböj och kantkrok på det sågade virket.
- Det finns ett antal möjliga metoder för mätning av kärnvedsandel i stockar och plankor. Följande sensortyper har testats: röntgen, färgkamera, IR-kamera och lasersystem.

Ett resultat från projektet är en sågsimulator som kan beräkna kärnvedsandelen i stockar och plankor och sortera plankor enligt olika andra egenskaper för att få fram mest lönsamma produktionsmetod.

6.1 Introduction

6.1.1 Background

Wood is a biological material with many excellent properties but wood also has some undesirable properties such as warp and rot. The result of these negative properties is that wood products have lost market shares in applications where wood traditionally has been the natural choice of material. Steel studs and PVC windows are two examples where wood has been substituted. Lack of straightness is the main reason for substitution of wood in the stud application. In the case of window construction it was the extensive rot damage in wooden windows during the nineteen-seventies that started the substitution of PVC for wood in window construction.

Many scientists have studied warp and which parameters that have an influence on the magnitude of the warp when the moisture content changes. Warp can be divided into three modes: twist, bow and crook. Twist is mainly influenced by the magnitude of spiral grain and the distance from the pith. Bow and crook are influenced by differences in the longitudinal shrinkage in different parts in a piece of wood. The differences in longitudinal shrinkage depend mainly on the distribution and magnitude of compression wood and juvenile wood.

In the Nordic countries, windows have traditionally been made of heartwood from Scots pine (*Pinus sylvestris*). In the beginning of the nineteen-sixties the window manufacturing process became more and more industrialised. This resulted in less time and attention being spent on selection of wood with proper properties. The good durability of pine heartwood, for instance was no longer utilized consistently.

The fact that the windows were no longer made of pine heartwood was also one a factor in the extensive rot damages in wooden windows during late nine-

teen-sixties and the nineteen-seventies. The problem was temporarily solved by preservative treatment of the windows. Today, preservative treatment has become more and more called into question. One way to avoid preservatives is, of course, to go back to the old method and begin to utilize the natural durability of pine heartwood in a systematic way.

6.1.2 Objectives

The main objectives for this project are:

- Study and develop methods for industrial measurement of spiral grain, compression wood and pine heartwood.
- Study how the straightness of the timber is affected by local variations of spiral grain and compression wood.
- Develop strategies and algorithms for control of the production process.

6.2 Results and discussion

6.2.1 Production control based on measurement of spiral grain and compression wood

Distortions due to moisture changes during drying or in service are a major problem for construction timber. Twist, caused mainly by the cylindrical geometry, the orthotropic nature of the wood material and the tendency of the wood fibres to grow in a spiral around the stem, is often regarded as the most detrimental distortion of sawn timber.

There is a need for a basic mechanical understanding of how the twist distortion arises and also a need for a simple formula to predict the amount of twist distortion. In the STDUT-project such a formula has been developed, and theory and experimental data which indicate the validity of the formula are shown (Ekevad, 2005). The first term in the formula is a modification of a traditional expression which is proportional to the mean value of the spiral grain angle in the cross-section in question. The second term in the formula is new and is proportional to the gradient of the spiral grain angle, and this term normally counteracts the first term so that a stud with a left-handed spiral grain might achieve a right-handed twist.

Warp of sawn timber is induced by moisture content changes which indicate that most of the warp will take place during kiln drying of the timber. The magnitude of warp depend on wood parameters such as degree of spiral grain and compression wood pattern but also on a lot of other parameters such as drying schedule, loading during drying, sticking practises e.t.c. In order to model the warp during drying a model has been devel-

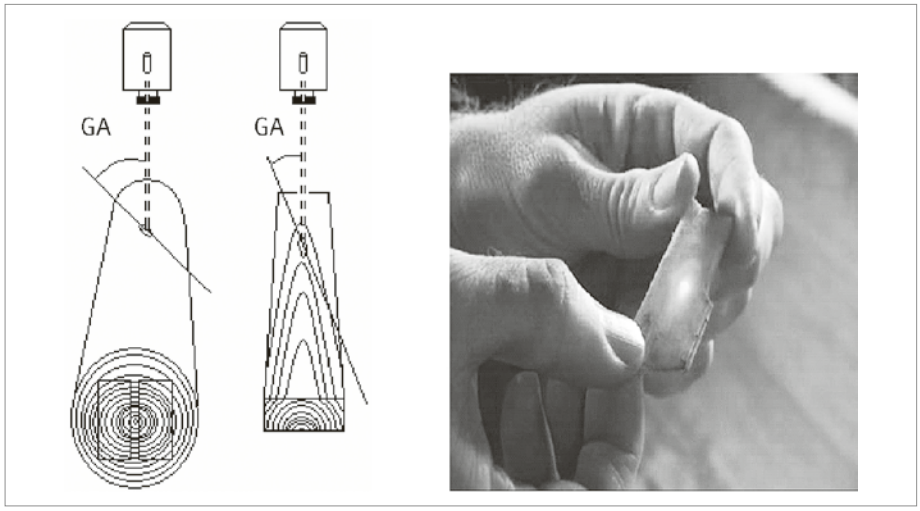

Figure 1. Measurement of spiral grain angle (GA) on logs and boards. This specimen with left-handed spiral grain represents an individual piece prone to distort during drying. The picture on the right shows the tracheid effect.

oped. This model include a method to measure the diffusion coefficient from CT- images (Danvind and Ekevad, 2006), a FEM based drying module and a FEM based warp module. The results from the model have been validated against laboratory experiments and against industrial drying tests. The model results fit well against the laboratory results and fairly well against the industrial tests, (Ekevad et.al. 2006).

Spiral grain on logs and boards can be measured using the tracheid effect, Figure 1. The tracheid effect utilizes the light-conducting properties of the softwood tracheids to measure the direction of spiral grain. A small laser point is projected onto the wood surface. The light transmitted in the wood and scattered back forms an elliptic shape extended in the direction of the fibres. The ellipse of light is registered with a camera and the orientation of the ellipse's major axis corresponds to the fibre direction (Nyström, 2000).

In an industrial test the spiral grain angle on log surface was measured with the tracheid method (Figure 1) during longitudinal transport of the debarked logs. The test material consisted of 250 Scots pine (*Pinus sylvestris*) logs in the top diameter range between 121 and 130 mm. The logs were sorted into three grain-angle groups (GA1 – GA3) where GA3 is the most left-handed group. Of the test logs, 70% were sorted into GA1 and 15% each into the two other groups.

The logs were live sawn to boards with a green target size of 24 mm and dried to 8% MC. All boards were pressured with a top load during drying and half of the boards were pre-twisted in the drying stack in the opposite direction of the normal twist direction. The other half was stored flat in the drying stack. After drying the boards were planed and glued together to 2500 mm long and 600 mm wide edge glued panels. After sanding the twist of the panels was measured on a length of 2000 mm and a width of 600 mm.

The result of this test is shown in Figure 2. It is quite evident that the group with the lowest left-handed grain angle (GA1) is least twisted. It is also quite clear that pretwisting during drying in the opposite direction to the expected twist direction reduces the twist of the finished edge-glued panels.

A method for prediction of warp based on the outer shape of the logs has been developed. Input data to the modelling work comes from CT-scanned logs. It has been proved that the outer shape is a better predictor of warp than the amount of visible compression wood on the butt end of the logs. The basic idea of the method is to measure the outer shape of the log in segments along the log. The results show that algorithms based on outer shape can sort logs according to compression wood content in two classes reasonable well.

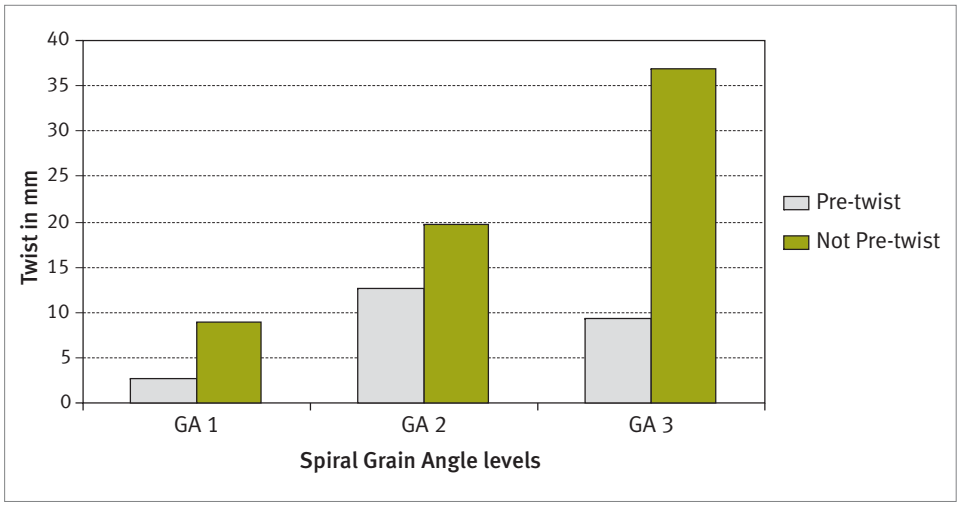

Figure 2. Twist in mm of edge glued panels 18 x 600 x2000 mm for different spiral grain angle levels (GA) and the influence from pre-twist during drying.

6.2.2 Measurement of heartwood on logs and boards

1. Sampling for modelling purposes

Two Scots pine samples were selected for data collecting and analysis within this task: a set of industrial logs collected from the normal raw material flow and a forest sample collected at different sites in Finnish Lapland.

1.1 - From industrial partner Stora Enso Timber (Honkalahti) 95 logs were sampled. Logs were measured with Opmes 604 on the bark. After debarking process, logs were measured with Opmes 202.A sub-sample of 20 logs was selected to be scanned by VTT´s x-ray scanner. Further these 20 logs were sawn into 20 mm flitches and these were RGB scanned. A line was drawn in the logs butt and top ends so different scanning systems could have the same position reference. For validation purposes images of butt and top ends of the logs were register with this reference line. From the remaining 75 logs 2 central lumber pieces were sawn after x-ray scanning.

1.2 - In Finnish Lapland 45 logs were sampled from 38 stems in 5 sites. Field information was registered concerning age, live and dead crown height and log stem position and diameters. At VTT premises these were x-ray scanned and further 2 central pieces of lumber were sawn and scanned.

2. Measurement protocol

Scanning was performed as follows.

X-ray scanning of the logs. Logs were mounted on movable carriage in fixed position and passed X-ray zone. After one pass under the X-ray source and detector the log to be measured is returned and rotated by 15°. This procedure is repeated 24 times to cover the whole circle of the log. Log's feeding speed in to the system was 0,55 m/s. Wet and dry timber pieces were measured outer face upwards with the same feeding speed of 0,55 m/s. All images were stored as individual data sets and were processed further.

IR-measurements. Wet and dry timber were measured on both flat faces using feeding speed of 0,5 m/s. Faces of the pieces were illuminated with IR radiation of 200-230 °C. The reflected waves were observed by IR-scanning system. Also other type of IR-camera was tested too. This unit makes images by a whole frame instead of one line imaged by the permanently mounted IR-scanner.

RGB-measurements. All four faces of wet and dry timber were scanned producing high quality colour pictures. These images were processed and studied.

3. Data processing for heartwood detection

Data received from different sensors was stored into databases and analysed using software systems in order to develop algorithms for heartwood detection. Research has mainly devoted scanning of logs by x-ray instrumentation. Data was processed and heartwood was detected directly from the x-ray image of logs and central boards from the two sample sets. Also logs were reconstructed by tomographic algorithms to 3D-images. The 20 sub-sample logs from SET material were also 3D reconstructed by the flitch method and data was used for methodology validation.

4. Development of heartwood sawing simulator

A new version of sawing simulator was developed in 2005 for heartwood products sawing simulation and optimization. With the newly developed algorithm for heartwood content based classification of sawn timber, the simulator can be used to evaluate value yield of sawing value-added heartwood lumber products for special user requirements.

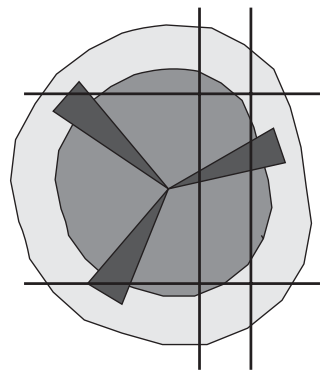

Figure 3. A piece of lumber is cut with four cutting planes on a log with internal knots and log envelope and heartwood core.

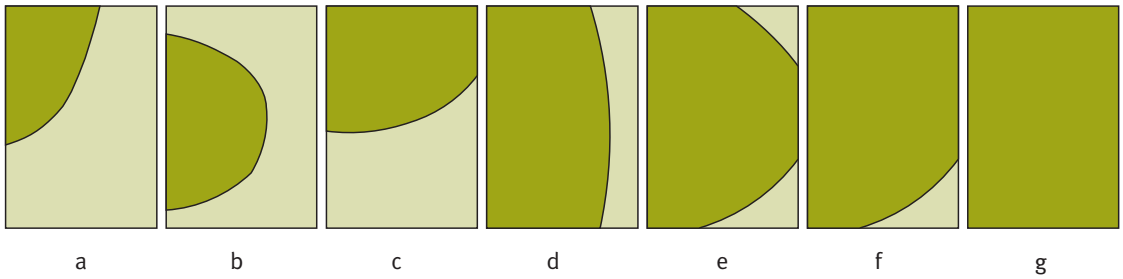

Figure 4. Different cases of heartwood wanes in lumber cross-sections.
Heartwood is painted with darker colour and lighter areas stand for sapwood.
a: One face and one edge are of 100 % heartwood wane.
b: One face and two edges have 100% heartwood wane.
c: One edge has 100% heartwood wane and another edge is of 100% heartwood.
d: One face is of 100% heartwood wane and another face is full of heartwood.
e, f: are the normal cases with some heartwood wanes on out-face and one or two edges.
g: is a lumber case with 100 % heartwood content .

As the input data for the heartwood sawing simulator, a 3D numerical log model was built with an external envelope, internal knots structure and a heartwood core envelope. With the log model, a standard log data format is defined for log reconstruction during log measurement data processing. Figure 3 shows that a lumber is cut as a result of two horizontally parallel cutting planes and two vertically parallel cutting planes on a 3D numerical log.

Heartwood content of a sawn product is presented with so called heartwood wanes of a lumber. Heartwood content based classification rules, which are defined according to customers' requirements, are used to evaluate heartwood content class of lumbers. In the sawing simulator, heartwood lumber classification is done by checking "heartwood wanes" of both faces and the edge with more "heartwood wane". Figure 4 displays several cases of heartwood wane of a lumber.

The traditional lumber product model was enhanced with heartwood sawn products specification and price data. With the new simulator, lumbers can be graded with both some conventional grading rules (such as Nordic Timber grading rules) and heartwood content based classification, and then evaluated with their category prices accordingly. Therefore value-adding impact of cutting heartwood lumbers can be assessed with this simulation tool. The simulator is a module of VTT's WoodCIM® software package, which is an integrated model and software system for optimisation of activities within wood conversion chain – from forest to final products.

6.3 Conclusions

Our work has shown that it is possible to calculate needed pretwist and top load in order to avoid twist on the sawn boards during drying with fairly good accuracy.

The method for measurement of spiral grain on the mantle surface with aid of the tracheid effect works well and seems to have good correlation with twist on the sawn boards. Measurement of spiral grain magnitude on sawn board in cross transportation does also show a good correlation with twist on sawn boards.

Models for prediction of warp due to compression wood based the outer shape of the logs or the green span of the boards are promising but need further evaluations in industrial tests.

Detection of heartwood inside the logs is possible using x-ray sensor. On the top end and butt end of the log heartwood core is visible using IR-measuring method. There is potential to detect heartwood in the sawn timber pieces using x-ray-, IR-, RGB- and laser sensors.

Heartwood sawing simulator has been developed in order to demonstrate economic potential of manufacturing special heartwood sawn timber products. The simulator can be used in production planning procedures.

6.4 Capabilities generated by the project

Doctoral thesis

Ekevad, M. 2006. Modelling of Dynamic and Quasi-static Events with Special Focus on Wood-Drying Distortions. Luleå University of Technology, DT 2006:39. ISSN: 1402-1544.

Computer program

A FEM - program for calculation of suitable pretwist during drying has been developed.

New processes and practices

A method for reduction of twist on sawn timber has been tested and implemented in the industry.

Transferring of results into practice

The results are transferred to the industry through SP-Träteks and VTT:s industry service program.

6.5 Scientific publications and communication of information

a) Scientific publications within the project

Ekevad, M. 2004. A method to compute fibre directions in wood from computer tomography images. *J. Wood Science,* Vol.50, No.1, pp.41-46, 2004.

Grönlund, A.; Grundberg, S.; Oja, J.; Nyström, J. 2005. Scanning Techniques as Tools for Integration in the Wood Conversion Chain – some Industrial Applications. In proceedings of the COST Action E44 Conference "Broad Spectrum Utilisation of Wood". June 14 – 15, 2005 BOKU Vienna, Austria.

Ekevad M.2005. Modelling the effect of local variations in the wood material on the quality of the sawn timber. IUFRO Working Party 5.01.04. Wood Quality Modelling. 20-27 November 2005. Waiheke Island Resort, New Zealand.

Ekevad M. 2005. Twist of wood studs: dependence on spiral grain gradient. J Wood Sci 51:455-461.

Danvind J. & Ekevad, M.2006. Local water vapour diffusion coefficient when drying Norway spruce sapwood: J Wood Sci. 52:195-201..

Ekevad, M.; Salin, J.-G.; Grundberg, S.; Nyström, J. & Grönlund, A. (2006). Modelling of adequate pretwist for obtaining straight timber. Wood Material Science and Engineering 1:76-84.

Öhman, M. 2005. Prediction of compression wood in sawn products of *Picea abies*, by a 3-D sequential outer shape modeling of the log. IUFRO Working Party 5.01.04. Wood Quality Modelling. 20-27 November 2005. Waiheke Island Resort, New Zealand.

Pinto I., Usenius A., Song T., and Pereira H. (200-) Sawing simulation of maritime pine (Pinus pinaster Ait.) stems for production of heartwood containing components. Reviewed and accepted for publication in Forest Product Journal.

Pinto, I., A. Usenius, T. Song, and Pereira H. (2005). Sawing simulation of maritime pine (*Pinus pinaster* Ait) stems for production of heartwood containing components. Forest Prod. J. 55(4): 88-96.

Song, T., Pinto, I., Usenius, A. 2005. Sawing Simulation of pine heartwood products as a new WoodCIM® feature. In: Nepveu, G. (ed.). Proceedings of IUFRO WP S5.01-04 Fifth Workshop 'Connection between forest resources and wood quality: Modelling approaches and simulation software'. Waiheke Island Resort, Auckland, November 20-26, 2005, New Zealand. (in print)

Usenius, A. Optimisation of sawing operation based on internal characterization of the logs. Paper presentated in ScanTech 2003 Conference in Seattle USA 4-5.11.2003.

b) Publishing and dissemination of information outside the scientific community

Seminar "FRAMTIDENS SÅGTIMMERMÄTNING" in Stockholm 2003-04-10. Organiser: Rådet för virkesmätning och redovisning.

Oral presentations by Stig Grundberg, Micael Öhman, Johan Oja and Anders Grönlund.

Seminar "TRÄFESTIVALEN" in Skellefteå 2003-10-21 – 2003-10-22. Organiser: Stiftelsen Träenigheten.

Seminar "RAKT VIRKE" in Sundsvall 2003-11- 05. Organiser Trätek.

Oral presentations by Stig Grundberg and Johan Oja.

Seminar "RAKT VIRKE" in Växjö 2004-05- 26. Organiser Trätek.

Oral presentations by Stig Grundberg and Johan Oja.

Seminar "Process IT" in Stockholm 2005-02-03. Organiser IVA (Royal Swedish Academy of Engineering Sciences). Oral presentation by Anders Grönlund.

Seminar "Skogens dag" in Umeå 2005-03-04. Organiser Norra skogsklubben. Oral presentation by Anders Grönlund.

Ny processteknik- nya möjligheter, SLU Virkesdagarna 2005 Uppsala. Oral presentations by Stig Grundberg.

Optimering av såglinjer, FSS Vinterexkursion i Bygdsiljum/Umeå 7-8 december 2005. Oral presentations by Stig Grundberg.

Usenius, A. Optimisation and controlling activities in complex fores – wood – end user chain. Keynote speech in eBusiness for forest industries conference 2003. 23.11.2003. Scottish Forest Cluster.

Usenius, A. Konenäkö ja sen sovellukset puuteollisuudessa (Machine vision and it's application in wood industry). Mekaanisen puun mittauspäivät. 18-19.9.2003 Vuokatti.

Usenius, A. Puun käytön optimointi puutuoteteollisuudessa (Optimisation of raw material use in wood industry). Interreg Northwood Workshop 4.12.2003 Rovaniemen maalaiskunta.

Usenius, A. Optimization of Sawing Operation Based on Internal Characterisation of the Logs. ScanTech 2003. The tenth international conference on scanning technology and process optimization for the wood industry. Holiday Inn, Seattle-Tacoma International Airport, Seattle, Washington, USA. Nov. 3-4, 2003. Wood Machining Institute. 8 p.

Usenius, A., Song, T., Marjavaara, P., 2005, Automated Heartwood Detection and Optimization of the Manufacturing of Heartwood Components, ScanTech Conference 2005, Las Vegas, USA.

Usenius, A., Heikkilä, A. and Song, T. (2006) WoodCIM®Sistema de Software Integrado para Soporte en la Toma de Decisiones en Aserraderos – desde el Bosque hasta los Productos Finales (WoodCIM® - integrated software system supporting decision making at the sawmills - from the forest to the end products. Proceedings of Scantech 2006. Buenos Aires, Argentina 2-3.11.2006 Expo VESTAS and Wood Machining Institute, Berkeley, USA, pp. 4-30.

6.6 National and international cooperation

Advisory group

Jouko Silen, R&D manager, Stora Enso Timber (chairman)

Leena Paavilainen, Programme Director, Wood Material Science Research Programme

Juha Lakotieva, Project Manager, Rovaniemi

Per-Micael Samuelsson, Plant Manager, Rundvik sawmill, SCA.

Göran Storm, Technical Manager, Setra group

Peter Nilsson, Development Engineer, Norra Skogsägarna.

Important partners outside the project are companies that are delivering measurement equipment to the Wood Products Industry and of course the Wood Products Industry itself.

7 Chemical microanalysis of wood tissues and fibres (MICROAN)

FINAL REPORT

Name of research project	**Chemical microanalysis of wood tissues and fibres (MICROAN)**
Coordinator of the project	Bjarne Holmbom

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Chemical microanalysis of wood tissues and fibres**
Project period	1.1.2003–31.12.2005
Organization in charge of research	Åbo Akademi University, Process Chemistry Centre
Sub-project leader	Bjarne Holmbom
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Åbo Akademi University, Process Chemistry Centre Laboratory of Wood and Paper Chem. Porthansgatan 3, 20500 Åbo Akademi Tel. +358 2 215 4229, Fax +358 2 215 4868 bjarne.holmbom@abo.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	237 900
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Academy of Finland	237 900

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	Funder	Total person-months of work conducted by the research team
Bjarne Holmbom, Ph.D., prof., project leader	M	Åbo Akademi University, Process Chemistry Centre	ÅA	-
Andrey Pranovich, Ph.D., researcher	M	Åbo Akademi University, Process Chemistry Centre	AoF, ÅA	11
Pedro Fardim, Ph.D., prof. (from 01.08.2005)	M	Åbo Akademi University	AoF, ÅA	10
Elena Tokareva, M.Sc., Ph.D. student (from 15.12.2003)	F	Åbo Akademi University, Process Chemistry Centre	AoF	25

Total person-months of work conducted by the research team 46

person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2008	Ph.D.	F	Tokareva Elena, 1976, 1999, M.Sc.	Åbo Akademi	Prof. Bjarne Holmbom

Name of the sub-project 2	**Chemical microanalysis of wood tissues and fibres**
Project period	1.1.2003-31.12.2005
Organization in charge of research	Swedish University of Agricultural Sciences Department of Wood Science
Sub-project leader	Geoffrey Daniel
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Swedish University of Agricultural Sciences Department of Wood Science Tel. +46 18 672 489 Fax +46 18 673 489 geoffrey.daniel@trv.slu.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	68 218
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	68 218

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	Funder	Total person-months of work conducted by the research team
Geoffrey Daniel, Ph.D., prof., project leader	M	Swedish University of Agricultural Sciences Department of Wood Science	SLU	2
Nasko Terziev Ph.D. Researcher	M	Swedish University of Agricultural Sciences Department of Wood Science	SLU	3
Dinesh Fernando B.Sc. Ph.D. student		Swedish University of Agricultural Sciences Department of Wood Science	SLU	3

Total person-months of work conducted by the research team 8

person-month = full-time work for at least 36 h/week, paid holidays included

Abstract

Methods for chemical microanalysis of wood or fibres, with sample amounts as low as 0.5–2 mg, have been evaluated and further developed. The spatial distribution of different wood components across annual growth rings in spruce and aspen trees was studied using thin microtomed wood sections. Extractives were determined by solvent extraction and gas chromatography (GC), hemicelluloses and pectins by acid methanolysis and GC, lignin content by acetyl bromide treatment followed by UV absorbance determination, and lignin structure by thermally-assisted hydrolysis and methylation (THM). Metal profiles in the micrometer range across annual growth rings were determined by ICP-MS. Formation of compression wood was found in very straight and symmetric spruce trees stems, but only in some of the annual rings, and only in certain directions (sectors) of the stem. Compression wood was formed increasingly towards the end of the growth season. Compression wood in spruce was verified by the high content of galactans and lignin, and by structural features in the lignin. The profiles of certain metals were also very different in the annual rings where compression wood was formed.

Methods for Field-Emission Scanning Electron Microscopy (FE-SEM) and Time-of-Flight Secondary-Ion Mass Spectrometry (ToF-SIMS) have been applied for wood analysis. Different sample preparation procedures have been evaluated and compared for FE-SEM and ToF-SIMS. Sample preparation techniques for ToF-SIMS analysis of wood have been developed. Localization of Ca, Mg, Na, Fe in the bordered pit tori and ray cells, and low level lignification in the same regions have been found in spruce by ToF-SIMS. Both untreated and spruce sections treated with alkali or acid have been subjected to treatments with different metal markers and analysed by ToF-SIMS. At WURC, morphological features of fibre nanostructure structure and presence of dislocations after compression failure were characterised by microscopy (light (LM), Scanning Electron (SEM), Transmission (TEM)) techniques on whole spruce wood and macerated fibres. Histochemical techniques for following aspects of desresination in birch kraft pulps have also been developed.

Tiivistelmä

Puun ja kuitujen analysoimista varten arvioitiin ja kehitettiin kemiallisia mikroanalyysimenetelmiä, joissa näytemäärä on 0.5-2 mg. Mikrotomileikkeiden avulla tutkittiin puun eri komponettien vaihtelua kuusen ja haavan vuosirenkaiden eri osissa. Uuteaineet määritettiin uuttamalla ja kaasukromatografialla (GC) sekä hemiselluloosat ja pektiinit happamalla metanolyysillä ja GC:lla. Ligniinipitoisuuden määrittämiseen käytettiin asetyylibromidi-käsittelyä ja UV-absorbanssimittausta, ja ligniinin rakennetta tutkittiin lämpöhydrolyysillä ja metyloimalla (THM). Metallijakauma vuosirenkaissa määritettiin mikrometrin tarkkuudella ICP-MS-laitteella. Lylypuun muodustuminen havaittiin myös hyvin suorien ja symmetristen kuusten rungoissa, mutta vain eräissä vuosirenkaissa ja vain tietyissä rungon sektoreissa. Lylypuun muodostuminen lisääntyi kasvukauden päättymistä kohden. Lylypuun määrittämisessä käytettiin galaktaanien ja ligniinin suuria määriä ja ligniinin rakennetyyppejä. Tiettyjen metallien profiilit vaihtelivat niissä vuosirenkaissa, joissa lylypuuta muodostui. Puunäytteiden alyyseissä käytettiin Field Emission Scanning Electron Microscopy (FE-SEM) ja Time-of-Flight Secondary-Ion Mass Spectrometry (ToF-SIMS) menetelmiä. Näissä menetelmissä käytettäviä näytteiden esikäsittelymenetelmiä arvioitiin ja verrattiin ja ToF-SIMS:a varten kehitettiin puulle soveltuvia esikäsittelymenetelmiä.

Kuusen sädesoluissa ja huokosten torus-kalvoissa havaittiin ToF-SIMS:in avulla porilaisia Ca-, Mg-, Na-, ja Fe-määriä ja alhaisia ligniinipitoisuuksia. Alkali- ja happokäsiteltyihin näytteisiin sekä käsittelemättömiin näytteisiin lisättiin erilaisia metallimarkkereita ennen ToF-SIMS-analyysejä. Kuidun rakenteen erityispiirteitä ja paineen aiheuttamia muutoksia tutkittiin WURC:ssa erilaisilla mikroskopiamenetelmillä (valomikroskopialla (LM), elektronipyyhkäisymikroskopialla (SEM), läpivalaisu-elektronimikroskopialla (TEM)) sekä kuusen puunäytteillä että maseroiduilla kuiduilla.

Sammanfattning

Metoder för kemisk mikroanalys av vedprover och fibrer med så små provmängder som 0,5–2 mg har

testats och utvecklats vidare. Fördelningen av olika vedkomponenter över årsringar i gran- och aspträd undersöktes genom analys av tunna vedspån uttagna med en mikrotom. Extraktivämnen bestämdes genom lösningsmedelsextraktion och analys med gaskromatografi (GC), hemicellulosor och pektiner med sur metanolys och GC, innehåll av lignin med behandling med acetylbromid (AcBr) följt av UV-absorbansmätning, och ligninstruktur med termisk hydrolys och metylering (THM). Metallprofiler i mikrometerskala över årsringar bestämdes med ICP-MS. Bildning av tryckved påvisades även i mycket raka och symmetriska granstammar, men hade skett endast i vissa årsringar och i vissa sektorer av stammen. Tryckved konstaterades bildas i ökande utsträckning mot slutet av växtperioden. Tryckveden bekräftades av högt innehåll av galaktaner och lignin, och av vissa ligninstrukturer. Profilerna för vissa metaller var också mycket annorlunda i de årsringar där tryckved hade bildats.

FE-SEM (Field Emission Scanning Electron Microscopy) och ToF-SIMS (Time-of-Flight Secondary-Ion Mass Spectrometry) har använts för analys av vedprover. Olika metoder för provbehandling före FE-SEM och ToF-SIMS har utvärderats och jämförts. Med ToF-SIMS var det möjligt att analysera förekomsten av Ca, Mg, Na, Fe i ringporernas membraner och i märgstrålceller, samt att påvisa ett lågt lignininnehåll i dessa strukturelement. Prover av granved förbehandlade med alkali eller syra samt obehandlade prover behandlades med olika markörer för metaller och analyserades sedan med ToF-SIMS. Vid WURC utfördes morfologisk karaktärisering av fiberstrukturer, och dislokationer efter brott under tryck karaktäriserades med ljusmikroskopi, SEM, och TEM-tekniker på granved samt på macererade fibrer.

7.1 Introduction

7.1.1 Background

The fundamental knowledge of the spatial distribution and structure of various components in wood and wood fibres is still incomplete particularly regarding the detailed analysis of wood tissues, different cell types (e.g. fibre, parenchyma cells, vessels) and fibre primary and secondary cell wall layers. Extension of wood and fibre science towards both the ultrastructural and molecular levels is of fundamental importance, not only for wood science but also for the forest industry. New methods for chemical microanalysis of different components in wood tissues are required. In addition to chemical microanalytical techniques, the location and distribution of wood components and chemical composition can be analysed by microscopic and spectroscopic methods. ToF-SIMS is a new and very sensitive technique with a great potential for chemical characterization of wood surfaces at 1 nm analysis depth. However it requires further development and critical comparison with more used techniques such as SEM. The development and consequences of ‘‘cell-wall deformation’’ (commonly termed dislocations) on wood fibres, and their ultimate impact on the mechanical properties of paper products remains rather obscure and controversial.

7.1.2 Objectives

The main aim of the project was to develop, evaluate and apply new methods for characterization of wood tissues and fibres by chemical microanalysis and chemical microscopy. This can generate new fundamental knowledge about the location and spatial distribution of components in wood and fibres at the micro- and nanometer levels.

7.2 Results and discussion

7.2.1 Methods for chemical microanalysis

A scheme for chemical microanalysis has been devised and the different methods have been evaluated and further developed (Fig. 1).

All methods in the scheme can be used with samples of a few mg, and some even with μg-quantities. The methods are also fairly convenient. Extractives can be determined by solvent extraction and gas chromatography (GC) providing both concentrations and detailed composition of extractive types. Acid methanolysis and GC can determine the concentration and sugar composition of hemicelluloses and pectin directly on wood or fibre samples. Lignin content can be accurately determined by acetyl bromide (AcBr) treatment fol-

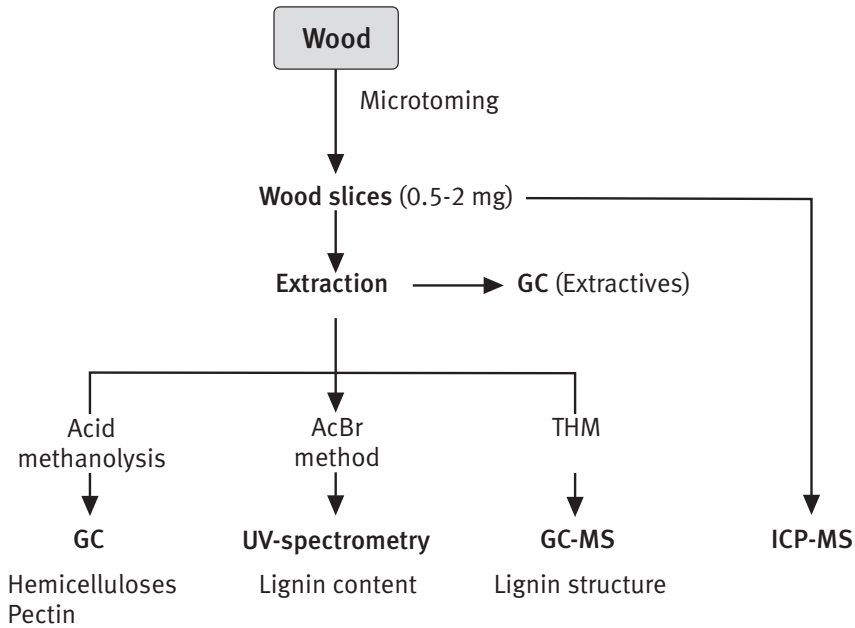

Figure 1. Scheme used for the chemical microanalysis of wood. The same methods can also be used for fibres.

lowed by determination of UV-absorbance. The AcBr method gives similar results as the Klason-lignin method that in contrast requires several grams of sample. Lignin structure can be assessed by pyrolysis GC-MS, but the technique of thermally-assisted hydrolysis and methylation (THM) provides even better structural information. Metal profiles in the micrometer range, over individual growth rings, can be determined by ICP-MS for a large number of metals in one analysis.

7.2.2 Distribution of wood components across annual rings

The distribution of chemical components over annual rings in several spruce and aspen trees were analysed on thin microtomed (125-μm) wood sections using the methods described above. The content of both hemicelluloses and pectins, composed of galacturonic acid and rhamnose units, was higher in spruce latewood (LW) than in earlywood (EW) within the individual annual rings. The content of lipophilic and hydrophilic extractives was 30-50% higher in EW than in LW. The hydrophilic extractives were mainly composed of monomeric sugars. Analyses revealed compression wood formation in the very straight and symmetric trees studied, however, only in some of the annual rings, and only in certain directions (sectors) of the stem.

Compression wood was verified not only by the high content of galactans, but also by the higher lignin content and the structural features of lignin. The lignin content was as high as 380-390 mg/g wood in LW where compression wood occurred, compared to 310-330 mg/g wood on the opposite side of the stem. Compression wood, measured by the galactan content, was formed increasingly during the latter part of the growth season in the LW (Fig. 2). The mannan content decreased when compression wood was formed.

In aspen, a smaller difference was found between the total amount of hemicelluloses in LW and EW compared to spruce wood. Slightly more pectin was found in LW (30 mg/g wood) than in EW (27 mg/g wood). In contrast to spruce wood, the variation in the distribution of individual sugars (after methanolysis of corresponding wood samples) was also less significant, except for mannose units. Unlike spruce, the variation in mannose distribution occurred in both EW and LW. Also in aspen the content of lignin in EW was very close to that in LW. Moreover, it was evenly distributed across the annual growth rings.

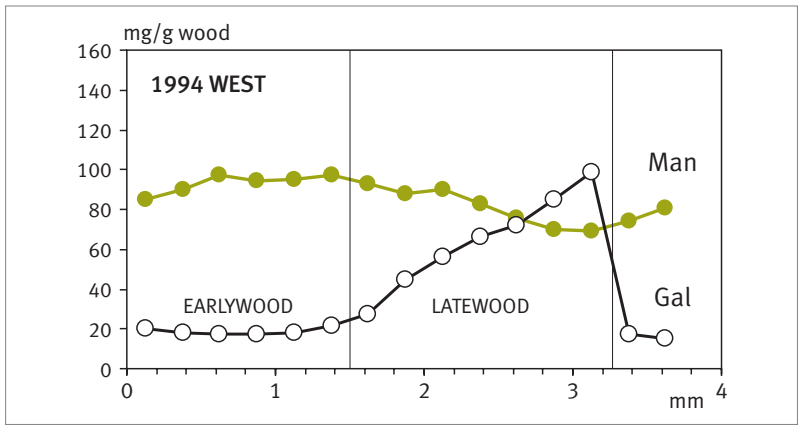

Figure 2. Spatial distribution of mannan and galactan across an annual growth ring of spruce.

Sections from spruce were analysed by ICP-MS in order to examine the spatial distribution of 17 metals across each individual annual growth ring. The profiles of some metals were substantially different in year rings where compression wood was formed. The concentrations of Ca, Mg, Mn, Ba, Zn and Sr increased in the latewood (LW) towards the end of the growth season. In opposite and normal wood the variation of all the analyzed metals across the annual growth ring was less pronounced.

Among the various wood constituents, pectin (i.e. partially methyl-esterified polygalacturonic

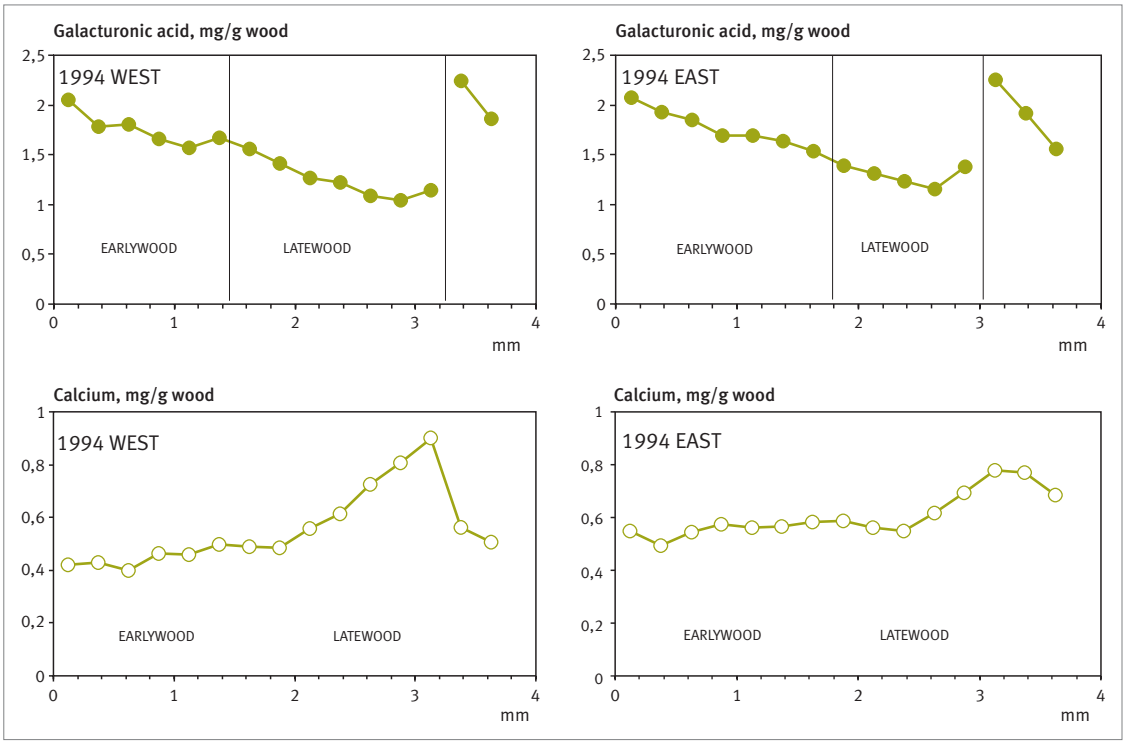

Figure 3. Distribution of galacturonic acid and calcium across the 1994 annual growth ring from the west and east directions of the spruce stem.

acid) is of special interest since it contains many anionic groups and has a high capacity to bind counter-ions, particularly metals. Pectin is known to be involved in so-called "egg-box" structures with Ca^{2+}. In our case however, the distribution profile of pectin (expressed as galacturonic acid units) was quite different to that of Ca (Fig. 3). Moreover, the profile of Ca was similar to that of both lignin and galactose.

7.2.3 Chemical microscopy

Various sample preparation procedures, including different sectioning techniques (i.e. cryomicrotoming, microtoming by steel knife, microtoming by disposable blade, and cutting by razor blade) and different drying preparation techniques have been investigated by FE-SEM. The final protocols with different methods of drying that have been used for sample preparation of wood samples for FE-SEM analysis are shown in Fig. 4.

Evaluation of the sample preparation techniques was made by FE-SEM and ToF-SIMS using different wood sections (i.e. radial, tangential and transverse sections). Sectioning with a disposable blade followed by critical point drying and gold-palladium coating was the best protocol for FE-SEM. The different cell wall layers, middle lamellae and the porous surface of the cell wall could be observed using this approach. FE-SEM analysis of the spruce samples confirmed the presence of compression wood in one sector (i.e. appearance of rounded cells, helical cavities inside the cells, intercellular spaces between the cells) and normal wood structure in the other sectors within the annual growth ring (Fig. 5).

In contrast to FE-SEM observations, ToF-SIMS investigation of wood samples prepared for SEM analysis (without coating by gold palladium) showed considerable contamination of the section surface by PTFE (polytetrafluoroethylene). PTFE was located in regions disturbed by the blade during sectioning. The improvement of sample preparation protocols for ToF-SIMS analysis included the cleaning of disposable blades in dichloromethane using an ultrasonic processor. Using this approach FD, AD, CPD and AEND wood sections have been analyzed and compared. The distribution of secondary ions originating from lignin, carbohydrates, extractives (ethanol-, acetone-soluble substances) and inorganic components in wood sections were obtained by imaging ToF-SIMS. We found that in the case of FD and AD sample preparation techniques, the wood section surfaces were covered in an even layer of extractives that interfered with the distribution of other wood components (for example ions originated from sodium have not been detected on surfaces covered with extractives). However, with samples prepared by CPD and AEND, extractives were removed, at least partially, and the distribution of sodium and other wood components was observed. Localization of inorganic wood constituents (i.e. calcium, magnesium, sodium, iron etc.) was observed in

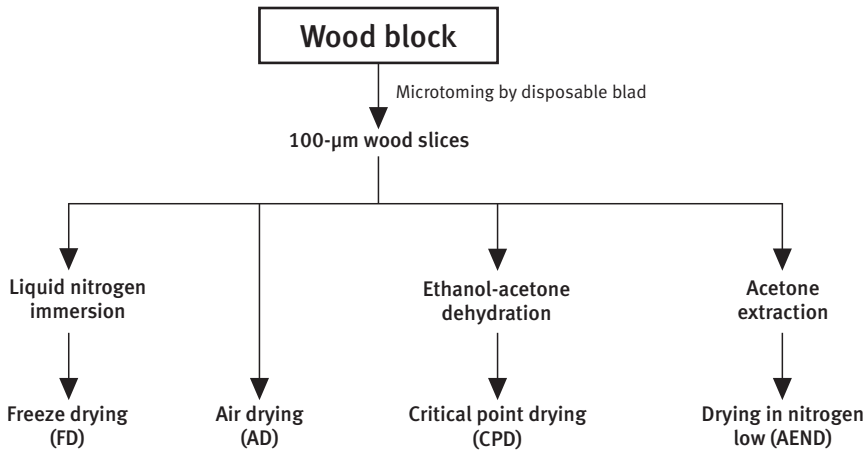

Figure 4. Sample preparation procedures for FE-SEM analysis.

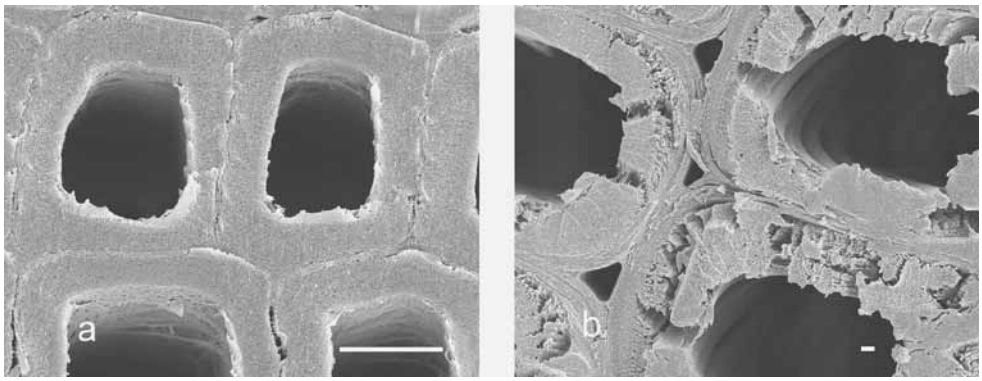

Figure 5. FE-SEM images showing transverse sections from the NE (a) and SW (b) directions of spruce sapwood from the 1994 annual ring. Bars: a, 10 µm; b, 1 µm.

bordered pit tori (Fig. 6b-d) and ray cell walls in spruce sapwood. In contrast, only little lignin was detected in the same morphological regions (Fig. 6f). Carbohydrates were evenly distributed across the wood section surface (Fig. 6e).

In addition, ToF-SIMS was applied to freshly-cut and acetone-extracted spruce sections, chemically treated in different ways (e.g. acid-treated and alkaline-treated). Treated and untreated samples were subjected to $MgCl_2$ and $SrCl_2$ impregnation. Preliminary results show that Mg and Sr appeared in bordered pit tori and ray cells in all samples (AD, AEND, alkali- and acid-treated). This study is being continued.

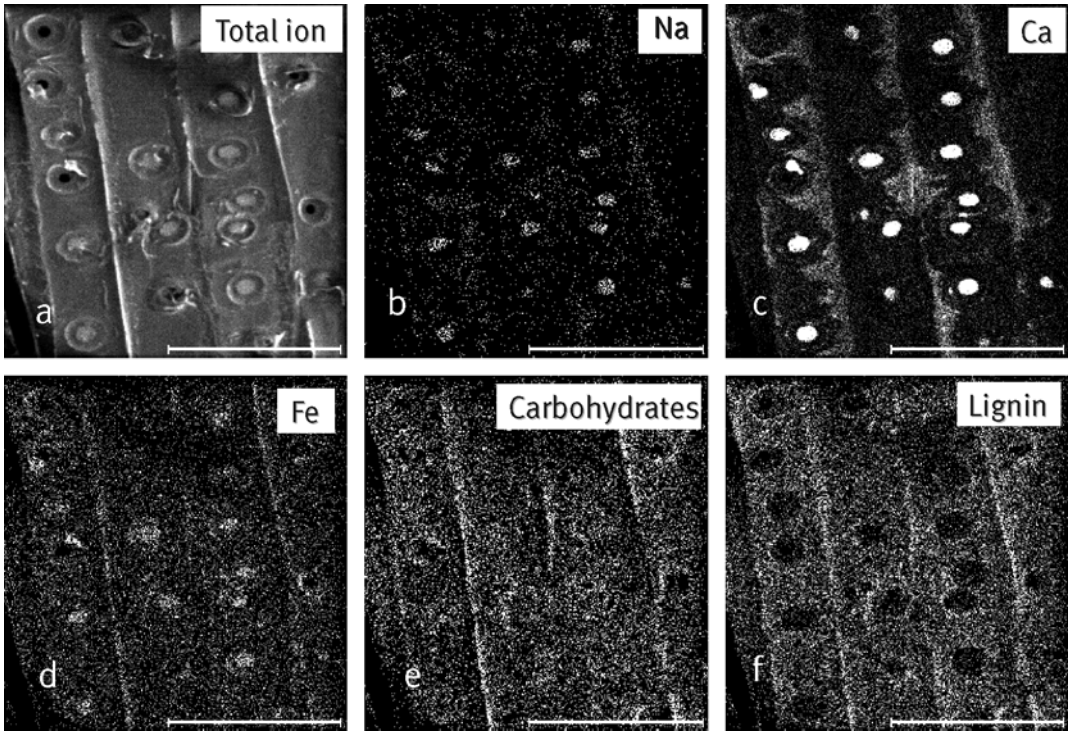

Fig. 6. ToF-SIMS imaging of a radial sapwood spruce section: (a) total ion image, (b) sodium, (c) calcium, (d) iron, (e) carbohydrates, (f) lignin. White color in images indicates the high intensity of secondary ions and black color indicates low intensity. Scale: 100 µm.

7.2.4 Ultrastructural and chemical studies on fibre cell wall structure

In the present work measurable quantities of dislocations were induced in spruce fibre cell walls by exposing the whole wood fibres to compression stress. Micro- and ultrastructural morphological features of fibre structure and presence of dislocations after compression failure were characterised by microscopy (light, SEM, TEM) techniques on the whole wood and macerated fibres.

Results have shown induced dislocations to significantly and negatively affect the mechanical properties of paper products (i.e. paper handsheets). Results show that the inclusion of 20 % fibres with dislocations results in a *ca* 12 % decrease in tear strength of handsheets and will therefore significantly affect fibre mechanical properties. Comparisons have also been made using the scale of increased percentages of dislocations as a mechanical property reference for other wood tissues including: juvenile, native compression and opposite wood as well as samples from cultivated forests receiving artificial irrigation or fertilization. Comparisons show that native compression wood and wood from the sapwood of irrigated and fertilised trees all contribute negatively to the mechanical properties of paper in comparison to clear mature sapwood.

To confirm changes in the mechanical properties of artificially compressed fibres, single fibre micro-tensile testing of artificially *compressed* and *reference fibres* has been conducted. Results emphasize differences in force-strain curve characteristics in earlywood and latewood fibres and that fibres exhibiting dislocations are weaker than comparative reference control samples taken either within the compression zone or from outside. Additional TEM studies to observe the ultrastructural changes occurring in the wall during compression are currently being conducted.

A collaborative microscopy (LM, SEM, TEM) approach was developed to follow the removal and spatial redistribution of extractives (i.e. fats, sterols etc.) from bleached birch kraft pulps, particularly from parenchyma cells. A set of histochemical staining techniques selective for extractives such as sterols and fats were applied to native birch wood and mill sampled unbleached and hydrogen peroxide bleached birch kraft pulps. Observations showed that extractives remaining in parenchyma cells of unbleached pulps were present as spheres and small agglomerates. These structures were larger than the simple pit openings of the parenchyma cells and were thus physically obstructed during the deresination of the pulp. Staining studies were applied to unbleached pulps subjected to washing at low and high dilution factors and showed that extractive removal was more successful with the latter process. These results were interpreted as an effect of an increased soap solubility in the digester washing zone allowing micelles to diffuse more readily through the parenchyma cell walls and pits to allow solubilization of the extractive spheres and agglomerates. Staining results indicated that most of the sterols had been removed from the parenchyma cells in the bleached birch pulps and the material remaining inside the cells was mainly saturated fatty acids. Using the staining approach, numerous particles of unsaturated as well as saturated fatty acids most likely as metal soaps were present outside the parenchyma cells in both the bleached pulp and handsheets of mill refined paper. The relative abundance of sterols and unsaturated fatty acids were found to correlate with amounts detected by GC analysis

7.3 Conclusions

We now have appropriate methods for detailed chemical analysis of wood and fibre samples as small as a few mg. Wood with a composition typical of compression wood was found even in symmetric stems of straight spruce trees. Compression wood was formed increasingly towards the end of the growth season. Compression wood was found only in part of the annual rings, and only in a certain sector of the stem. The profiles of some metals were dramatically different in annual rings where compression wood was formed. The calcium profile across annual rings seems to be related to the formation of mild compression wood in spruce as seen by a distinct profile of galactans and lignin. Both FE-SEM and ToF-SIMS have been applied and evaluated for wood sample analysis. Different drying techniques (AD, FD, CPD and AEND) have been used for sample preparation of wood tissue and compared by both FE-SEM and ToF-SIMS methods. It was found that sample preparation

techniques for FE-SEM are not appropriate for ToF-SIMS analysis of wood tissue. Sample preparation techniques for ToF-SIMS analysis of wood were developed. ToF-SIMS imaging has shown that bordered pit tori are enriched in some metals and have no lignin.

The combined information from GC, histochemical staining and SEM observations made possible not only to understand how much of the extractives were removed during the birch kraft process but also provided details concerning the mechanisms of removal, particularly the micro- and ultrastructural factors affecting deresination.

7.4a Capabilities generated by the project

New optimized sample preparation techniques for ToF-SIMS and SEM has been generated allowing for novel features of the nanostructure of wood cell walls to be visualized. New microanalytical methodologies have also been developed deepening our insights into wood chemistry at the morphological level. Both these aspects will form an important basis for future studies in the research area. An essential part of Elena Tokareva's doctoral thesis has also been completed.

7.4a Utilisation of results

The project has allowed for the development and application of a number of complementary and advanced chemical microanalytical techniques for studies on both solid wood and pulp fibres. The work has emphasized that for a greater understanding of wood cell wall structure and for following changes (e.g. compression wood formation, kraft pulping), gross chemical techniques such as ICP-MS, MS, GC need to be combined with spectroscopical and electron microscopy techniques such as XPS, ToF-SIMS, SEM and TEM. Results from the histochemical studies on the spatial distribution/redistribution and removal of extractives during kraft pulping of birch pulps and the techniques developed are now utilized in certain Swedish mills. The information on the importance of fibre dislocations has also lead to a greater understanding and renewed interest on the consequences these defects can have on final products and paper strength. This has contributed to even greater interest from pulp mills for the improved sorting of raw fibre materials for retaining homogeneity during processing and final product.

7.5 Publications and communication

Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

* Daniel, G., Duchesne, I., Tokoh, C. and Bardage S. 2004. The surface and intracellular nanostructure of wood fibres: Electron microscope methods and observations. In COST Action E20 Wood Fibre Cell Walls: Methods to Study Their Formation, Structure and Properties, Eds: U. Schmitt, P. Ander, J. Barnett, A.M. Emons, G. Jeronimidis, P. Saranpää, and S. Tschegg, Printed by SLU, Uppsala, December 2004. ISBN 91-576-6803-5, pp. 87-104.

* Fardim, P., Gustafsson, J., von Schoultz, S., Peltonen, J., Holmbom, B. 2005. Extractives on fiber surfaces investigated by XPS, ToF-SIMS and AFM. Colloid Surf. A, 255 (1-3) 91-103.

Fardim, P. Holmbom, B. 2005. Origin and surface distribution of anionic groups in different papermaking fibres, Colloids Surf. A. 252 (2-3) 237-242.

* Fernando, D., Lidén, J., Daniel, G. 2005. Staining techniques for determining the state and spatial distribution of extractives in birch kraft pulp. Nord. Pulp Pap. Res. J. 20 (4), 383-392.

Kokkonen, P., Fardim, P., Holmbom, B. 2004. Surface distribution of extractives on TMP handsheets analyzed by ESCA, ATR-IR, ToF-SIMS and ESEM. Nord. Pulp Pap. Res. J. 19 (3), 318- 324.

Terziev, N., Daniel,G., Marklund, A.. 2005. Dislocations in Norway spruce fibres and their effect on the properties of pulp and paper. Holzforschung, 59, 163-169.

Terziev, N., Daniel, G. And Marklund, A. 2007. Effect of abnormal fibres on the mechanical properties of paper made from Norway spruce (*Picea abies* (L.) Karst.) (submitted to Holzforschung).

* Tokareva E. N., Fardim P., Pranovich A. V., Fagerholm H.-P., Daniel, G., Holmbom B. 2007. Imaging of wood tissue by ToF-SIMS: Critical evaluation and development of sample preparation techniques (Submitted to Applied Surface Science)

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Fernando, D., Lidén, J., Daniel, G. 2005. Staining techniques for determining the state and spatial redistribution of extractives during birch kraft pulping. In: International Symposium on Wood Science & Technology (IAWPS 2005), 27-30 November 2005, Yokohama, Japan. pp 286-287.

Fernando, D. Lidén, J., Daniel, G., 2005. The nature and behavior/redistribution of extractives during birch kraft pulping: microscopical histochemical approach. In: proceedings 6th Pacific Regional Wood Anatomy Conference (PRWAC), December 1-5 2005, Kyoto University, Kyoto, Japan, pp 37-38.

Pranovich, A., Konn, J. and Holmbom, B. 2003. Variation in chemical composition of earlywood and latewood in Norway spruce. Proc. 12th ISWFPC, June 9-12, Madison, USA, Vol.3, pp. 203-206.

Pranovich, A., Konn, J. and Holmbom, B. 2004. Spatial distribution of metals across annual growth rings of Norway spruce. Proc. 8th European Workshop on Lignocellulosics and Pulp, Riga, Latvia, pp. 279-282.

Pranovich, A., Konn, J., and Holmbom, B. 2004. Spatial distribution of organic compounds accross annual growth rings of Norway spruce. 4th RCCWS International Symposium (Wood structure, properties and quality '04), St. Petersburg, Russia, Vol. 2, pp. 572-575.

* Pranovich, A., Konn, J. and Holmbom, B. 2005. Variation in spatial distribution of organic and inorganic compounds across annual growth rings of Norway spruce and aspen. Oral presentation,13th ISWFPC, Auckland, New Zealand. Vol. 2, 453-460.

Pranovich, A., Konn, J., Holmbom, B. 2006. Methodology for chemical microanalysis of wood. Proc. 9th European Workshop on Lignocellulosics and Pulp, Vienna, Austria, pp. 436-439 (2nd POSTER AWARD).

Terziev, N., Daniel, G and Marklund, A. 2006. Regeneration features of Norway spruce forests and mechanical properties of paper. In: Proceedings of the 5th Plant Biomechanical Conference – Stockholm, August 28 – September 1, 2006., pp. 175-181 (*Reviewed*). ISBN 01-86018-12-4.

Tokareva, E., Fardim, P., Pranovich, A., Holmbom, B. 2006. Analysis of wood tissues by ToF-SIMS. Proc. 9th European Workshop on Lignocellulosics and Pulp, Vienna, Austria, pp. 557-560 (1st POSTER AWARD).

6. Other scientific publications

Pranovich, A., Konn, J. and Holmbom, B. 2003. Variation in chemical composition of earlywood and latewood in Norway spruce. Annual Conference of Åbo Akademi Process Chemistry Centre, Åbo Akademi, Turku/Åbo, Finland. (Poster)

Pranovich, A., Konn, J., Holmbom, B. 2007. Methodology for chemical microanalysis of wood (Manuscript for submission to special issue of journal Holzforschung in occasion of EWLP 2006).

Tokareva, E., Pranovich, A., Fardim, P., Holmbom, B. 2005. Chemical microscopy of wood tissues. Annual Conference of Åbo Akademi Process Chemistry Centre, Åbo Akademi, Turku/Åbo, Finland. (Poster + short presentation)

Tokareva E. N., Pranovich A. V., Fardim P., Holmbom B. 2006. Surface distribution of wood components in spruce by ToF-SIMS. Oral presentation, 5th European Workshop on Secondary Ion Mass Spectrometry, Muenster, Germany. Book of Abstracts, p. 114

Tokareva, E., Fardim, P., Pranovich, A., Daniel, G., Holmbom, B. 2007. Analysis of wood tissues by ToF-SIMS (Manuscript for submission to special issue of journal Holzforschung in occasion of EWLP 2006).

7.6 National and international cooperation

The project reflects cooperation between Åbo Akademi and SLU/WURC. We do not have an Advisory group. Both groups, at ÅA and SLU, have extensive contacts outside the project, both nationally and internationally.

8 Fibre wall modelling (BUNDLE)

FINAL REPORT	
Name of research project	**BUNDLE, Fibre wall modelling**
Coordinator of the project	KCL

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**Bundle-KCL: Fiber strength and morphology**
Project period	1.1.2003–31.12.2005
Organization in charge of research	Oy Keskuslaboratorio – Centrallaboratorium Ab (KCL)
Sub-project leader	Tarja Tamminen
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	KCL, Box 70, FIN-02151 Espoo, Finland, Tel. +358 9 4371333 Fax +358 9 464305 tarja.tamminen@kcl.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	476 250
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	176 250
Other funding	
Industrial group	300 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school
Tarja Tamminen, Senior Scientist	F	KCL
Rolf Wathén, Product Manager	M	KCL
Olli Joutsimo, Research Engineer	M	KCL
Tiina Liitiä, Senior Scientist	F	KCL

Total person-months of work conducted by the research team: 29
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. degree	University	Supervisor of thesis, supervisor's organization
2005	D.Sc. (Tech.)	M	Olli Joutsimo, 1971, M.Sc. 1999	HUT	Leif Robertsén, KCL
2006	D.Sc. (Tech.)	M	Rolf Wathén, 1975, Lic. Tech. 2003	HUT	Mikko Alava, HUT, Tarja Tamminen, KCL

Name of the sub-project 2	Bundle: HUT/Physics
Project period	1.4.2003–31.12.2005
Organization in charge of research	Helsinki Univ. Tech., Laboratory of Physics
Sub-project leader	Mikko Alava
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	PO Box 1100, HUT 02015, Tel. 050-4132152 Fax 09-4513116 mja@fyslab.hut.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	148 500
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	148 500

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	Funder
Jari Rosti, M.Sc., researcher	M	HUT, National Graduate School in Materials Physics	Tekes
Lauri Salminen, Dr.Tech., researcher	M	HUT	Tekes
Juha Koivisto, under-graduate student, part-time researcher	M	HUT	Tekes
Mikko Alava, docent, team leader	M	HUT	HUT

Total person-months of work conducted by the research team: 43
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. degree	University	Supervisor of thesis, supervisor's organization
2007	M.Sc.	M	Juha Koivisto, 1982	HUT	Mikko Alava, HUT
2007	Dr.Tech.	M	Jari Rosti, 1974	HUT	Mikko Alava, HUT

Name of the sub-project 3	Bundle-STFI Properties of wood components and their rheological behaviour (BUNDLE4)
Project period	1.1.2003–31.12.2005
Organization in charge of research	STFI-Packforsk AB
Sub-project leader	Lennart Salmén
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	STFI-Packforsk AB/Div. Fibre, Pulp & Energy Box 5604, 114 86 Stockholm, Sweden Tel. +468-6767340, fax +468-4115518 lennart.salmen@stfi.se
URL of the project	http://www.stfi-packforsk.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	255 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	122 000
Other funding	
Industrial group	133 000

RESEARCH TEAM

Name, degree, job title [1)]	Sex (M/F)	Organization, graduate school
Lennart Salmén, docent, research manager	M	STFI-Packforsk Ab
Fredrik Berthold, Ph.D., Senior Research Associate	M	STFI-Packforsk Ab
Eva-Lisa Lindfors, Research Assistant	F	STFI-Packforsk Ab
Jesper Fahlén, Ph.D., Research Associate	M	STFI-Packforsk Ab
Margaretha Åkerholm, Ph.D., Research Associate	F	STFI-Packforsk Ab
Lisa Klockare, M.Sc., Research Assistant	F	STFI-Packforsk Ab

Total person-months of work conducted by the research team: 15,2
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name of the sub-project 4	Bundle-Kau, Strain to failure for wood fibres effect of morphology and process (Bundle3)
Project period	1.1.2003–30.6.2006
Organization in charge of research	Karlstad University
Sub-project leader	Fredrik Thuvander
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Karlstad University SE 651 88 Karlstad Tel. +46 54 7002124 Fax +46 54 7001449 fredrik.thuvander@kau.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	119 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Formas	119 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school
Fredrik Thuvander	M	Karlstad University
Peter Lilja	M	Karlstad University
Total person-months of work conducted by the research team 22.4 person-month = full-time work for at least 36 h/week, paid holidays included		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Abstract

Fiber strength is a product of several factors, derived from fiber morphology, deformations and damage. Fiber morphology is defined during the growth of the tree and thus cannot be affected by process, contrary to the other factors. We have identified factors that contribute to fiber strength and clarified how they are affected by chemical and mechanical damage during the process. The results help to understand the role of single fiber on paper performance.

Tiivistelmä

Kuidun lujuuteen vaikuttavat monet tekijät, jotka riippuvat kuidun morfologiasta, deformaatioista ja vaurioista. Kuitumorfologia määräytyy puun kasvun aikana, eikä siihen siten voida vaikuttaa prosessilla, toisin kuin muihin tekijöihin. Olemme tunnistaneet tekijöitä, jotka vaikuttavat kuidun lujuuteen, ja selvittäneet, miten kemiallinen ja mekaaninen vaurioitus prosessin aikana vaikuttavat niihin. Tulokset auttavat ymmärtämään, mikä on yksittäisen kuidun merkitys paperin kestävyydelle.

Sammanfattning

Fiberstyrka påverkas av flera faktorer, vilka beror på fiberns morfologi, deformationer och skador. Fibermorfologin bestäms under trädets växt och kan således inte påverkas med processen, i motsats till de andra faktorerna. Vi har identifierat faktorer som påverkar fiberns styrka, samt redogjort för hur kemiska och mekaniska skador under processen påverkar dem. Resultaten hjälper oss att förstå vilken betydelse den enskilda fibern har för papperets hållbarhet.

8.1 Introduction

Chemical pulp is used to improve the strength of paper products. The pulping process affects fiber properties, both the strength of the single fiber and its performance in fiber network. The relationships between single fiber strength and product strength are very complicated. In addition, fiber strength is difficult to evaluate separately from the network properties. The Bundle project aimed at clarifying these questions.

8.1.1 Background

As a necessary starting point for the project, sound knowledge was needed about the factors influencing fiber strength. One problem in the work was related to the measurement of single fiber strength. Suitable measurement techniques are not widely available, and typically they are quite time-consuming. In Bundle, we used the single fiber fracture (SFF) method at Karlstad University. In addition, we developed methods to extract information on fiber strength from handsheet tests.

Morphological factors, especially the microfibril angle (MFA), affect fiber stiffness and strength. In the outer fiber wall layers, the microfibrils have rather random orientation, but in the thick S2 layer they are more aligned in the axial direction. Low MFA in the S2 layer increases the stiffness of the fiber. The thin outer fiber wall layers are probably important for the z-directional fiber strength rather than axial strength.

In addition to morphological factors, fiber ultrastructure and its components affect fiber strength. Cellulose is the most important chemical component in this respect. It is arranged in a complicated manner in the fiber wall. Cellulose chains in the fibrillar structure form a highly ordered, crystalline structure via hydrogen bonds. Part of the cellulose is in a less ordered form on the fibril surfaces. Cellulose chain length affects fiber strength, but the correlation is not straightforward. Hemicelluloses may also contribute to fiber strength, but their main role is to provide bonding between fibres in paper.

8.1.2 Objectives

The objective of the project was to develop mathematical models that connect the strength and rheological properties of chemical softwood pulp fibers to their chemical composition and morphology.

8.2 Results and discussion

8.2.1 Single fiber strength

In his doctoral thesis, Olli Joutsimo studied the formation and consequences of cell wall damage formed by mechanical force at high temperature at the end of cooking, a phenomenon that normally takes place to some extent at mill fiber line. In intact fiber, the cellulose fibril aggregates form a network with even structure. This kind of network is stiff and capable of carrying load. If the network in the fiber wall is damaged, the fiber is no longer capable for even distribution of stress. This leads to lowered stiffness and strength. Thus, ultrastructural fiber wall damage was recognized as one significant factor contributing to single fiber strength.

Measurements by dynamic FTIR of cellulose accessibility using deuterium showed that only surface areas of the cellulose were affected. The strength giving connections of the cellulose chains were totally unaffected by the changes in the climate. This indicates that cellulose aggregates or fibrils do not have distorted regions, accessible to moisture, that run across the whole cellulose aggregate width. Thus the cellulose fibrils are more or less intact in their length direction why they may be seen as infinite reinforcements in the structure of the fibre wall irrespective of the humidity. This

may well explain the very high strength of the cellulose fibre and that the strength is only marginally affected by moisture.

Chemical extraction and dissolution of hemicelluloses was shown to have only small effects on the fibre stiffness and strength as such. Only when the cellulose structure itself across fibrils was affected was the fibre properties lowered. Deliberate alterations of the aggregate structure were also found difficult to achieve.

Fiber damages naturally deteriorate fiber strength. One type of damage in the ultrastructural level was described above. However, fibers may be damaged also locally. Damaged sites may be formed as a consequence of fiber bending into kinks, or chemically e.g. under acidic conditions. These two also interact, as the acid attack is fastest into damaged sites in the fiber.

The starting point in Bundle was to test and develop further the hypothesis by Berggren /1/, related to the different types (homogeneous vs. heterogeneous) of cellulose degradation. Handsheets were used for the degradation tests to avoid problems related to pulp treatments.

We found that the weak points formed under acidic conditions cleave easily. However, if the fiber segments between the damaged sites are intact, strong fiber network may compensate for the damage, the effect being analogous to shortening of the fibers. Fibers that were weakened slowly and homogeneously under humid heat conditions were stronger than the acid-weakened fibers, when comparing at the same average cellulose chain length (at the same viscosity). This indicates that the neighbouring cellulose chains in the fibrillar structure distribute the load via hydrogen bonding, thus adding to fiber strength.

It is noteworthy that all the handsheets treated under degradative conditions showed the same tensile stiffness. This indicates that fiber stiffness is defined by fiber ultrastructure and is not affected by chemical damage. Contrary to this, the stiffness is negatively affected by mechanical damage of the fiber wall.

By acoustic emission measurements, the acid treated sheets in particular were found to behave in a much more brittle fashion in testing. This agrees with the idea that a substantial fraction of the fibers in the sheets had local damage and thus would break easily, leading to catastrophic sheet failure.

In native fiber, hemicelluloses and lignin are arranged between the cellulose fibrils. After delignification, the main part of lignin is removed as well as large part of the hemicelluloses. In this process, pores are formed into the fiber wall between the cellulose fibrils. The residual lignin and hemicelluloses remain attached onto the cellulose fibril surfaces. This attachment may be strong enough to contribute to fiber strength via a general mechanism shown in Figure 1. According to this hypothesis, the short hemicellulose chains act as bridges between cellulose chains, creating a composite structure. Due to the hemicellulose attachment, the effective polymer chain length is longer and its strength contribution bigger.

We tested this hypothesis experimentally and found support, even if not definitive proof for it. The exact nature of the linkage is not known.

When dissolving softwood fibers in DMAc/LiCl, a significant insoluble residue remains. This fraction was studied by advanced techniques, such as solid state NMR (CP MAS, subcontracting from the University of Helsinki). The results supported the theory that glucomannan is ordered on the cellulose fibril surfaces, hindering its dissolution.

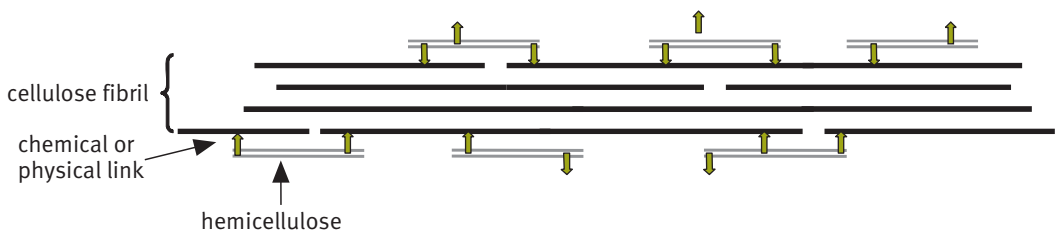

Figure 1. Possible reinforcement of cellulose fibrils by hemicelluloses.

Damaged fibers were more soluble, probably because the glucomannan layer was loosened. This was interpreted to support the contribution from hemicelluloses to fiber strength.

Fiber deformations usually co-occur with damage. However, deformations affect fiber properties differently from damage. Curl as such does not affect fiber strength, even if it has clear effects on network properties. If fiber curl is increased in a controlled manner without inducing fiber damage, a network structure with high strain potential is achieved /2, 3/. In another study, the role of single fibers on the fracture mechanism of paper was clarified by testing oriented sheets /4/. That study confirmed the critical role of stress distribution in the fracture process zone when paper breaks, and thus the significance of fiber form in addition to strength for paper strength.

8.2.2 Evaluation of single fiber strength

Zero-span strength has been assumed to reflect mainly fiber strength, but it has not been clear whether network structure affects the result to some extent, too. In addition, the measured result is the contribution from fibers to the strength of the handsheet, not the strength of the individual fiber. A simulation model was developed to extract the actual single fiber strength parameters from the handsheet tests /5/. The necessary measurement data is the statistical distribution of zero-span measurements, information about the handsheet structure and fiber dimensions. This model involves a simple description of the zero-span geometry. It indicates that the zero-span strength should follow a normal distribution. This seems indeed to be true, but the standard deviation of the measured values is surprisingly high, beyond that predicted by the model. Further tests will be performed in order to find the cause for this variation. One should note that the large variation of zero-span strength has implications for the use of the zero-span test for pulp evaluation in general.

Our belief is that both wet and dry zero span strength reflect single fiber strength rather than network strength. The difference between these values originates from the weakening effect of moisture on the intra- (rather than inter-) fiber hydrogen bonds.

Refining usually improves zero-span strength. This has been interpreted to originate from improved network structure, and thus support the theory of network structure contributing to zero-span strength. However, we have shown that the observed improvement of zero-span strength during refining is due to partial recovery of the damaged cell wall structure. This explains why refining has no effect on the zero-span strength of special laboratory-cooked intact fibers.

Single fiber fragmentation was used to study the variation of fiber strain at failure, the distribution of which can be described using Weibull statistics. With these tools, we could compare the variation in the properties both within one fiber population and between different populations.

As an example, we compared the effects of the acid and humid heat degradative treatments. The acid damaged the structure of the cell wall severely and the strain at which the fiber is likely to fail was low. This is indicative of large critical damages that are effective at low strains. On the contrary, the humid heat treated fibers exhibited a higher likelihood of failure at strains above about 5%. The higher survival rate for the acid treated fiber indicates that the frequency of damages of critical size at strains above 5% is lower in that case. This result thus confirms the localized nature of acid-induced degradation.

8.2.3 Viscoelastic properties

In a composite material such as wood fiber, the contributions of its components to the overall viscoelastic properties may sometimes be difficult to discern. Understanding of the origin of the property changes in this material requires knowledge both of the properties of the individual components as well as knowledge of how they interact in the native structure. Thus, studies were made both on isolated material of cellulose and xylan as well as of the composite material in the form of wood fibers and on fibers that were chemically altered.

Studies have utilized moisture scanning dynamic mechanical testing as well as humidity controlled dynamic-FT-IR (Fourier Transform In-

fra-Red spectroscopy) to deduce how softening affects the interaction between the wood polymers. The results were utilized in fiber wall modeling for better understanding of the property relations.

Measurements on the isolated wood components showed that the hemicelluloses had an elastic modulus of about 2 GPa, about 4 times lower than the only data so far given in the literature. During softening, taking place between 70 and 80% RH at 20 °C, the modulus fell to about 20 MPa. Also amorphous cellulose was shown to have a modulus comparable to the hemicelluloses but showed to be more resistant to moisture showing no tendency for softening below 80 %RH. Utilizing these data it was possible to model the fiber wall properties of the wood fiber with a better agreement with reality than previously has been possible. This comparison showed still some discrepancy between the model and actual measurements in the direction across the fibers. It is probable that the structure on the nanoscale of the fiber wall is more complex than measurements so far has been able to show.

8.2.4 Simulation and modeling

The main modelling effort was done via the construction of a fiber bundle model to extract the single fiber strength statistical parameters from zero-span measurements. Had this been successful, we would have been able to continue towards the modeling of single fiber strength, including in particular the effect of various pulp treatments. However, the much larger than expected variation in the experimental standard deviation of zero-span strength means that the model does not yet explain the results satisfactorily.

8.3 Conclusions

Several factors were identified, which define single fiber strength. However, from the practical point of view, we need to be able to measure these crucial fiber characteristics from the fiber material.

In Bundle, we have induced the desired (from research point of view) weakening effects using specially designed procedures into the fiber, but we did not try to identify the consequent effects by fiber analysis. However, some hints can be suggested based on related studies.

Ultrastructural damage is one of the main causes of decreased fiber strength. The misalignment of cellulose fibrils is accompanied by altered pore size distribution. This is probably the most potential approach when evaluating the degree of this type of damage. Changes in ultrastructure affect even other fiber properties in addition to strength, especially its water retention ability. Even for this purpose, it is important to define a practically applicable protocol for the measurement of this fiber property.

Local fiber damages decrease fiber strength in an analogous way as shortening of fiber length. Kinks may be an indication of potential sites of fracture. However, all weak points are probably not visible in the fiber directly. Ander /6/ has proposed a technique that might be applicable for this purpose. He uses acid treatment and mild refining to reveal the weak points in fiber, and then detects the degree of fiber cleavage by fiber length analysis.

Degradation of cellulose in fiber may be homogeneous or heterogeneous. A special case of heterogeneous degradation is the local weakening of fiber, discussed above. In this kind of degradation, minor average decrease in cellulose chain length induces significant strength loss. If the degradation is homogeneous, typically induced by alkaline conditions, fiber may retain its strength in spite of severe degradation. Cellulose degradation can be detected by the viscosity measurement. Due to the complexity of the cellulose degradation patterns, there is no universal correlation between viscosity and fiber strength. However, if the cause of the cellulose degradation is known, correlation between fiber strength and viscosity exists. Therefore, viscosity is a useful measurement for fiber strength, with certain limitations.

Future developments of mathematical models for single fiber strength should include as degradation mechanisms both the local and more homogeneous possibilities discussed above. This means that one should explicitly include local defects (as created in the acid treatment) and the statistical loss of microfibril strength due to cellulose degradation. This points to the question of how to measure local fiber defects, including their real

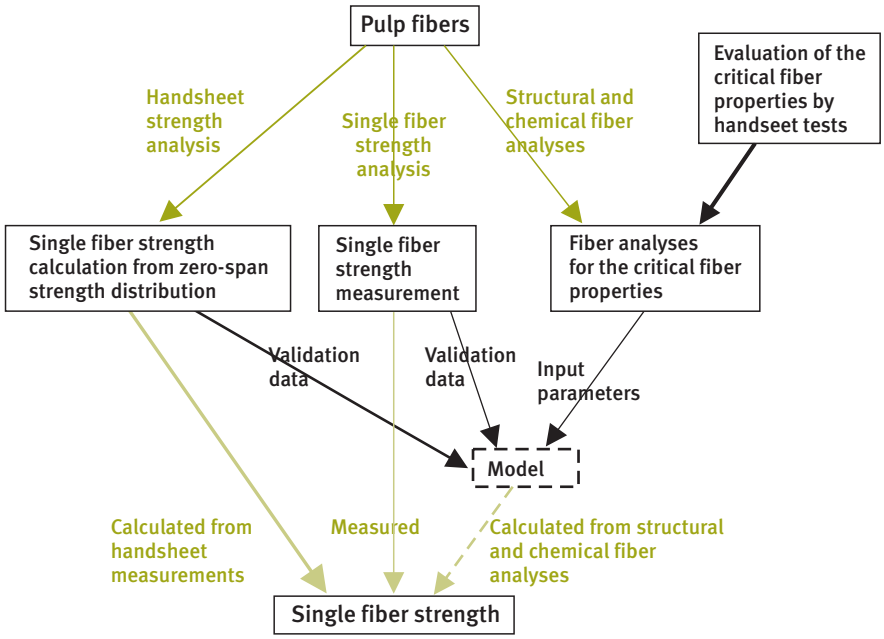

Figure 2. Approaches towards measuring and modelling single fiber strength. The line widths in the figure reflect the emphasis of the various tasks in the project.

strength-reducing effect. If the mathematical model is to be developed further in the future, it should use the critical fiber strength related analytical data described above as input parameters.

The current status of the project is illustrated in Figure 2. In it we have outlined a variety of possible ways of characterizing fibers, both individually and as a part of a sheet. The "thick" arrows are indicative of relationships that have been studied in the project. The role of fiber ultrastructure in determining fiber strength can be studied naturally as such, or via the question how it affects typical mechanical testing quantities, such as zero-span strength or sheet strength and fracture toughness. In other words, one can investigate how strong individual fibers are, and how that is reflected in paper strength. We have made good progress in some aspects, as evaluating critical properties via handsheet tests and understanding the relation of single fiber strength and zero-span test behavior, and the role of various fiber treatments in these. Unfortunately, not enough real single fiber strength statistical data became yet available, which hampered the development of the intended fiber strength model.

8.4a Capabilities generated by the project

One doctoral thesis was published in 2005 and one in 2006. A third doctoral thesis and a Master's thesis will be published in 2007.

The project has generated knowledge related to the evaluation of paper strength. The results help in assigning the significance of each individual factor to the overall strength of single fiber and paper.

8.4b Utilisation of results

Strength is one of the key parameters required from a chemical pulp fiber used for reinforcement purposes. This project has identified the main factors contributing to fiber strength, also pointing to critical process stages or conditions that may deteriorate fiber strength.

By improving the quality of the chemical reinforcement pulp, its amount in the paper furnish can be reduced. This leads to savings in raw material costs. In fine papers, this leads additionally to improved smoothness and opacity of the paper surface. In this case, the same effect may be reached by improvement in the hardwood pulp fiber strength.

8.5 Publications and communication

Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

Wathén, R., Batchelor, W., Westerlind, B., Niskanen, K. Analysis of strains in the fracture process zone, Nord. Pulp Pap. Res. J., 20(2005)4, pp. 392-398

*Wathén, R., Rosti, J., Alava, M., Salminen, L. and Joutsimo, O. Fiber strength and zero-span strength statistics –some considerations, Nordic Pulp and Paper Research Journal, 21(2006)2:193-201.

*Salmén, L., Micromechanical understanding of the cell wall structure, C. R. Biologies 327(2004)9-10:873-880.

Koivisto, J., Rosti, J., Salminen, L.I., Alava, M.J. Creep of a fracture line in paper peeling, submitted to Physical Review Letters

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Salminen, L.I., Rosti, J., Alava, M.J. Acoustic emission or crackling noise in paper fracture, 8th experimental chaos conference, Firenze, June 14-17, 2004, AIP Conf. Prog. No., (2005) 115-121

Wathén, R., Westerlind, B., Batchelor, W. and Niskanen, K., Determining the critical strain in the fracture process zone by different methods, International Paper Physics Conference, Sept. 7-11 2003, Victoria, Canada, p. 19-25

*Wathén, R., Joutsimo, O., Tamminen, T. Effect of different degradation mechanisms on axial and z-directional fiber strength, Advances in Paper Science and Technology: 13th fundamental research symposium, Cambridge, 11-16 Sept. 2005, edited by S. J. l'Anson, vol. 1, pp 631-647

Joutsimo, O., Wathén, R., Robertsen, L. Meaning of fiber deformations and damage from fiber strength to end user, Advances in Paper Science and Technology: 13th fundamental research symposium, Cambridge, 11-16 Sept. 2005, edited by S. J. l'Anson, vol. 1, pp 591-611

Olsson, A.-M., Salmén, L. The softening behavior of hemicelluloses related to moisture, ACS Symp ser. 864 "Hemicelluloses: Science and technology" Eds. P. Gatenholm, M. Tenkanen, American chemical Society, Washington, (2004) p. 184-197.

*Salmén, L. Ultra-structural arrangement and rearrangement of the cellulose aggregates within the secondary cell wall. In "Proc.of Fifth Plant Biomechnaics Conf." Ed. L. Salmén, STFI-Pf, (2006) p. 215-220.

3. Articles in Finnish and Swedish journals with referee practice

*Joutsimo, O., Wathén, R., Tamminen, T. Effects of fiber deformations on pulp sheet properties and fiber strength, Pap. Puu, 87(2005)6, pp.392-397.

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice

5. Scientific monographs

Joutsimo, O. Effect of mechanical treatment on softwood kraft fiber properties. Doctoral thesis, Department of Forest Products Technology, Laboratory of Pulp Technology, HUT, Espoo, Finland, 2004.

Wathén, R. Studies on fiber strength and its effect on paper properties. Doctoral thesis, Department of Forest Products Technology, Laboratory of Forest Products Chemistry, HUT, Espoo, Finland, (2006)

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Wathén, R., Liitiä, T., Joutsimo, O., Tamminen, T. Contribution of material properties to fiber strength, 8th European workshop on lignocellulosics and pulp, Aug 22-25, 2004, Riga, Proceedings 291-294

Liitiä, T., Hortling, B., Honkonen, P., Sneck, A., Joutsimo, O., Tamminen, T. Fiber morphology responsible for restricted solubility of softwood kraft pulp into LiCl/DMAc, 13th International symposium on wood, fiber and pulping chemistry, Auckland, New Zealand, May 16-19, 2005, Proceedings vol. II, 371-375

Joutsimo, O., Wathén, R., Robertsen, L., Tamminen, T. The influence of fibre ultrastructural damage on pulp and paper properties, 60th Appita annual conference and exhibition, Melbourne, Australia, Apr 3-5, 2006, 191-197.

Rosti, J. oral presentation at the Statistical Physics in Mechanics, Grasse, France (2006)

Rosti, J. oral presentation at the Euromech Colloquium 486, Deformation and Fracture Processes in Paper and Wood Materials, Sundswall, Sweden (2006)

Bonenfant Boris, Mick Mathieu, Comparison of Probability of Survival by Weibull Parameters for 3 Fibre Types, Karlstad University Report, 2006.

b) Other dissemination

Reporting in the Wood Material Science Program: Year Book 2004, pp 49–52, Year Book 2005, pp 64–71, Year Book 2006, pp 81-90, Presentation at the program board meeting 2005 and program seminar 2006.

Meetings with the industrial contact group twice per year.

Article about Rolf Wathén's doctoral thesis work in Tekniikka ja Talous 25.1.2007, p 19.

8.6 National and international cooperation

Members of the contact group

Tea Sunden, Sunila Oy
Veikko Jokela, Stora Enso, Imatra
Kalle Ekman, Stora Enso, Imatra
Pirkko Liias, Metsä-Botnia, Rauma
Camilla Wikström, Metsä-Botnia, Äänekoski
Kirsi Riekkinen, UPM-Kymmene, Lappeenranta
Susanne Eriksson, Korsnäs, Gävle
Hans-Inge Frölind, Billerud, Skärblacka
Karin Sjöström, Södra Cell, Mörrum
Christine Hägström-Näsi, Tekes
Leena Paavilainen, Wood Wisdom, Helsinki

The project covered both strength and rheological aspects of fiber. Studies related to fiber strength were performed mainly as cooperation between KCL, HUT and KaU. STFI-Packforsk concentrated mainly on rheological aspects.

References

/1/ Berggren, R. Cellulose degradation in pulp fibers studied as changes in molar mass distributions. Doctoral thesis, Department of Fibre and Polymer Technology, KTH, Stockholm 2003, 183 p.

/2/ Joutsimo, O., Wathén, R., Tamminen, T. Effects of fiber deformations on pulp sheet properties and fiber strength, Pap. Puu, 87(2005)6, pp.392-397.

/3/ Joutsimo, O., Wathén, R., Robertsen, L. Meaning of fiber deformations and damage from fiber strength to end user, Advances in Paper Science and Technology: 13th fundamental research symposium, Cambridge, 11-16 Sept. 2005, edited by S. J. l'Anson, vol. 1, pp 591-611.

/4/ Wathén, R., Batchelor, W., Westerlind, B., Niskanen, K. Analysis of strains in the fracture process zone, Nord. Pulp Pap. Res. J., 20(2005)4, pp. 392-398.

/5/ Wathén, R., Rosti, J., Alava, M., Salminen, L., Joutsimo, O. Fiber strength and zero-span strength statistics –some considerations, Nordic Pulp and Paper Research Journal, 21(2006)2:193-201.

/6/ Ander, P., Daniel, G., Garcia-Lindgren, C., Marklund, A. Characterization of industrial and laboratory pulp fibres using HCl, Cellulase and FiberMaster analyses, Nordic Pulp and Paper Res. J. 20(1) 2005, 84-89.

THE SUB-PROGRAMME FOR INNOVATION TARGETED RESEARCH & DEVELOPMENT PROJECTS

9 Value-added products from barks of Nordic wood species using bioconversion and chemical technology (WoodBiocon)

FINAL REPORT

Name of the research project	**Value-added products from barks of Nordic wood species using bioconversion and chemical technology (WoodBiocon)**
Coordinator of the project	Tiina Nakari-Setälä

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Fractionation and chemistry of bark components**
Project period	1.1.2004–31.3.2007
Organization in charge of research	Åbo Akademi
Sub-project leader	Bjarne Holmbom
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Åbo Akademi University Laboratory of Wood and Paper Chemistry Porthaninkatu 3, FI-20500 Turku, Finland Tel. +358 2 215 4229 Fax +358 2 215 4868 bjarne.holmbom@abo.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	435 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	250 000
VINNOVA	185 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Bjarne Holmbom, Prof.	M	Åbo Akademi		Åbo Akademi
Jarl Hemming, M.Sc.	M	Åbo Akademi		Tekes
Christer Eckerman, M.Sc.	M	Åbo Akademi		Tekes

Reija Harlamow, student	F	Åbo Akademi		Tekes
Anca Balas, M.Sc.	F	Åbo Akademi	Technical University Iassy, Romania	EU Marie Curie Fellowship 4 months (EU contract with Åbo Akademi Process Chemistry Centre)
Göran Gellerstedt, Prof.	M	KTH		Vinnova
Liming Zhang, Ph.D.	M	KTH		Vinnova
Total person-months of work conducted by the research team 70 person-month = full-time work for at least 36 h/week, paid holidays included				

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	M.Sc.	F	Reija Harlamow, 1981	Åbo Akademi University	Bjarne Holmbom, Åbo Akademi University

Name of the sub-project 2	**Microbial conversions**
Project period	1.1.2004–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Tiina Nakari-Setälä
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O.Box 1000 FI-02044 VTT, Finland Tel. +358 20 722 5134 Fax +358 20 722 7071 tiina.nakari-setala@vtt.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	452 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	273 000
Other funding	
VTT	179 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Tiina Nakari-Setälä, Ph.D., Technology Manager	F	VTT		Tekes, VTT
Markku Saloheimo, Ph.D., Docent, team leader	M	VTT		Tekes, VTT
Pasi Halonen, Ph.D., research scientist	M	VTT		Tekes, VTT
Hanna Kontkanen, Ph.D., research scientist	F	VTT		Tekes, VTT
Jaana Uusitalo, Msc, research scientist	F	VTT		Tekes, VTT
Ismo Mattila, Msc, research scientist	M	VTT		Tekes, VTT
Anu Tamminen, Msc, research scientist	F	VTT		Tekes, VTT
Anu Vaari, MSc (Tech), research scientist	F	VTT		Tekes, VTT
Riitta Nurmi, technician	F	VTT		Tekes, VTT
Eila Leino, technician	F	VTT		Tekes, VTT

Total person-months of work conducted by the research team 35
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2008	M.Sc.	F	Mari Häkkinen, 1983	University of Helsinki	Tiina Nakari-Setälä, VTT

Name of the sub-project 3	**Development of enzymatic and chemical modification processes**
Project period	1.1.2004–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Johanna Buchert
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O.Box 1000 FI-02044 VTT, Finland Tel. +358 20 722 5146 Fax +358 20 722 7071 johanna.buchert@vtt.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	912 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	347 000
VINNOVA	330 000
Other funding	
VTT	235 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Johanna Buchert, Prof.	F	VTT		Tekes, VTT
Marjaana Rättö, Msc, research scientist	F	VTT		Tekes, VTT
Hanna Kontkanen, Ph.D., research scientist	F	VTT		Tekes, VTT
Riitta Leppänen, technician	F	VTT		Tekes, VTT
Salme Koskimies, Ph.D., Docent	F	VTT		Tekes, VTT
Pauliina Pitkänen, graduate student	F	VTT		Tekes, VTT
Juha Karttunen, technician	M	VTT		Tekes, VTT
Mikael Lindström, Ph.D.	M	STFI-Packforsk		Vinnova
Tommy Iversen, Ph.D.	M	STFI-Packforsk		Vinnova

Total person-months of work conducted by the research team 70

person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	M.Sc.	F	Pauliina Pitkänen, 1980	University of Helsinki	Salme Koskimies, VTT

Abstract

WoodBiocon project has aimed at exploiting low-value bark materials of Nordic tree species as a source for valuable chemicals. During the project suitable extraction and modification processes have been developed for bark components using both biotechnological processing by enzymes and microbial cells, and chemical modification. Selected bark components have been produced for chemical characterization, further modifications and application studies, including e.g. suberin, stilbenes and tannins. In addition, optimal biocatalysts for bioconversion processes have been screened, and selected enzymes have been produced in large quantities for application studies. Experiments with available commercial biocatalysts indicated that enzymes can be used for modification of the natural polyester suberin, e.g. polymerization of purified suberin monomers and hydrolysis of the polymer to oligo and monomeric components. Chemical methods have been developed for selective oligoester synthesis.

Tiivistelmä

WoodBiocon-projektin tavoitteena on ollut hyödyntää pohjoisten puulajien kuorta kemikaalien tuotannossa. Projektissa on tutkittu puun kuoren kemiaa lupaavimpien komponenttien löytämiseksi sekä kehitetty erotusmenetelmiä, mm. suberiinille, stilbeeneille ja tanniineille. Lisäksi on kehitetty sekä bioteknisiä että kemiallisia menetelmiä kuoresta eristettyjen komponenttien jatkojalostamiseksi korkea-arvoisemmiksi tuotteiksi. Projektissa on kehitetty uusia biokatalyytteja kuorikomponenttien jalostamiseksi ja mm. uusia entsyymejä on tuotettu sovellustutkimusta varten. Projektissa on havaittu, että koivun kuoren suberiinia voidaan muokata entsymaattisesti. Entsyymeillä voidaan sekä polymeroida suberiinihappoja että niillä voidaan hajottaa suberiinipolymeeriä. Projektissa on myös kehitetty kemiallisia menetelmiä, joilla suberiinirasvahapoista voidaan syntetisoida oligoestereitä.

Sammanfattning

WoodBiocon-projektets syfte har varit att utnyttja lågvärdig bark av nordiska trädslag som en källa för värdefulla kemikalier. I projektet har lämpliga extraktions- och modifieringsmetoder utvecklats för barkkomponenter genom användning av både biotekniska processer med enzymer och mikrobiella celler samt kemisk modifiering. Barkkomponenter har isolerats för kemisk karakterisering, modifiering och applikationsstudier, bl.a. suberin, stilbener och tanniner. Därtill har optimala biokatalysatorer för biokonvertering isolerats och vissa enzymer har producerats i större mängder för applikationsstudier. Försök med kommersiellt tillgängliga biokatalysatorer visade att enzymer kan användas för modifiering av den naturliga polyestern suberin, bl. a. för polymerisering av renade suberinmonomerer och hydrolys av polymeren till oligomera och monomera komponenter. Kemiska metoder har utvecklats för selektiv syntes av oligoestrar.

9.1 Introduction

9.1.1 Background

Bark is an important part of trees and is produced in large amounts by pulp and paper mills and saw mills world-wide. To date the major use of bark is as a fuel. However, bark is a potentially valuable raw material from which various compounds, among others, suberin, betulinol, stilbenes and tannins could be separated. Many of these compounds, or components thereof, have application potential as additives, composite ingredients, and specialty chemicals in the pulp and paper, biotech, chemical and pharmaceutical industries. Softwood barks also contain considerable amounts of carbyhydrates. The application potential of extracted bark components could be further increased using biotechnological conversion by enzymes or microbes, and by chemical modification. These conversion processes may, for example, increase the structural diversity or lead to compounds with novel functional or structural properties.

9.1.2 Objectives

The main objective of the project was to exploit bark, a low-value pulp and paper and saw mill by-product, as a source of valuable chemicals. The project aim was to develop processes for separation and conversion of wood bark components into value-added products. A major target was to evalu-

ate the exploitation potential, as well as the technical and economical feasibility, of developed products and processes

9.2 Results and discussion

9.2.1 Fractionation and chemistry of bark components

Understanding of the chemistry of bark is necessary for broader exploitation of various bark components. In addition, methods to separate selected components are needed. The barks of the four main Nordic tree species, spruce, pine, birch and aspen, were studied in the project in order to produce chemically well characterized bark extracts and pure components for further modification and application studies.

The chemical composition of the inner and outer bark differs largely and the two bark fractions were therefore studied separately, when possible. Birch bark is special in its physical properties, which allows separation into inner and an outer bark fractions by flotation in water, even in an industrial scale. Aspen bark can not be fractionated in the same manner because the outer bark is too thin. There is neither a simple separation method nor any definite distinction between inner and outer bark sections in spruce. Therefore, the inner soft and light-brown material was separated by hand cutting from the darker hard material located on the outer part of the bark. The separated inner and outer barks of spruce, pine and aspen were further fractionated by sequential extractions to obtain enriched extracts with varying chemical compositions.

Spruce and pine bark

The chemical composition of the different bark fractions of spruce and pine has not been known in detail previously. All bark samples, viz. inner and outer bark, collected during winter and summer respectively, were successively extracted with different solvents and the yields of the extracts and the insoluble residues together with their composition are listed in Table 1. The corresponding winter and summer samples were found to be very similar, whereas the composition of inner and outer bark showed distinct differences.

The spruce inner bark was found to contain a higher amount of acetone-soluble components than the outer bark. The predominant components were found to be a mixture of stilbene glucosides together with a low-molar-mass tannin fraction (Figure 1). The stilbene glucosides astringin (1), isorhapontin (2) and piceid (3) were present in the approximate ratio of 7:2:0.7, and the amount of the

Table 1. Yields of the extracts in per cent of the original dry bark (Picea abies).

Solvent	Winter bark		Summer bark		Major components
	Inner	Outer	Inner	Outer	
Petroleum ether	4.0	3.1	1.8	5.2	Resin acids, fatty acids,
CH_2Cl_2	1.2	1.4	0.6	2.6	terpenoids
Acetone	15.1	7.0	17.6	7.9	Stilbene glucosides, tannin
Water	12.3		10.0		Glucose, fructose, sucrose
		7.0		6.8	Glucose, fructose, sucrose, tannin
Acetone/water (2:1)	1.7	8.0	1.9	7.9	Condensed tannin
Residue	65.7		68.1[1)]		"Cellulose"
		73.5		69.6[2)]	Polysaccharides, polymeric tannin-lignin (BMTL)

1) A dominance of glucan (~70%) with minor or trace amounts of other neutral sugar components. In addition, a minor amount of a tannin-lignin complex

2) A dominance of a tannin-lignin complex (>50%) with neutral sugars present in fair (glucose) or minor amounts.

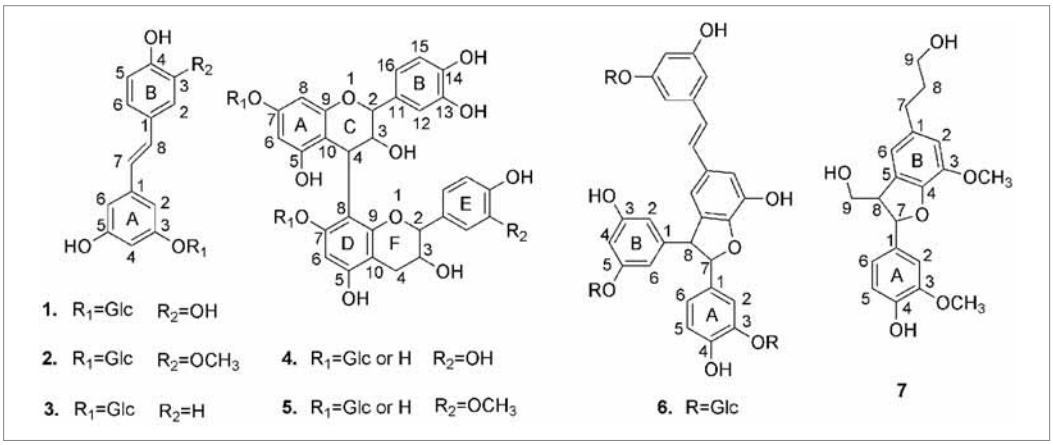

Figure 1. Identified components in an acetone extract from inner bark (Picea abies).

main components, astringin and isorhapontin, was 10% by weight of inner bark. By treatment with β-glucosidase, the free stilbenes, known to have biological activity, could be obtained. The amount of stilbenes in spruce inner bark did not show extensive seasonal variation (Figure 2).

The stilbene glucosides can be easily extracted also with water at low temperatures, monomeric and dimeric sugars being the major impurities (Figure 3). At higher temperatures, the coextraction of hemicellulose sugars and tannins increases. Good water extractability allows the development of an industrial extraction process for stilbenes.

The acetone-water extract was found to contain a condensed tannin with a higher abundance in the outer bark as compared to the inner bark. Based on NMR data, the structure shown in Figure 4 can be suggested, i.e., with some phenylpropane (lignin)

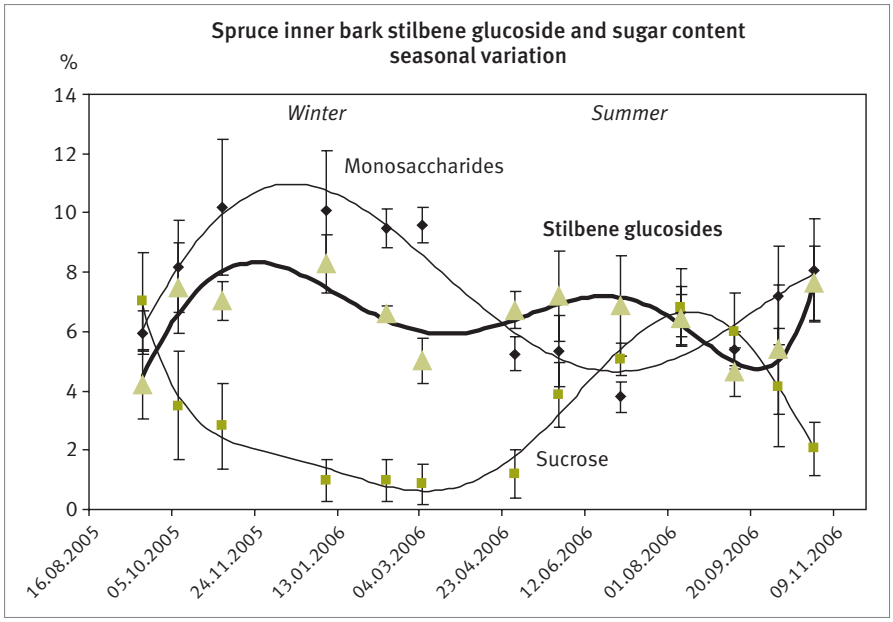

Figure 2. Seasonal variation in stilbene glucosides and sugar content of spruce inner bark.

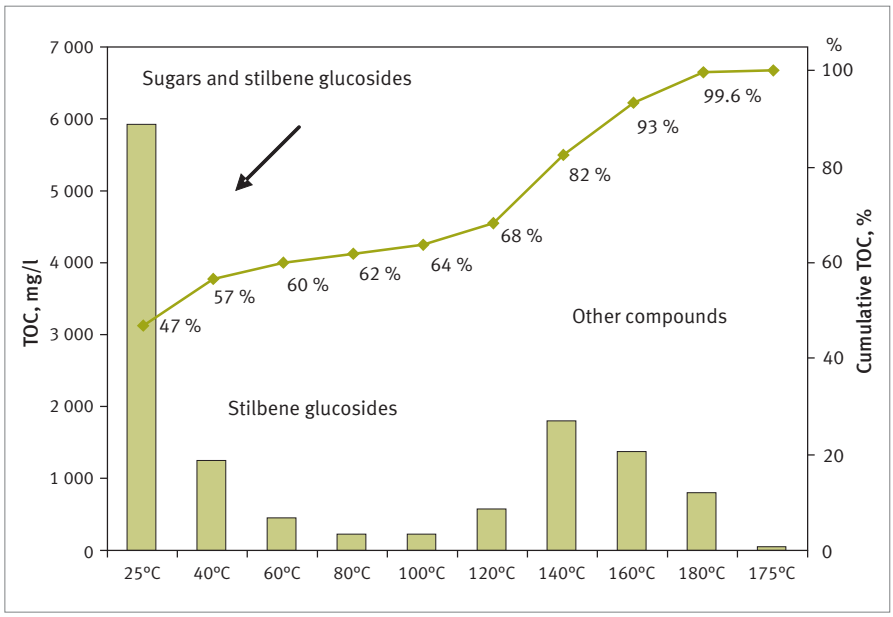

Figure 3. Consecutive water extraction of spruce inner bark at different temperatures. Extraction conditions 2 times 5 min. static cycles, 2000 psi.

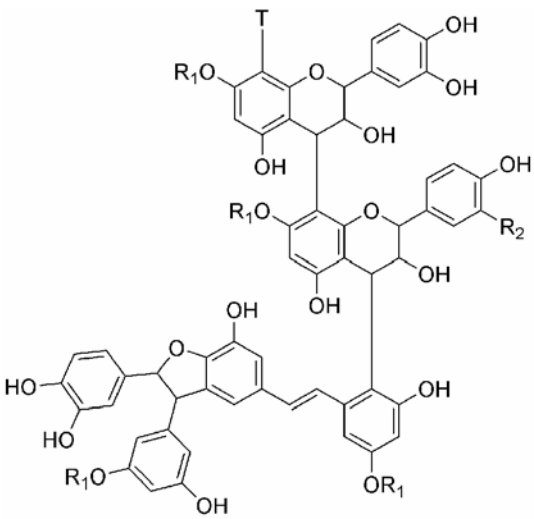

Figure 4. Suggested structure of the condensed tannin present in the outer bark (Picea abies). R_1 = glucose or H; R_2 = OH or OCH_3; T = tannin.

units attached to the catechin-stilbene backbone. In addition, glucose units are abundant.

After the complete sequential extraction, a substantial amount of insoluble material (~70%) was still present in both inner and outer bark. The inner bark residue contained mainly glucose (cellulose), whereas the outer bark residue contained a tannin-lignin complex with a structure similar to that shown in Figure 4 but with a much larger number of attached guaiacylpropane (lignin) units in addition to the glucose units.

In pine, the amounts of extractives and low-molar-mass sugars were found to be quite similar to spruce. Pine does not contain any stilbene glucosides, but contains a corresponding flavonoid glucoside fraction. When going from inner to outer bark, the amount of condensed tannin with the presence of glucose units strongly increased in analogy to spruce and, again, methoxyl groups were also present indicating the occurrence of lignin units. The major difference between the two wood species may therefore be the presence/absence of stilbene units, both as low-molar-mass glucosides and as high-molar-mass tannin.

Aspen bark

Aspen bark has been previously poorly characterized. Chemical analysis of aspen bark from freshly cut trees was performed. Figure 5 summarizes the gravimetric results (w/w) of ASE-extracted aspen total bark.

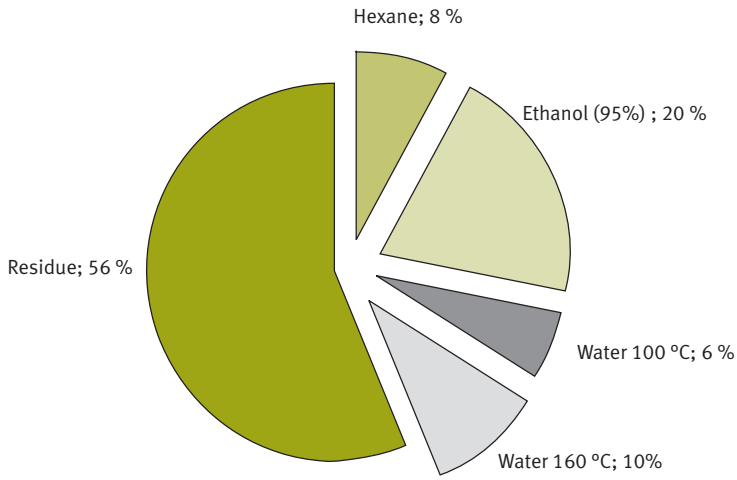

Figure 5. Aspen total bark extracts (w/w).

The ethanol-water extract containing mainly glucosides made up about 20% of the dry bark weight. No individual compound was dominating in this extract and the amount of free phenolics was very low. While typical lipophilic extractives and mono-functional long-chain fatty acids were found in the hexane extract, hemicellulose sugars were the major constituents in the water extracts and in the bark residue. The amount of suberin-type acids in the bark residue was low and consequently aspen bark is not a good source for suberin. In summary, aspen bark contains a wide variety of components but all in relatively low concentrations.

Birch bark

The composition of birch bark is fairly well known based on the literature. Birch outer bark is known to be a rich source of betulinol and suberin. *Tables 2 and 3* summarize the amounts of these main components in outer bark. Betulinol and suberin have been purified in preparative scale from bark collected from a birch kraft pulp mill. A sequential extraction procedure with ethanol and a technical-grade hydrocarbon yielded 85% pure betulinol in a yield of over 95%. The developed method could be used for production of betulin even at technical scale. The outer bark residue after extraction of betulin contains mainly suberin and can be depolymerised by mild alkali to yield a crude mixture of suberin monomers. The major suberin monomer 9,10-epoxy-18-hydroxy-octadecanoic acid was purified to 97% purity for further modification studies. Also 22-hydroxydocosanoic and docosane-1,22-dioc acid were been prepared in gram-scale at purities of 85 %.

Table 2. Major triterpenoids in birch outer bark (kg/t).

Betulinol	210
Lupeol	21
Betulinic acid	10
Erythrodiol	7
Other	18

Table 3. Major suberin acids in birch outer bark (kg/t).

9,10-epoxy-18-hydroxyoctadecanoic	124
22-hydroxydocosanoic	48
18-hydroxyoctadec-9-enoic	35
9,10,18-trihydroxyoctadecanoic	26
docosane-1,22-dioic	21
octadec-9-ene-1,18-dioic	12

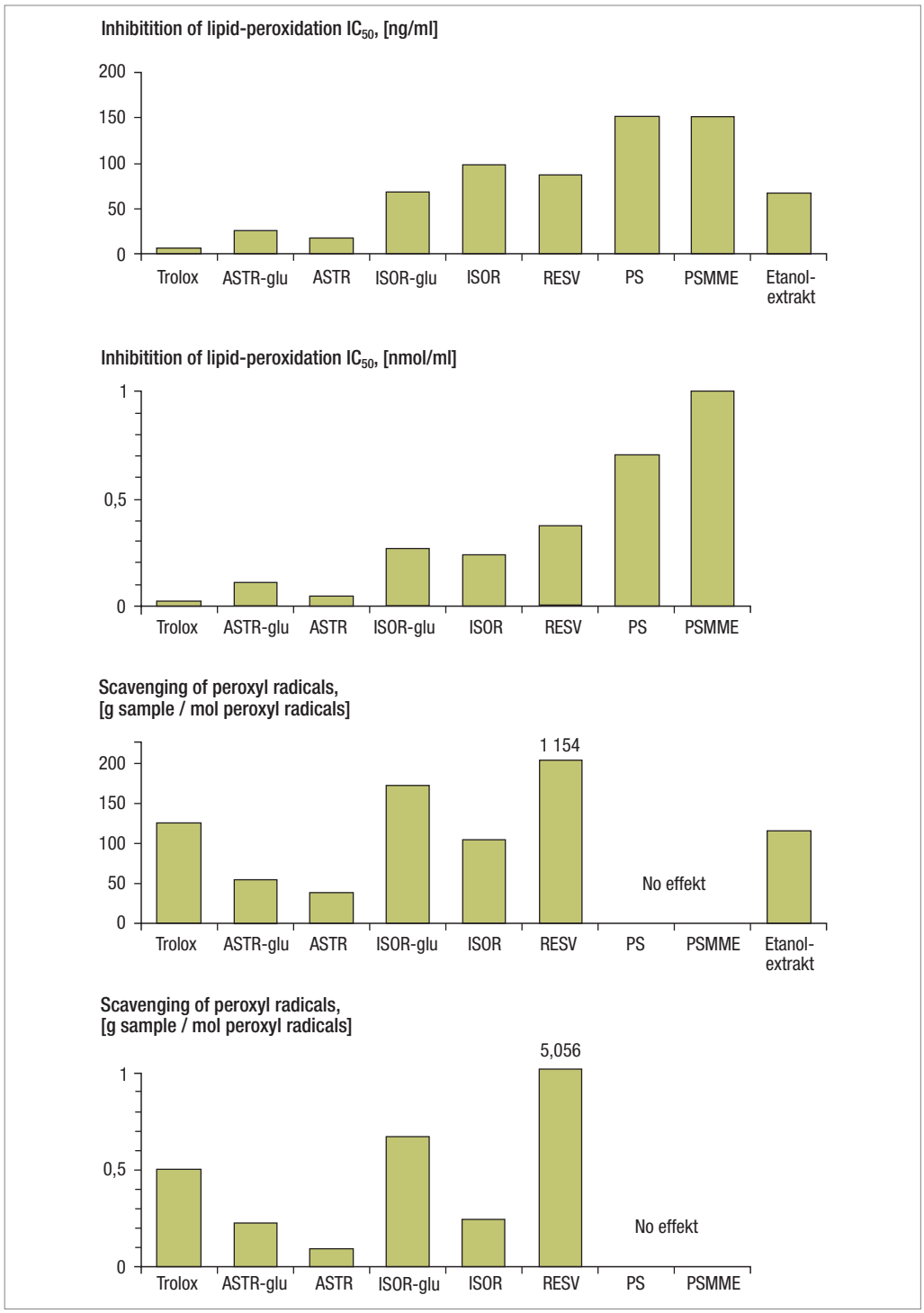

Figure 6. Inhibition of lipid peroxidation and radical scavenging by stilbenes. Trolox, vitamin E standarad; ASTR, astringin, ASTR-glu, glucoside of astringin; ISOR, isorhapontin; ISOR-glu, glucoside of isorhapontin; RESV, resveratrol; PS, pinosylvin; PSMME, pinosylvin monomethyl ether; Ethanol extrakt, spruce inner bark ethanol extract.

9.2.2 Bioactivity of spruce stilbenes

Spruce stilbenes were isolated at 95-98% purity by ethanol extraction and reverse-phase flash chromatography, and their bioactivity was compared with resveratrol and the stilbenes in pine heartwood, pinosylvin and pinosylvin monomethyl ether. The stilbene glucosides isorhapontin and astringin were extracted from spruce inner bark and the aglucones of these stilbenes, astringenin and isorhapontigenin, were produced by enzymatic treatment using β-glucosidase. The unfractionated ethanol extract, rich in stilbenes, was also used in the biotests.

The results from radical scavenging and lipid peroxidation experiments (Figure 6) showed that the spruce bark stilbenes are strong antioxidant. The number of hydroxyl groups has a decisive role for the antioxidant effect. Astringenin and astringin were the most prominent antioxidants. Stilbenes were also specific against some bacteria, but did not inhibit the growth of a wider range of different microorganisms. Pinosylvin showed the highest and broadest antimicrobial effect. The cytotoxicity of stilbenes was analysed using mouse hepatoma cell line Hepa-1 by determining cell viability as relative total protein concentration (TPC) of the samples. All the tested aglucone stilbenes showed the same cytotoxicity as pinosylvin and pinosylvin monomethyl ether, while the glucosides were considerably less cytotoxic.

9.2.3 Chemical and enzymatic processes for modification of birch bark suberin

One of the main targets in the project has been to develop novel enzymatic, chemical and chemo-enzymatic methods for fractionation and modification of bark-derived suberin biopolymer. Both partial or complete hydrolysis and partial polymerization have been addressed.

Suberin separated from birch outer bark has been alkali hydrolyzed to produce samples for further product development. A simple, direct NMR method was also developed, which made it possible to analyse suberin fatty acids quantitatively as well as to study the reactivity of different functional groups (COOH, different OH-groups and epoxide) in various systems. A typical suberin acid distribution is presented in Table 4. The NMR

Table 4. Typical suberin fatty acid distribution determined by NMR, and comparison of NMR and GC-MS techniques.

Monomer	NMR (mol-%)[VTT]	GC-MS (mol-%)[x]
HO $OC(CH_2)_7CH = CH(CH_2)_7COOH$	16	4.9
HO $CH_2(CH_2)_7CH = CH(CH_2)_7COOH$		13.2
HO $CH_2(CH_2)_7CH$–$CH(CH_2)_7COOH$ \ / O	37	40.3
HO $CH_2(CH_2)_6CH(CH_2)_7COOH$ | OH	2	3.6
HO $CH_2(CH_2)_7CH$–$CH(CH_2)_7COOH$ | | OH OH	18	8.2
Other monomers without secondary functionalities*	27	29.8

* = cannot be identified by NMR
x = calculated from R. Ekman et al., Paperi ja Puu 67 (1985) 255

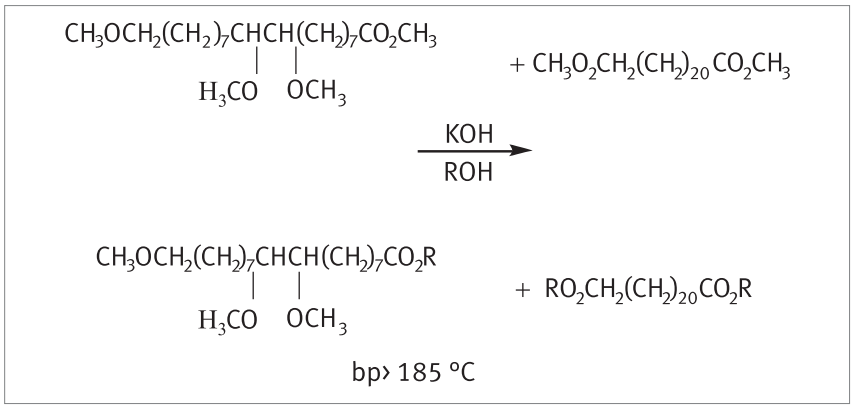

Figure 7. Preparation of oligoesters via in situ intermediates.

method proved to be more quantitative than a regular GC-MS method. An *in situ* synthesis for methylester - ether intermediates was developed (Figure 7). The intermediates were successfully used as a starting material and reacted further to various industrially important ester oligomers and polymers such as lubricants and binders.

Enzymatic polymerization of purified monocomponent suberin fatty acids has been studied to obtain highly functional green polyesters. Polycondensation (Figure 8) of *cis*-9,10-epoxy-18-hydroxyoctadecanoic acid (1), the major suberin monomer in birch outer bark, were performed at 75°C in organic solvents and at 85°C in bulk using immobilized *Candida antarctica* lipase B as a catalyst. The polycondensation performed in toluene in the presence of molecular sieves gave the polyester (2) with the highest molecular weight (M_w 20 000, reaction time 68 h, M_w/M_n 2.2). A nearly as high molecular weight at shorter reaction time (M_w 15 000, reaction time 3 h, M_w/M_n 2.2) was obtained by bulk polymerization in an open vial without any drying agent present.

The aim of enzyme-aided suberin depolymerisation is to selectively hydrolyse suberin to low DP oligomeric fragments without loosing any labile functionalities present in the initial raw material. In testing of several commercial lipase and esterase preparations for suberinolytic activity one esterase preparation capable of suberin hydrolysis was detected. GC-MS analysis of enzymatic hydrolysate of birch outer bark suberin showed that various fatty acids, hydroxy acids and e.g. 9,10-epoxy-18-hydroxy-octadecanoic acid were liberated by the enzyme. Various substrate pre-treatments, including steam explosion, extrusion and enzymatic removal of residual carbohydrate polysaccharides, were applied to increase suberin accessibility. However, only up to 1,5 % of birch outer bark suberin was hydrolyzed by the commercial esterase. These

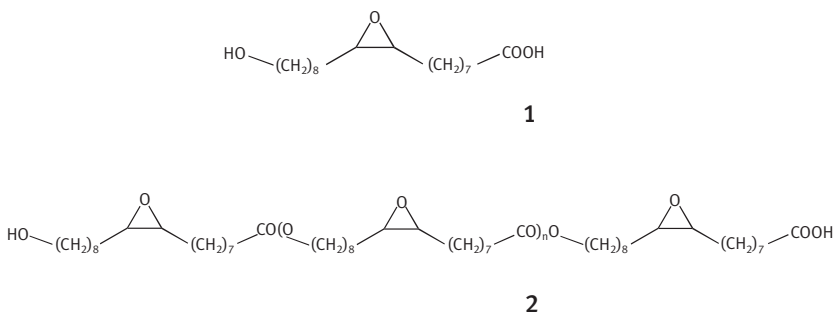

Figure 8. Candida antarctica lipase B catalyzed polymerization of cis-9,10-epoxy-18-hydroxyoctadecanoic acid (1) to give the corresponding epoxy-functionalized polyester (2).

results demonstrate the fact that enzymatic depolymerization of suberin is likely to require a multitude of enzymes due to the heterogeneity of the target polymer.

9.2.4 Development of novel biotools for modification of bark components

Enzyme and cell biocatalysts are active in water-containing solutions, and offer advantages such as selectivity and mild reactions conditions. The aim has been to identify enzyme catalysts and microbes that use bark materials and processed bark fractions as substrates and convert them into value-added chemicals. Especially novel enzymes acting on polymeric suberin to hydrolyze it into oligo and/or monomeric components have been screened. Also, microbial biotransformation of selected bark extractives, and the sugar fraction in softwood barks have been addressed.

A selection of 55 microbes, mostly filamentous fungi isolated from trees and woods, have been screened to identify novel suberin hydrolysing enzymes. The aim was to obtain enzymes with varying activities which in combination would allow efficient hydrolysis of suberin into functional oligomers. Many of the selected microbial strains had never been cultivated in liquid cultures before, and cultivation methods were first developed. In addition, screening procedures based on small molecular weight model substrates were develop to analyse suberin degrading esterase activity in the culture media. Naphtol substrates and a fluorescently labelled aromatic compound were used to screen hydrolysis of the suberin aliphatic and aromatic domains. Finally, radioactively labelled suberin was used as a substrate for the final hit verification by detecting the enzymatic release of radiolabelled entities from the native-like suberin. Also, GC-MS was used to analyse the suberin hydrolysis products. Several microbes were identified that produced suberinase activity.

In the next step, genes encoding the suberinase enzymes needed to be identified. This proved to be laborious for microbes without published genomes, and could not be accomplished by using e.g. a proteomics approach. The identification of suberinase genes in two fungi with available genome data, and the over-expression of the corresponding enzymes for application studies are on-going.

Biotransformation of selected extractives using microbial cells has been addressed. The focus has been on specific oxidation reactions. Selected strains were tested in liquid and solid state cultivations containing pre-treated spruce bark, and pure betulinol or sitosterol. The samples taken from the cultures were extracted with different solvents and analysed by metabolite profiling using GC-MS. In the case of each of the substrates studied, the identification of putative transformation products proved to be difficult based on GC-MS data alone, and structure determination by NMR is needed. The work is on-going.

Bioethanol production from steam pre-treated (different temperatures with and without SO_2 impregnation) technical spruce bark has been carried out in shake flask and bioreactor scale. Various process concepts were compared, including simultaneous and separated enzyme hydrolysis and fermentation. The pre-treated bark material was not too toxic either to the enzymes or to the yeast. However, it seems that enzymatic hydrolysis had not been efficient enough and the ethanol yields turned out to be relatively low. Therefore, tailoring the enzymatic hydrolysis of bark sugars to monosaccharides, including finding the optimal enzyme mixture and dosage, is needed. It also seems that pre-treatment of the material was incomplete and needs to be further studied.

9.3 Conclusions

The project emphasis has been on the characterization and production of selected bark components, and on development of suitable modification processes for these components for application studies. Birch suberin and suberin monomers have been produced in hundreds of grams and used for chemical and enzymatic modification experiments. It has been shown that an immobilized *Candida antarctica* lipase B is an efficient catalyst for the polycondensation of *cis*-9,10-epoxy-18-hydroxyoctadecanoic acid to give epoxy functionalized polyesters with high molecular weights.

Experiments with available commercial biocatalysts indicate that enzymes can be used for suberin processing. The hydrolysis efficiency is,

however, low with a monocomponent enzyme and it is expected that a synergistic action of several enzymes is needed for break down of suberin. We have been able to identify novel putative suberinolytic enzymes but their biochemical characterization and exploitation in suberin processing needs to be verified.

We have been able to develop a selective chemical method for oligoester synthesis from suberin acids, and the produced oligoesters have properties suitable for lubricant and binder products. In addition, studies have been carried out to explore the exploitation of softwood bark sugar fraction using bioethanol as an product. Bottle necks in the bioethanol process, such as pre-treatment and enzymatic hydrolysis of the bark raw material, have to be solved to increase process feasibility.

Chemistry of Nordic tree barks have been studied to further find suitable components for value-added products. Aspen bark contains a wide variety of components but all in low concentrations, and is thus an unattractive raw material. The major components present in spruce bark are stilbene glucosides, simple sugars and a complex tannin ranging from low to very high molecular mass. The tannin was found to contain catechin and stilbene structures with incorporated phenylpropane units. The latter increased in relative amount with increasing molecular mass. In pine a similar distribution was found but instead of stilbenes, flavonoids were present. Some spruce stilbenes were isolated in gram amounts and shown to be antioxidant. Microbial biotransformation of selected bark extractives was addressed but characterization of the putative transformation products proved to be laborious.

9.4a Capabilities generated by the project

Pitkänen, P., Koskimies, S., Heiskanen, N., Hulkko, J., Yli-Kauhaluoma, J., Wähälä, K., Menetelmä karboksyylihappoesteriseoksien valmistamiseksi ja niiden käyttö (A method to prepare mixtures of carboxylic acid ester mixtures and their use). FI Pat. Appl. FI20055568

9.4b Utilisation of results

The results will be transferred and implemented in EU FP6 Specific targeted research project "New concepts for upgrading pulp and cork mill waste streams to value-added chemicals (WaCheUP)" to advance one further step towards industrial implementation. IPR generated in the project will be developed and exploited together with the industrial partners of the project.

9.5 Publications and communication

a) Scientific publications

1. Articles in international scientific journals with referee practice,

Ann Olsson, Mikael Lindström, Tommy Iversen (2007) Lipase Catalyzed Synthesis of an Epoxy-Functionalized Polyester from the Suberin Monomer cis-9,10-Epoxy-18-hydroxyoctadecanoic Acid. Biomacromolecules 8. Asap article.

Pietarinen, S.P., Willför, S.M., Ahotupa, M.O., Hemming, J. and Holmbom B.R. (2006), Knotwood and bark extracts: strong antioxidants from waste materials. *J. Wood Sci.* 52: 436-444.

Välimaa, A-L., Honkalampi-Hämäläinen, U., Pietarinen, S., Willför, S., Holmbom, B. and von Wright, A. (2006): Antimicrobial and cytotoxic knotwood extracts and related pure compounds and their effects on food-associated micro-organisms. *Int. J. Food Microbiol.*, published online first: http://dx.doi.org/10.1016/j.ijfoodmicro.2006.10.031.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Zhang, L. and Gellerstedt, G. (2007) Chemical composition of bark from Norway spruce by 2D heteronuclear (1H-13C) single quantum correlation (HSQC) NMR analysis. In Characterization of Lignocellulosic Materials (Ed. T.Q. Hu), Blackwell Publishing Ltd. In press.

Harlamow, R., Willför, S., Hemming, J., Honkalampi-Hämäläinen, U., von Wright, A. and Holmbom, B. (2007) Extraction and biotests of stilbenes in spruce inner bark. Proceedings of the Meeting 50 years of the Phytochemical Society of Europe, Cambridge, UK, 11-14 April. In press.

3. Articles in Finnish and Swedish journals with referee practice

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice

5. Scientific monographs

Pitkänen, Pauliina (2006) Suberiinin hyödyntäminen teollisuuden raaka-aineena (Utilization of suberin as raw material for industry). MSc thesis, University of Helsinki

Harlamow, Reija. (2006) Utvinning och biotestning av stilbener i granbark (Isolation and biotesting of stilbenes from spruce bark). MSc thesis. Åbo Akademi

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

b) Other dissemination

A radio review of Tiina Nakari-Setälä on exploitation of bark components in YLE1 in June 2006.

Several newspaper articles has refered to WoodBiocon project, including Skogsland No 2/3 January 2006.

LoA Teknisk och ekonomisk tidskrift för lantbruket, bilaga i Landsbygdensfolk No 7 February 2006. Nyteknik http://www.nyteknik.se/art/46195

Oral presentations given

Tommy Iversen. Presentation of the WoodBiocon project at Wood Material Science Seminar, 11.4. 2005, Stockholm, Sweden.

Tiina Nakari-Setälä. Biotekniikan uudet sovellukset biomassan hyödyntämisessä. Clean Word seminar, 27.10.2005, Espoo, Finland.

Göran Gellerstedt. On the chemical composition of bark from Norway spruce (Picea abies). Pacifichem 2005, 15.-20.12.2005, Honolulu, USA.

Tiina Nakari-Setälä. Presentation of the WoodBiocon project at Wood Material Science Seminar 6.4.2006, Helsinki, Finland

9.6 National and international cooperation

The industrial advisory board of WoodBiocon

Lars Gädda, M-Real, chairman
Veikko Jokela, Anders Brolin, StoraEnso
Åsa Forss / Louise Staffas, Södra Cell
Helena Tufvesson, Korsnäs
Ulf Hotanen, UPM-Kymmene
Walter Rüf, Mondi Packaging Paper
Antti Hamunen, Raisio Benecol, vice chairman
Reijo Aksela, Kemira

In 2005 the activities were widened to cork material components through cooperation with the EU-STREP WaCheUp. In this network collaboration with University of Aveiro and University of Minho, Portugal, has been started leading to

1. joint development of suberin derivatives.
2. collaboration on structural analysis of microbial biotransformation products of bark extractives, mainly triterpenoids
3. successful use of methodologies developed in WoodBiocon to process cork raw materials.

VTT has collaborated with Prof. Wolfgang Zimmerman (Chemnitz University of Technology, Germany) in developing methods to measure suberinase enzyme activity and effects of suberinase activity directly on the surface of the suberin polymer.

10 New Cellulose Derivatives from Wood for High Value Products (NewCell)

FINAL REPORT

Name of the research project	**New Cellulose Derivatives from Wood for High Value Products (NewCell)**
Coordinator of the project	Professor Sigbritt Karlsson (KTH)

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Cellulose Accessibility**
Project period	1.1. 2004–31.12.2006
Organization in charge of research	KTH, Fibre and Polymer Technology
Sub-project leader	Assoc. Professor Monica Ek
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	KTH, Fibre and Polymer Technology, Div. Wood Chemistry and Pulp Technology, Teknikringen 56, SE-100 44 Stockholm, Sweden Tel. +46 8 790 8104 Fax +46 8 790 6166 monica.ek@polymer.kth.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	1 078 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	146 280
VINNOVA	835 000
Other funding	
VTT funding	97 520

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Monica Ek, Ph.D., Assoc. Professor, Sub-project Leader	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Sigbritt Karlsson, Ph.D., Professor	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Viviana Hermosilla,	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA

Ann-Charlotte Engström	F	KTH, Fibre and Polymer Technology, Sweden	KTH/VINNOVA
Matti Siika-aho, Senior Scientist	M	VTT Technical Research Centre of Finland, Finland	
Stina Grönqvist, Scientist	F	VTT Technical Research Centre of Finland, Finland	
Pertti Nousiainen, Ph.D., Professor	M	TUT, Fibre Materials Science, Finland	
Päivi Talvenmaa, M.Sc.	F	TUT, Fibre Materials Science, Finland	
Marja Saloniemi	F	TUT, Fibre Materials Science, Finland	
Pedro Fardim, Professor	M	ÅA, Fibre and Cellulose Technology, Finland	
Jan Gustafsson, Academic Lecturer	M	ÅA, Fibre and Cellulose Technology, Finland	
Roland Agnemo, Ph.D., Assoc. Professor, Senior Scientist	M	Domsjö Fabriker AB, Sweden	
Göran Kloow, Ph.D., R&D Manager	M	CPKelco AB, Sweden	
Leif Karlson, Ph.D.	M	Akzo Nobel Surface Chemistry AB, Sweden	
Hans Henrik Øvrebø	M	Borregaard, Norway	
Camilla Wikström	F	Oy Metsä-Botnia AB, Finland	
Pirkko Liias	F	Oy Metsä-Botnia AB, Finland	
Total person-months of work conducted by the research team 100 person-month = full-time work for at least 36 h/week, paid holidays included			

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007 (to be earned)	Tech.Lic.	F	Ann-Charlott Engström (1972) M.Sc. (2002)	KTH	Assoc. Prof. Monica Ek, KTH
2008 (to be earned)	Tech.Lic.	F	Viviana Hermosilla (1976) M.Sc. (2004)	KTH	Assoc. Prof. Monica Ek, KTH

Name of the sub-project 2	Controlled Synthesis
Project period	10.1. 2004–31.3.2007
Organization in charge of research	University of Helsinki (UH)
Sub-project leader	Prof. Sirkka Liisa Maunu
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	University of Helsinki, Laboratory of Polymer Chemistry, P.O. Box 55, FI-00014 University of Helsinki, Finland Tel. +358 9 191 50323 Fax +358 9 191 50330 sirkka.maunu@helsinki.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	1 175 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	340 000
VINNOVA	835 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sirkka Liisa Maunu, Ph.D., Professor, Sub-project leader	F	UH, Laboratory of Polymer Chemistry, Finland		UH
Heikki Tenhu, Ph.D., Professor	M	UH, Laboratory of Polymer Chemistry, Finland		UH
Miia Hiltunen, M.Sc., Research Scientist,	F	UH, Laboratory of Polymer Chemistry, Finland		UH/TEKES
Harri Jokinen, M.Sc., Research Scientist	M	UH, Laboratory of Polymer Chemistry, Finland		UH/TEKES
Tommi Virtanen, M.Sc., Research Scientist	M	UH, Laboratory of Polymer Chemistry, Finland		UH/TEKES
Sigbritt Karlsson, Ph.D., Professor	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Sara Axelsson, M.Sc., Ph. D. Student	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Anu Moilanen, Ph.D., R&D Manager	F	CPKelco, Finland		
Ismo Lokinoja, M.Sc., R&D Manager	M	CPKelco, Finland		
Göran Kloow, Ph.D., R&D Manager	M	CPKelco, Sweden		

Bengt Wittgren, Ph.D., Professor	M	AstraZeneca R&D, Sweden
Leif Karlson, Ph.D.	M	Akzo Nobel Surface Chemistry AB, Sweden
Kjell Stridh	M	Akzo Nobel Surface Chemistry AB, Sweden
Peter van der Horst	M	Akzo Nobel Surface Chemistry AB, Sweden

Total person-months of work conducted by the research team 103
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2009 (to be earned)	Ph.D.	F	Miia Hiltunen, 1977 M.Sc. 2001	UH	Prof. Sirkka Liisa Maunu, Universtiy of Helsinki
2007 (to be earned)	M.Sc.	M	Simon Riihelä, 1981	UH	Prof. Sirkka Liisa Maunu, University of Helsinki

Name of the sub-project 3	**Properties of New Derivatives**
Project period	1.4.2004–31.3.2007
Organization in charge of research	KTH
Sub-project leader	Professor Sigbritt Karlsson
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Prof. Sigbritt Karlsson Royal Insitute of Technology (KTH) Teknikringen 56-58 SE-100 44 Stockholm, Sweden Tel. +46 (0)8 790 85 81 sigbritt@polymer.kth.se
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	847 866
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	7 720
VINNOVA	835 000
Other funding	
VTT funding	5 146

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sigbritt Karlsson, Ph.D., Professor, Sub-project Leader	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Jonas Enebro, M.Sc., Ph. D. Student	M	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Dane Momcilovic, Ph.D., Scientist	M	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Monica Ek, Ph.D., Assoc. Professor	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
Sirkka Liisa Maunu, Ph.D., Professor	F	UH, Laboratory of Polymer Chemistry, Finland		
Matti Siika-Aho, Senior Research Scientist	M	VTT Technical Research Centre of Finland		
Göran Kloow, Ph.D., R&D Manager	M	CP Kelco Sweden		
Roland Agnemo, Ph.D., Assoc. Professor, Senior Scientist	M	Domsjö Fabriker AB, Sweden		
Bengt Wittgren, Ph.D., Professor	M	AstraZeneca R&D, Sweden		
Leif Karlson, Ph. D.	M	AkzoNobel Functional Chemicals, Sweden.		

Total person-months of work conducted by the research team 57
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007 (to be earned)	Tech.Lic.	M	Jonas Enebro, 1979 M.Sc. 2003	KTH	Prof. Sigbritt Karlsson, KTH

Name of the sub-project 4	**Applications of New Derivatives**
Project period	10.1.2004–31.3.2007
Organization in charge of research	Tampere University of Technology (TUT)
Sub-project leader	Päivi Talvenmaa
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Tampere University of Technology P.O. Box 589, FIN 33101 Tampere, Finland Tel. +358 3 3115 2549 Fax +358 3 3115 2955 paivi.talvenmaa@tut.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	835 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	835 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Päivi Talvenmaa M.Sc. (Tech.), Senior Scientist	F	TUT, Fibre Materials Science, Finland		
Pertti Nousiainen, Ph.D., Professor	M	TUT, Fibre Materials Science, Finland		
Marja.Saloniemi	F	TUT, Fibre Materials Science		
Pedro Fardim, Ph.D., Professor	M	ÅAU, Laboratory of Fibre and Cellulose Technology, Finland		
Jan Gustafsson, D.Sc (Tech.),Acad. Lecturer	M	ÅAU, Laboratory of Fibre and Cellulose Technology, Finland		
Nasir Ali, M.Sc. Research Scientist	M	ÅAU, Laboratory of Fibre and Cellulose Technology, Finland		
Monica Ek, Ph.D., Assoc. Professor	F	KTH, Fibre and Polymer Technology, Sweden		KTH/VINNOVA
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Total person-months of work conducted by the research team 82

person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc, D.Sc., ect. degree	University	Supervisor of thesis, supervisor's organization
2005	M.Sc.	F	Elina Orblin, 1976	ÅA	Prof. Pedro Fardim, ÅA Prof. Bruno Lönnberg, ÅA

Abstract

In the project, new knowledge on various pre-treatment methods, e.g. mechanical, chemical, and/or enzymatic, to increase the accessibility of cellulosic materials to swelling and reactive agents at different hierarchical levels, has been developed. It was demonstrated that enzymes are very effecient at increasing the cellulose reactivity, and the effects of monocomponent cellulases, cellulase mixtures and other cellulose acting proteins have been analysed in particular. One challenge was to find new reliable methods to measure the reactivity and accessibility of the hydroxyl groups on cellulose.

A heterogeneous slurry process was chosen and applied to prepare carboxymethyl cellulose (CMC) with a target degree of substitution (DS) by which the effects of the pre-treatment methods could be analysed. Reversible addition-fragmentation chain transfer (RAFT) agents with carboxylic acid function were synthesized and used further in esterification reactions with hydroxyl groups of CMC, ethyl hydroxyethyl cellulose (EHEC) and pretreated cellulose to prepare cellulose based graft-copolymers.

Matrix assisted laser desorption/ionization time-of-flight mass spectrometry (MALDI- TOFMS) was evaluated chemical structure characterization of cellulose derivatives. By the use of selective hydrolysis, with purified endoglucanases, it was possible to reveal information on the substitution pattern in CMC. Methods to prepare electrospun fibres, cellulosic fibrous casings, cellulosic sponges and beads, were developed with target applications in pharmaceuticals and foods. Encapsulation of model drug in nanofibres during the electrospinning was tested and analysis of the release properties is ongoing.

Tiivistelmä

Selluloosamateriaalien aksessibiliteetin lisäämiseksi tutkittiin eri menetelmiä, kuten mekaanista, kemiallista ja/tai entsymaattista esikäsittelyä, turpoamisen ja reagenssien imeytymisen parantamiseksi eri hierarkisilla tasoilla. Erityisesti monokomponenttisten sellulaasien, sellulaasiseoksien tai muiden selluloosaa aktivoivien proteiinien käyttö osoittautuivat tehokkaiksi esikäsittelytavoiksi. Lisähaasteena oli myös löytää uusia luotettavia menetelmiä, joilla määrittää selluloosan hydroksyyliryhmien reaktiivisuus tai aksessibiliteetti.

Karboksimetyyliselluloosaa (CMC) valmistettiin heterogeenisella liuosprosessilla useista käsittelemättömistä ja eritavoin esikäsitellyistä liukoselluista tavoitteena tietty substituutioaste.

Karboksyylihappo-funktionaalisia RAFT-reagensseja valmistettiin ja käytettiin edelleen karboksimetyyliselluloosan (CMC), etyylihydroksietyyliselluloosan (EHEC) ja esikäsitellyn selluloosan hydroksyyliryhmien esteröinnissä ja selluloosapohjaisten oksa-kopolymeerien valmistamisessa.

MALDI-TOFMS menetelmän soveltuvuutta selluloosajohdosten rakenteiden määritykseen testattiin. CMC:n substituutioasemien selvittämisessä hyödynnettiin selektiivistä happohydrolyysiä ja puhdistettuja endoglukanaaseja osana analyysiprosessia.

Tuotteiden käyttöalueita ovat sähkökehrätyt kuidut, selluloosakuitukuoret, selluloosapohjaiset sienet, kalvot ja helmet, biolääketieteelliset materiaalit, farmaseuttiset liuokset sekä elintarvikkeissa ja maaleissa käytettävät lisäaineet.

Sammanfattning

Inom projektet har ny kunskap om mekaniska, kemiska och enzymatiska förbehandlingar utvecklats för att öka tillgängligheten i cellulosamaterial för vidare reaktioner på olika hierarkiska nivåer. Upprenade cellulaser, cellulasblandningar och andra cellulosaaktiva proteiner har visat sig vara effektiva förbehandlingar. En utmaning var att finna lämpliga metoder för att mäta reaktiviteten och tillgängligheten på hydroxylgrupperna i cellulosa. En heterogen slurryprocess användes för att framställa karboxymetylcellulosa (CMC) med en förbestämd substitutionsgrad (DS) som målsättning, dels från obehandlad, dels från förbehandlad cellulosa. Med den här CMCn kunde effekterna av förbehandlingsmetoderna studeras.

”Reversible addition-fragmentation chain transfer” (RAFT)-reagens med karboxylsyrafunktionaliteter framställdes och användes till esterfikationsreaktioner med fria hydroxylgrupper i CMC, etylhydroxyetylcellulosa (EHEC) och förbehandlad cellulosa för framställning av cellulosabaserade ymp-sampolymerer.

Matris assisterad laser desorption/jonisering flykttidsmasspektrometri (MALDI-TOFMS) utvärderades för karaktärisering av den kemiska strukturen hos cellulosaderivat. Selektiv hydrolys, med hjälp av upprenade endoglukanaser, i kombination med MALDI-TOFMS visade sig vara en användbar strategi för att erhålla information om substitutionsmönstret i CMC.

Metoder för elektrospinning av fibrer, cellulosafiberförpackningar, cellulosabaserade svampar och pärlor har utvecklats. Detta möjliggör framtida applikationer i läkemedel och livsmedelsprodukter. Inkapsling av en modellsubstans under elektrospinning har provats och försök pågår med att utvärdera frisättningsbetingelserna för denna produkt.

10.1 Introduction

10.1.1 Background

A high accessibility is an essential prerequisite for a homogeneous or designed substitution of cellulose material. The accessibility of cellulose in wood pulps to solvent and reactants is usually limited. Only the surfaces and the amorphous regions of the cellulose fibrils are available for reactions.

Commercial cellulose derivatives often contain inhomogeneities in their structures resulting in poor performance of the products. A first aim was to create homogeneously substituted carboxymethyl cellulose (CMC). CMC was chosen since it is one of the most important and widely applied cellulose ethers. To obtain more homogeneous cellulose derivatives it is also important to find homogeneous modification conditions and to use controlled techniques to derivatise cellulose. Pretreated cellulose and chosen cellulose derivatives were therefore modified further using living radical polymerisation reactions. Hydroxyl groups in starting materials were substituted in a way that facilitates the further polymerisation reactions. As a result a series of cellulose based graft-copolymers were prepared.

The properties of cellulose derivatives are influenced by several parameters related to the chemical structure and the molecular size. Therefore it is important to determine the molar mass and molar mass distribution, the degree of substitution (DS) and the substitution pattern along the cellulose polymer in order to foresee the performance of the material in a specific application.

In this project the chemical structure was investigated by MALDI-TOFMS, nuclear magnetic resonance (NMR) spectroscopy and various chromatographic techniques (e.g. HPLC, HPAEC-PAD) whereas the molar mass and molar mass distribution was determined by size-exclusion chromatography with online multi-angle light scattering and refractive index detection (SEC/MALS/RI).

The supra-molecular structures and technical properties were investigated by different microscopy techniques and rheology measurements.

10.1.2 Objectives

The project was divided into four sub-projects with the overall objectives:

- Preparations of cellulose derivatives with controlled chemical and supramolecular structure for sophisticated pharmaceutical and fine chemical applications

- Development of knowledge about improvement of the accessibility of cellulose
- Use of new controlled free-radical polymerization reactions on various cellulosic materials to develop new cellulose derivatives
- Characterization of new cellulose derivatives by chemical and physical means (MALDI-TOFMS, SEC/MALS/RI, NMR etc.)
- Application of the new derivatives from wood into a series of high-value products.

10.2 Results and discussion

10.2.1 Cellulose accessibility

Various methods to analyse accessibility and reactivity have been evaluated, i.e. Fock method, Iodine Sorption, alkali solubility (R10) and Swelling coefficient. A new method to evaluate the results of the pre-treatments is under development.

Chemical, mechanical and enzymatic pre-treatments to increase the accessibility of cellulose materials to swelling and reactive agents have been investigated. By the Fock's method, a micro-scale process similar to the viscose process, an increase in cellulose yield after enzymatic treatment with endoglucanases was demonstrated. Different factors may contribute to the mechanisms for this improvement in reactivity, i.e. swelling of the cell wall, endoglucanase affinity to cellulose II or lowering the degree of polymerization.

The relationship between viscosity and acid hydrolysis was also evaluated (Fig 1). The results indicated that the increase in reactivity according to Fock was not only due to a decrease in the degree of polymerization. The results of the molecular weight distribution, by SEC, support this assumption (Fig 2).

The influence of a monocomponent endoglucanase without a cellulose binding domain was also evaluated. The reactivity reached using a D-type enzyme is less than what is obtained when a C-type enzyme is used.

Additional endoglucanases, other proteins and commercial enzymes were also studied. The best results in terms of increased water accessibility

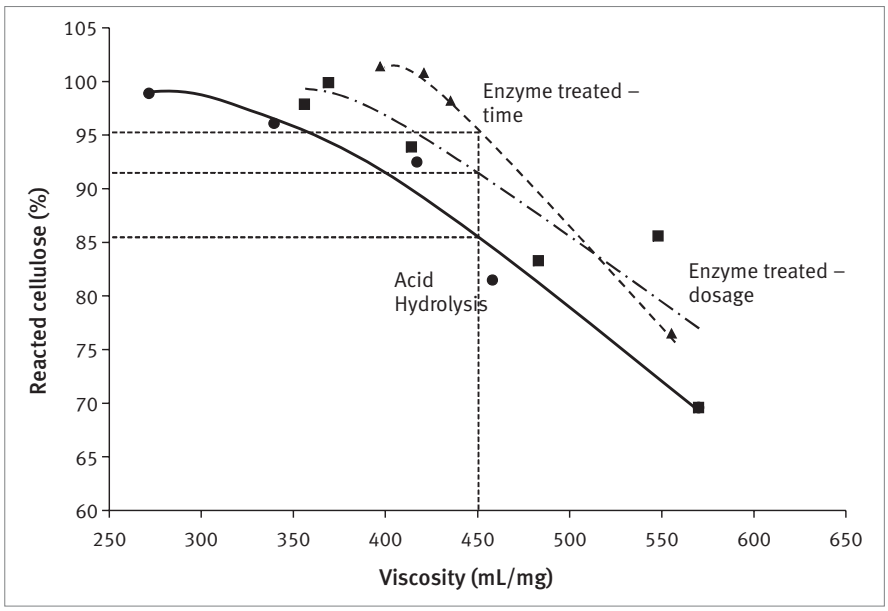

Figure 1. Reactivity according to Fock versus viscosity of dissolving pulp samples: (▲, -----) treated with endoglucanase with different incubation times (from the left in the figure 90, 30, 10 minutes and the reference), (■, - · - ·) treated with endoglucanase with different enzyme dosage (from the left in the figure 50, 30, 5, 0.5, 0.05 and 0 ECU/g dry weight pulp), (●,—) treated by acid hydrolysis for 30, 15, 8, 2 and 0 minutes (from the left in the figure).

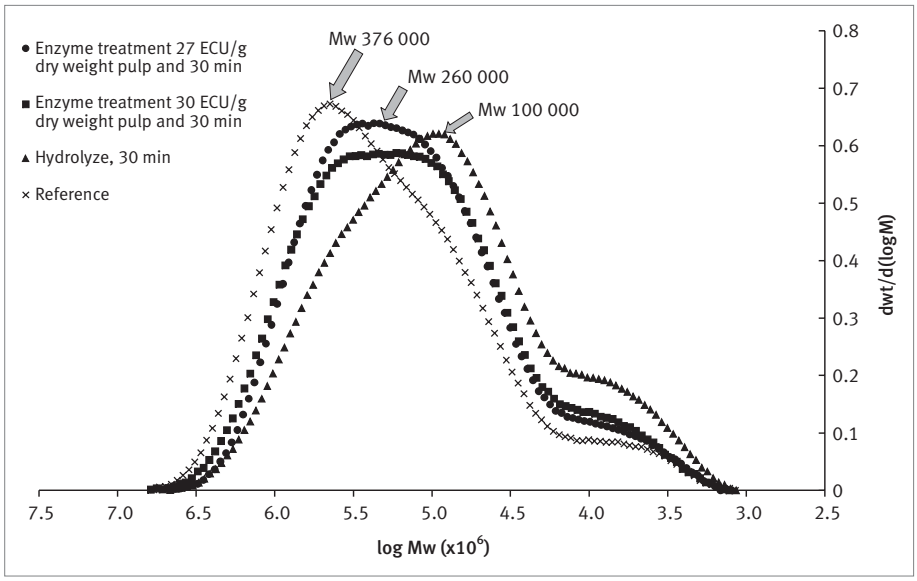

Figure 2. Molecular weight distribution for pretreated samples with 100% reactivity according to Fock.

and alkali solubility were obtained using endoglucanases, both purified and commercial products. The Swelling co-efficient was clearly correlated with the measured R10 values. The hydrolysis rate, estimated based on soluble products, was less than 1.2 % of pulp with purified enzymes and less than 2.5 % with commercial preparations. In terms of the accessibility, the drying history of pulp had a noticeable effect, and pre-treatments gave clear improvement in the accessibility (Figure 3).

The effects of enzyme treatments on birch kraft pulps were also analysed (Figure 4). On the basis of HPLC analysis of the reaction products it could be concluded that the hydrolysis was mainly targeted to surface xylans. The results indicate the major role of surface hemicellulose in the treatments and behaviour of the pulp.

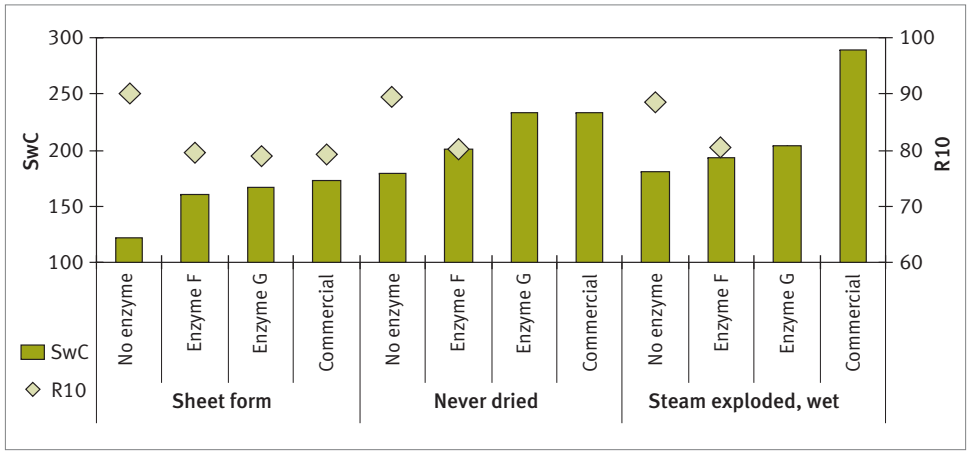

Figure 3. Measured swelling co-efficients (SwC) and R10 values of various dissolving grade pulps treated with purified or commercial endoglucanases (1 mg/g pulp, for 3 h).

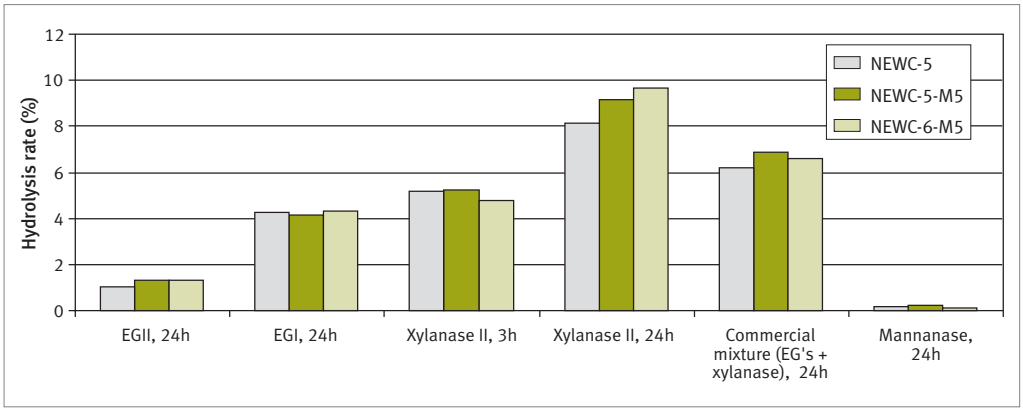

Figure 4. Hydrolysis of birch kraft pulps with purified cellulases and hemicellulases of Trichoderma reesei (1 mg/g pulp). Pulps (NEWC-5: never dried; NEWC-5-M5: mechanically pre-treated never dried; NEWC-6-M5: mechanically pre-treated sheet form pulp).

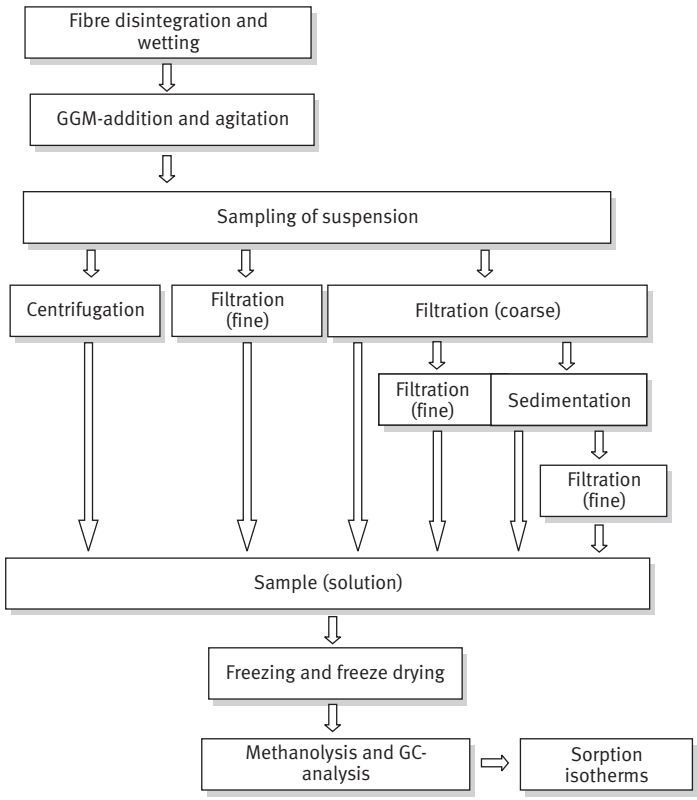

Figure 5. Overview of the experimental path way.

The impact of enzyme modification in which several different enzymatically treated pulps will be evaluated as raw materials for CMC manufacture.

The dissolution of differently treated pulp samples into 8 % LiCl/DMAc solvent was evaluated. It was demonstrated that the dissolution times of dif-

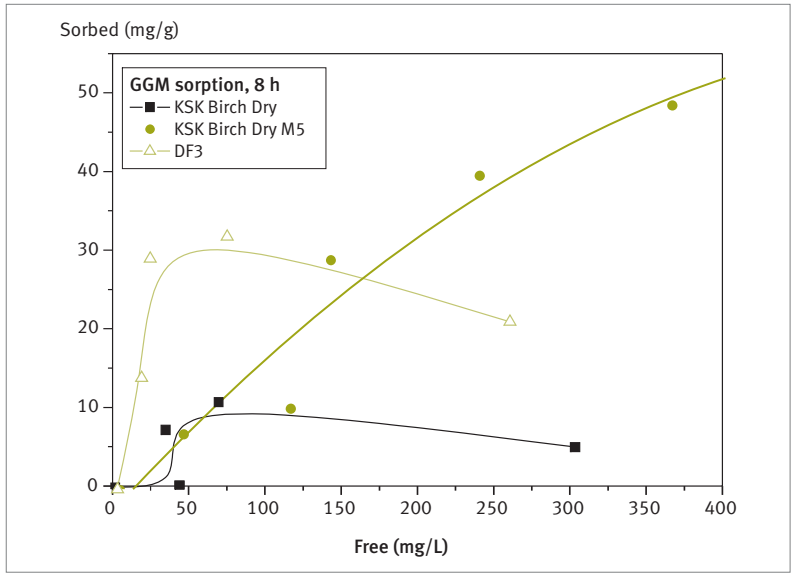

Figure 6. GGM (galactoglycomannan) sorption isotherms for different dissolving pulps.

ferently treated samples were in the same range, 1-2 days, and un-dissolved particles were not detected in large quantities in any sample. Mechanical shredding alone appeared not to affect the viscosity, instead the combined mechanical and chemical treatments had clear effect. The sample with lowest viscosity was selected for larger scale solution to be used for synthesis of RAFT-agents.

New method for determination of accessibility

Water-soluble, natural hemicelluloses such as galactoglucomannan (GGM) have a linear polymer back-bone. They have been found to adsorb onto polymers of analogous structure, *e.g.* cellulose. This attraction property may be utilised for determination of the free sites or the accessibility of cellulosic pulp fibres (Figure 5).The GGM method has been tested on different pulps and preliminary results indicates that it is a sensitive method which can be used for determination of accessibility (Figure 6).

10.2.2 Controlled synthesis

To obtain homogeneous cellulose derivatives it is important to find homogeneous modification conditions and to use controlled techniques to derivatise cellulose. Preparation of CMC was chosen as a model derivative since it is one of the most important and widely applied cellulose ethers. CMC, ethyl hydroxyethyl cellulose (EHEC) and under sub-project I pre-treated cellulose were used in graft-copolymerization.

During the work the focus of CMC synthesis was on a heterogeneous slurry process because it is the most relevant from an industrial point of view. Various pre-treatments and viscosities of the pulps were used in order to obtain controlled structures. CMCs were prepared from five different pulps, two dissolving pulps, microfibrillar cellulose (MFC) and two pre-treated pulps from sub-project I. The target was to obtain a degree of substitution (DS) of 1.24. MALDI-TOFMS and high-performance liquid chromatography (HPLC) were used to characterize the CMC products with regard to the *DS* and monomer composition. Table 1 shows a comparison between the DS values measured by HPLC and MALDI-TOF. It is noteworthy that both methods produce similar values. The product DS is higher for the low viscosity untreated and pre-treated DF pulp compared to the higher viscosity pulp. By HPLC significant differences in the substituent distribution on the monomer level were detected. The high viscosity pulp had a lower fraction of substituents at position 3 compared to the positions 2 and 6. The amount of reaction chemi-

cals and conditions during synthesis were identical, but the water content was higher in DF-Dissolving Pulp η=515 (never dry). Thus, both water content and viscosity diversity may contribute to the different substitution patterns.

Reversible addition fragmentation chain transfer polymerization (RAFT) makes possible the tailoring of macromolecules with sophisticated architectures including graft copolymers and was therefore used in this project to build up side chains on CMC, EHEC and cellulose backbones. The synthesis consists of a conventional free radical polymerization of a substituted monomer in the presence of a suitable chain-transfer agent. Various RAFT-agents with carboxylic acid function were prepared and used in esterification reactions with hydroxyl groups of CMC, EHEC and cellulose. Different reaction times and temperatures were used and polymer/RAFT-agent ratios were varied. The macroRAFT-agents were characterised with ATR-FTIR, ^{1}H, ^{13}C and/or ^{13}C CPMAS NMR (an example in Figure 7). The results were used to confirm that RAFT-agent was successfully bound to the backbone and the amount was analysed by elemental analyses.

Different monomers were polymerized in a controlled manner by grafting with the technique using the prepared macroRAFT-agents as listed in Table 2. Hydrophilic and hydrophobic grafts were polymerized successfully. The products, new cellulose based copolymers, were characterized using ATR-FTIR, ^{1}H, ^{13}C and/or ^{13}C CPMAS NMR.

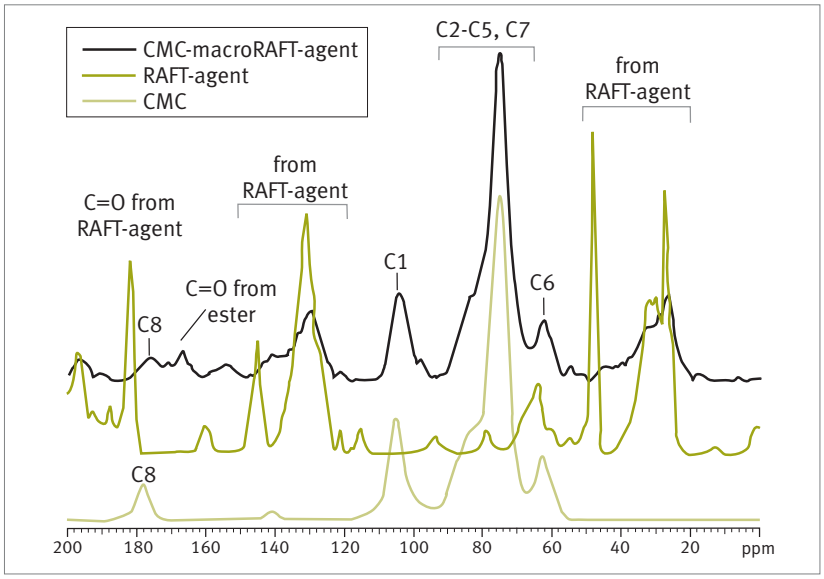

Figure 7. ^{13}C CPMAS NMR spectra of CMC, RAFT-agent and CMC-macroRAFT-agent (from bottom to top, respectively).

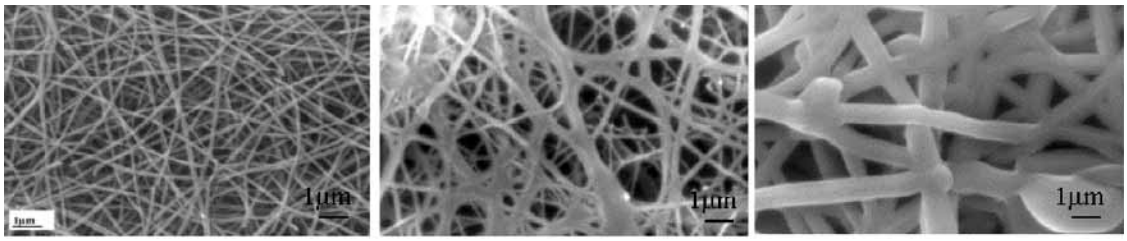

Figure 8. SEM images of a) electrospun 2,86% HPMC water/ethanol (1/1) solvent. b-c) electrospun 2,86% HPMC in mixture with 5% of a model drug, b) Dichloromethane/ethanol (1/1) solvent, c) water/ethanol (1/1) solvent.

10.2.3 Properties of new derivatives

MALDI-TOFMS of CMC was refined and can now be used for a rapid evaluation of the DS of multiple samples, e.g. in a screening process.[1]

The addition of ammonium sulphate to the sample-matrix mixture in the sample preparation procedure greatly facilitated the analysis. The spectrum acquisition procedure was more rapid and generated high-resolution spectra that could be processed using an automated peak identification and calculation procedure (MATLAB-script), which was developed in the project.

MALDI-TOFMS of enzymatically hydrolyzed CMCs was performed and valuable information on the chemical structure of the substrate, as well as the enzyme selectivity, could be obtained. The work involving enzymatic hydrolysis was performed in cooperation with VTT Technical Research Centre of Finland.

An approach for detecting variations in the substitution pattern along the cellulose backbone was developed.[2] In this approach, a purified endoglucanase with high selectivity, i.e. restricted to hydrolyzing low- or un-substituted segments along the cellulose backbone, was used. The molar mass distribution and the oligomer composition in the hydrolysates were analyzed by SEC/MALS/RI and MALDI-TOFMS, respectively.

Two CMCs, with similar chemical composition (e.g. similar DS and glucose content) and molar mass, displayed differences in the technical properties. These variations could be related to the variations in the substitution pattern as detected by the described method.

Furthermore, quantification of cellooligomers (e.g. cellotetraoses and cellopentaoses) from enzymatic hydrolysis could be performed with high reproducibility by standard addition and MALDI-TOFMS. The results were in good agreement with data from high-performance anion-exchange chromatography with pulsed amperometric detection (HPAEC-PAD) that were produced at VTT.

Besides MALDI-TOFMS, ^{13}C CPMAS NMR measurements were used at UH to evaluate the changes in fibre wall ultra-structure and to get information about the effects of mechanical and chemical treatments. The spectra were evaluated on the basis of cellulose fibril aggregation and the related schematic model.

10.2.4 Applications of new derivatives

Electrospun fibres

Electrospinning of nanofibers based on various cellulose derivatives, mainly commercially available, have been conducted. Different derivatives have been electrospun and the influence of the molecular weight (Mw), degree of substitution (DS) and distribution of the side-groups on the backbone (block or random) on the electrospinning process as well as on the obtained fibres have been investigated. Parts of the work have been published.[3] Electrospinning of fibres of new cellulose derivatives, developed within the frame of the NEWCELL project, has been conducted. Unfortunately, beads rather than fibres were produced.

During the first year, the work focused on the dissolution and electrospinning of enzymatically treated cellulose and CMC. During the second year, the process parameters for electrospinning of CMC with a carrier, HPMC, HPC (hydroxypropyl cellulose), MC, EC (ethyl cellulose), EHEC and MEHEC (methylethylhydroxyethyl cellulose) were investigated (parts published [3]).

During the third year, the work has continued with focus on the influence of the ratio between CMC and the carrier. The influence of the ratio was, however, proved to be low and similar fibres were produced from a broad range of mixtures. A part that has been initiated during the third year involves encapsulation of a model drug in the nanofibers during the electrospinning. In the SEM images below, pure HPMC-nanofibers are shown as well as fibres of HPMC in mixture with a model drug substance. A comparison shows that the fibre morphology is highly influenced. For instance, thicker fibres are formed when incorporating the model drug. Furthermore, the different solvents used for the mixed system, i.e. water/ethanol and dichloromethane/ethanol also influences the morphology of the final fibres. Thicker and more even fibres are formed from solutions based on water/ethanol as solvent.

Cellulosic casings

The main aim was to test the new derivatives developed in the project in order to substitute some or all of the cellulose xanthate that is currently used for production of fibrous casings. As the casings are in contact with food they cannot contain any harmful

chemicals or substances, which must be taken into consideration in developing work.

The primary test to find out proper coagulant agents for cellulose derivatives have been carried out in a limited range. Suitable solvents and coagulants are found for some derivatives, and more extensive laboratory trials will be carried out with them. The derivatives could maybe also be used as an additive to viscose prior to coagulation to form a casing.

The studies are continuing by literature search, and laboratory trials to form sheets with and without viscose solution onto base paper, and also by measuring the properties of elaborated films.

Cellulosic sponges, membranes and beads

A technology for manufacturing of cellulosic beads with tailored properties was developed using viscose and was further evaluated for different derivatives. Size, shape, surface morphology, porosity and degree of crystallization can be tuned to design innovative functionalities for bead applications in drug delivery and encapsulation. A modification in the bead-making process was developed by making beads by using different blends of pure viscose and CMC solutions in different proportions. Tailoring of the physicochemical properties and surface chemistry of the beads have been performed and it was found out that the properties like swellability, porosity, shape, morphology, surface charge and weight loss of the beads can be altered by blending CMC and viscose solutions. These innovative functionalities for beads can open new doors for further utilization of beads in drug delivery and encapsulation. EHEC solution was successfully used for producing membranes on a laboratory scale. In the area of sponge making, progress was made by producing sponges with tailored pore sizes. A thorough and deep understanding of the effects of addition of other derivatives to viscose is still required in order to achieve controlled functionality for the beads, membranes and sponges.

10.3 Conclusions

Various methods to increase the accessibility of cellulose materials to swelling and reactive agents at different hierarchical levels have been studied. Among mechanical, chemical, and enzymatic pre-treatments it was found that monocomponent cellulases, cellulase mixtures and other cellulose acting proteins were very effective pre-treatment methods. The best results were obtained using endoglucanases, both purified and commercial products. A new method to measure the reactivity or accessibility of the hydroxyl groups of the cellulose has been developed. A heterogeneous slurry process was chosen to prepare CMC with a target *DS*. This synthesis was used as a model method to develop knowledge about the pre-treatment effect on the derivatisation. The viscosity of the starting pulp and drying of the pulp before synthesis seem to have a large impact of the CMC product quality.

Various macro-RAFT agents were synthesized from CMC, EHEC and pre-treated cellulose and were further used as starting materials for graft polymerization. Various monomers were polymerized in a controlled manner to prepare side chains with a hydrophilic or hydrophobic character. Different starting materials required their own modification conditions as well as synthetic path ways, however new cellulose based copolymers were prepared successfully.

Valuable information regarding block-wise or random substitution pattern along the polymer backbone can be obtained by combining enzymatic hydrolysis by purified endoglucanases with MALDI-TOFMS and SEC/MALS/RI. The detected differences in substitution pattern present a plausible explanation to the measured variations in the technical properties. The method can also provide information regarding the selectivity of the endoglucanase used in the hydrolysis.

MALDI-TOFMS is a valuable tool for chemical structure characterization of cellulose derivatives. Improvements in the sample preparation procedure have made it possible to evaluate the DS in CMC as a compliment to more expensive and laborious methods.

Nanofibres of several commercial cellulose derivatives have been successfully produced by electrospinning. The spinnability of the derivatives was shown to be influenced by the material parameters (e.g. molar mass and substitution pattern) but also by other compounds, as seen when a model drug was incorporated into the fibre. Methods for developing beads from mixtures of viscose and CMC have been developed. The physicochemical properties and surface chemistry of the beads can

be tailored, changing properties like swellability, porosity, size, shape, morphology and surface charge.

References

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2. Enebro, J.; Momcilovic, D; Siika-Aho, M; Karlsson, S. *MANUSCRIPT*
3. Frenot, A.; Henriksson, M. W.; Walkenström, P. *Journal of Applied Polymer Science* 2007, *103*, 1473-1482.

10.4a Capabilities generated by the project

Two of the most important quality parameters for dissolving cellulose pulp are pulp reactivity and accessibility. These parameters have been studied in the project and suitable analytical tools have been used to characterize them. Several different methods have been elucidated in order to improve them, but the only one that gave a significant positive response was treatment with cellulose enzymes.

The project has developed:

- A new method for cellulose accessibility determination
- Kinetics of enzymatic pretreatments of pulps
- Synthesis of RAFT-reagents
- Synthesis of macroRAFT-agents from CMC, EHEC and cellulose
- Synthesis of graft-copolymers from CMC, EHEC and cellulose
- Synthesis of CMC with target DS
- MALDI-TOFMS protocol for analysis of DS in CMC.
- Automated spectrum processing and calculation program for rapid evaluation of results (MATLAB-script).
- Method for detecting differences in substitution pattern in CMCs.
- Evaluation of controlled synthesis reactions by MALDI-TOFMS.

10.4b Utilisation of results

Today it is difficult to use enzymes direct in process production, but if the enzyme technique can be used in a full mill operation, the pulp producers will be interested in it. The results from the project how to best characterise the dissolving pulp for different cellulose derivative applications have been so far the most important results for the pulp producers. The new developments within analytical methodology have provided manufacturers and users with new tools for chemicals structure analysis of cellulose derivatives. Nanofibre materials have opened up for new possibilities within in new market areas.

10.5 Publications and communication

Agnemo, Roland, "The NewCell project", presentation for board of Domsjö and 50 participants from Ekmans handelshus, Gothenburg, 2006.

Ek, Monica, "Cellulose accessibility", Oral presentation at NanoCell and NewCell Seminar (Stockholm, Nov-2006).

Ek, Monica, "The New Cell project", Japanese-European Workshop on "Cellulose and Functional Polysaccharides" Sep 1-14, 2006, Boku University, Vienna, Austria.

Karlsson, *Sigbritt, "New Cellulose Derivatives for High Value Products"*, Oral presentation at the Wood Material Science and Engineering Research Program, Annual Seminar (Helsinki, Apr-2006).

a) Scientific publications

Axelsson, Sara, Karlsson, Sigbritt, Controlled synthesis of carboxymethyl cellulose, ORAL PRESENTATION, 2nd Workshop on Cellulose and Cellulose Derivatives, Karlstad, Nov. 22-23rd 2005.

Ek, Monica, Engström, Ann-Charlotte, and Henriksson, Gunnar, "*Increase Reactivity of Dissolving Pulps by different pretreatments",* ORAL PRESENTATION at 9th European Workshop on Lignocellulosics and Pulp, Vienna, Austria, Aug. 28-30, 2006

Ek, Monica, "The New Cell project", Innovative Biopolymers Polysaccharides – from Discovery to Commercialization, August 20-21, 2006, Gullmarstrand, Fiskebäckskil, Sweden.

Enebro, Jonas, Karlsson, Sigbritt, "*Characterization of Homogeneous Cellulose Derivatives",* POSTER PRESENTATION at the Second International Symposium on the Separation and Characterization of Natural and Synthetic Macromolecules (SCM2, Amsterdam Feb-2005).

Enebro, Jonas, Karlsson, Sigbritt, *"MALDI-TOF MS Analysis of Cellulose Derivatives",* ORAL PRESENTATION at the Nordic Polymer Days (Gothenburg, Aug-2005).

Enebro, Jonas, Karlsson, Sigbritt,*"MALDI-TOF MS Analysis of Carboxymethyl Cellulose",* ORAL PRESENTATION at the Second Workshop Cellulose and Cellulose Derivatives (Karlstad Nov-2005).

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Hiltunen, Miia, "*New Cellulose Derivatives with Controlled Chemical Structure of Grafts via RAFT Polymerisation*", ORAL PRESENTATION at the Second Workshop on Cellulose and Cellulose Derivatives (Karlstad, Nov-2005).

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a) Scientific publications

1. Articles in international scientific journals with referee practice

Engström, A-C., Ek, M. and Henriksson, G., "*Improved Accessibility and Reactivity of Dissolving Pulp for the Viscose Process: Pretreatment with Monocomponent Endoglucanase"*, Biomacromolecules, 2006; 7(6); 2027-2031.

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Frenot, A.; Henriksson, M. W.; Walkenström, P. *Journal of Applied Polymer Science* 2007, *103*, 1473-1482.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice,

Ek, M., Engström, A-C., Henriksson, G., Vehviläinen, M., Heikkilä, E. and Agnemo, R., *Cellulose derivatives-improved accessibility and reactivity*, 13th International Symposium on Wood, Fibre and Pulping Chemistry (ISWFPC). Auckland, New Zealand, 2005, Proceedings, Vol. 2, pages 121-126.

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Maunu SL, Wikberg H and Hiltunen M : *Solid State NMR Possibilities in the Characterisation of Cellulose and Its Derivatives.* 13th International Symposium on Wood, Fibre and Pulping Chemistry (ISWFPC). Auckland, New Zealand 2005, Proceedings, Vol. 2., pages 349-352.

Hiltunen M, Maunu SL and Tenhu H, "*New Cellulose Copolymers with Controlled Chemical Structure of Grafts via RAFT Polymerization"*, 14th International Symposium on Wood, Fibre and Pulping Chemistry (ISWFPC). Durban, South Africa 2007, Proceedings-manuscript.

b) Other dissemination

Roland Agnemo, Sigbritt Karlsson, NewCell, interview in Domsjö journal *Insikt*, 2005

c) Under preparation

1. Articles in international scientific journals with referee practice

Siika-aho, M., Grönqcvist, S., et al. *"Enzymatic modification of dissolving grade softwood pulp: impact on pulp accessibility"* MANUSCRIPT in preparation.

Hiltunen M, Maunu SL and Tenhu H, "The Synthesis of EHEC-*g*-PAam Copolymer via RAFT-polymerisation", MANUSCRIPT

Enebro, J.; Momcilovic, D; Siika-Aho, M; Karlsson, S. *"Estimation of blockiness in Carboxymethyl Cellulosby Enzymatic hydrolysis, SEC/MALS/RI and MALDI-TOF MS",* MANUSCRIPT

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Hiltunen Miia: Oral presentation at Nordic Polymer Days 2007 with the title "*The synthesis and characterization of new cellulose derivatives via RAFT polymerization*" (Helsinki, May 2007)

Hiltunen Miia: Poster at European Polymer Congress 2007 with the title "*New Cellulose Copolymers with Controlled Chemical Structure of Grafts via RAFT Polymerization"* (Portoroz, July 2007)

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Riihelä Simon, M.Sc. thesis, Laboratory of Polymer Chemistry, University of Helsinki. (2007, in Finnish).

10.6 National and international cooperation

Steering committee

Bengt Wittgren, Chairman,
AstraZeneca R&D, Sweden
Sigbritt Karlsson, Coordinator,
Sub-project leader (SP3), KTH, Sweden
Pertti Nousiainen, TUT, Finland
Pedro Fardim, ÅA, Finland
Monica Ek, Sub-project leader (SP1),
KTH, Sweden
Sirkka Liisa Maunu, Sub-project leader (SP2),
UH, Finland
Matti Siika-Aho, VTT Technical Research
Centre of Finland
Göran Kloow, CP Kelco Sweden
Roland Agnemo, Domsjö Fabriker AB, Sweden
Pernilla Walkenström, IFP Research AB, Sweden
Leif Karlson, AkzoNobel Functional Chemicals,
Sweden
Ismo Lokinoja, CP Kelco Finland
Hans Henrik Øvrebø, Borregaard, Norway
Päivi Talvenmaa, Sub-project leader (SP4),
TUT, Finland
Camilla Wikström, Oy Metsä-Botnia AB,
Finland
Kurt Lönnqvist, Vivoxid Ltd, Finland
Jonna Tuominen, Oy Visko AB, Finland

KTH has initiated cooperations with the group of Prof. Thomas Heinze (Friedrich Schiller University of Jena) in areas related to SP2-3.

KTH have corporations with the research group of Prof. Ulf Germgård (University of Karlstad) related to SP1-3.

KTH have cooperations with professor Tommy Iversen, STFI-Packforsk SP 1

UH is in on the COST E41 action.

11 Nanostructured Cellulose Products (NanoCell)

FINAL REPORT

Name of the research project	**Nanostructured Cellulose Products**
Coordinator of the project	Prof. Tom Lindström

BASIC SUB-PROJECT DATA

Name and leader of the sub-project 1	**Nanofacility**
Project period	1.1.2004–31.12.2006
Organization in charge of research	STFI-Packforsk AB
Sub-project leader	Prof. Tom Lindström
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	STFI-Packforsk AB P.O Box 5604 SE-114 86 Stockholm, Sweden Phone: +46 8 67 67 000 Fax: +46 8 21 42 35 tom.lindstrom@stfi.se
URL of the sub-project	http://www.stfi-packforsk.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	328 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	164 400
Other funding	
Industrial funding in kind	164 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Tom Lindström, Prof., Sub-project leader	M	STFI-Packforsk AB		
Mikael Ankerfors, M.Sc., Scientist	M	STFI-Packforsk AB		
Total person-months of work conducted by the research team person-month = full-time work for at least 36 h/week, paid holidays included	18.4			

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name and leader of the sub-project 2	Modified Surfaces and their Characterisation
Project period	1.1.2004–31.12.2006
Organization in charge of research	Helsinki University of Technology (TKK)
Sub-project leader	Prof. Janne Laine
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Helsinki University of Technology (TKK) Laboratory of Forest products Chemistry P.O. Box 6300 FI-02015 TKK, Finland Tel. +358 9 451 4233 Fax +358 9 451 4259 janne.laine@tkk.fi
URL of the sub-project	http://www.hut.fi/Yksikot/Puukemia/index.html http://www.woodwisdom.fi/en/

FUNDING

Total subproject budget in EUR	246 200
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	216 902
Other funding	
Industrial funding	29 298

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Janne Laine, Prof. Sub-project leader	M	Helsinki University of Technology		
Monika Österberg, Ph.D., Senior scientist	F	Helsinki University of Technology		
Susanna Ahola, M.Sc. (Tech.), Research scientist	F	Helsinki University of Technology		
Marja Kärkkäinen, Lab. technician	F	Helsinki University of Technology		

Total person-months of work conducted by the research team 47.36
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
–	–	–	–	–	–

Name and leader of the sub-project 3	Functional Fibre Coatings
Project period	1.1.2004–31.12.2006
Organization in charge of research	STFI-Packforsk AB
Sub-project leader	Prof. Tom Lindström
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	STFI-Packforsk AB P.O. Box 5604, SE-114 86 Stockholm, Sweden Tel. +46 8 67 67 000 Fax +46 8 21 42 35 tom.lindstrom@stfi.se
URL of the sub-project	http://www.stfi-packforsk.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	202 400
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	101 200
Other funding	
Industrial funding	101 200

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Tom Lindström, Prof., Sub-project leader	M	STFI-Packforsk AB		
Mikael Ankerfors, M.Sc., Scientist	M	STFI-Packforsk AB		
Total person-months of work conducted by the research team person-month = full-time work for at least 36 h/week, paid holidays included			11.3	

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	M.Sc.	M	Karl Axnäs, 1979, M.Sc. 2006	KTH	Lars Wågberg, KTH, Gero Decher, ICS/ULP, France

Name and leader of the sub-project 4	High Performance Nanocomposites
Project period	1.1.2004–31.12.2006
Organisation in charge of research	Royal Institute of Technology KTH
Sub-project leader	Prof. Lars Berglund
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Royal Institute of Technology (KTH) Dept. Fibre and Polymer Technology Teknikringen 56 SE-100 44 Stockholm, Sweden Tel. +46 8 790 60 00 blund@kth.se
URL of the sub-project	http://www.kth.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	404 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	202 400
Other funding	
Industrial funding in kind	202 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Lars Berglund, Prof. Sub-project leader	M	Royal Institute of Technology (KTH)		
Said Azizi Samir, Ph.D., senior scientist	M	Royal Institute of Technology (KTH)		
Anna Svagan, M.Sc., Research scientist	F	Royal Institute of Technology (KTH)		
Total person-months of work conducted by the research team 36 person-month = full-time work for at least 36 h/week, paid holidays included				

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	Tech. Lic.	F	Anna Svagan, 1979	KTH	Lars Berglund
2004	Tech. Lic.	F	Marielle Henriksson, 1976	KTH	Lars Berglund

Name and leader of the sub-project 5	Functional Materials
Project period	1.1.2004–31.12.2006
Organisation in charge of research	Helsinki University of Technology (TKK)
Sub-project leader	Prof. Olli Ikkala
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Helsinki University of Technology (TKK) Molecular Materials Group P.O. Box 2200, FI-02015 Espoo, Finland Tel. +358 9 451 3154 Fax +358 9 451 3155 olli@focus.hut.fi
URL of the sub-project	http://www.stfi-packforsk.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	258 200
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	227 498
Other funding	
Industrial funding	30 702

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Oli Ikkala, Prof., sub-project leader	M	Helsinki University of Technology		
Robin Ras, Ph.D., Senior scientist	M	Helsinki University of Technology		
Marjo Pääkkö, M.Sc., research Scientist	F	Helsinki University of Technology		
Riita Silvennoinen, Lic.Sc. (Tech.), Research scientist	F	Helsinki University of Technology		
Jaana Vapaavuori, student	F	Helsinki University of Technology		
Harri Kosonen, Ph.D., senior scientist	M	Helsinki University of Technology		

Total person-months of work conducted by the research team 72
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2004	Ph.D.	M	Harri Kosonen, 1975	TKK	Olli Ikkala, TKK
2006	Lic. Tech.	F	Riitta Silvennoinen, 1978, M.Sc. 2003	TKK	Outi Krause, Prof. TKK Olli Ikkala, Prof. TKK

Name and leader of the sub-project 6	**Cellulose based superhydrophobic materials**
Project period	1.1.2004–31.12.2006
Organisation in charge of research	Royal Institute of Technology (KTH)
Sub-project leader	Prof. Lars Wågberg
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Royal Institute of Technology (KTH) Dept. Fibre and Polymer Technology Teknikringen 56, SE-100 44 Stockholm, Sweden Tel. +46 8 790 82 94 Fax +46 8 790 81 01 wagberg@polymer.kth.se
URL of the sub-project	http://www.kth.se http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	404 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	202 400
Other funding	
Industrial funding in kind	202 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Lars Wågberg, Prof., Sub-project leader	M	Royal Institute of Technology (KTH)		
Oskar Werner, Ph.D. student	M	Royal Institute of Technology (KTH)		
Lisa Persson, M.Sc. student	F	Linköping University (LiU)		
Karl Axnäs, M.Sc. student	M	Royal Institute of Technology (KTH)		

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	M.Sc.	F	Lisa Persson, 1980	LiU	Mikael Ankerfors and Tom Lindström, STFI-Packforsk, Lars Wågberg and Oskar Werner, KTH, Pentti Tengvall, LiU
2006	M.Sc.	M	Karl Axnäs, 1979	KTH	Lars Wågberg, KTH, Gero Decher, ICS/ULP, France

Abstract

Within the Nanostructured Cellulose Products project new methods for production of microfibrillated cellulose (MFC) have been developed and utilised. These methods involve treatments of pulp fibres using techniques such as mechanical disintegration, enzymatic hydrolysis and chemical modifications followed by high-pressure homogenisation. The production of MFC in kilogram quantities has been carried out and MFC has been characterised using techniques such as AFM, TEM, SEM, NMR and with respect to their rheology. The MFC has also been subjected to surface modifications, e.g. surface carboxymethylation. During the project, different applications for MFC have been studied. For example, bio/nanocomposites of MFC, starch and glycerol as well as pure MFC-films have been manufactured. Finally, the MFC has been used to develop advanced nanostructured materials (foams and aerogels) and nanostructured surfaces.

Tiivistelmä

Nanostructured Cellulose Products -projektissa on kehitetty ja hyödynnetty uusia menetelmiä mikrofibrillaarisen selluloosan (MFC) valmistamiseen. Näihin valmistusmenetelmiin kuuluvat sellukuitujen käsittelyt, kuten mekaaninen hajotus, entsymaattinen hydrolyysi ja kemialliset modifioinnit sekä lopullinen homogenisointi korkeassa paineessa. MFC:tä on valmistettu kilogramma-mittakaavassa ja materiaalia on karakterisoitu käyttämällä eri tekniikoita, kuten AFM, TEM, SEM ja NMR. Lisäksi on tutkittu MFC-geelin reologiaa. MFC:n pintaa on myös modifioitu esim. pinnan karboksymetyloinnilla. Projektin aikana on tutkittu erilaisia MFC:n sovelluskohteita. Esimerkiksi on valmistettu bio/nanokomposiitteja MFC:stä, tärkkelyksestä ja glyserolista sekä valmistettu filmejä pelkkää MFC:tä käyttäen. MFC:tä on myös käytetty uudenaikaisten nanomateriaalien (vaahdot ja aerogeelit) ja nanorakenteisten pintojen kehittämiseen.

Sammanfattning

Inom ramen för projektet "Nanostructured Cellulose Products" har nya metoder för att tillverka mikrofibrillär cellulosa (MFC) utvecklats och använts. Dessa metoder innefattar behandlingar av pappersmassafibrer med tekniker såsom mekanisk bearbetning, enzymatisk hydrolys och olika kemiska modifieringar följt av högtryckshomogenisering. Under projektet har flera kilogram av MFC tillverkats och MFCn har karaktäriserats med tekniker såsom AFM, SEM, TEM och NMR. Dessutom har de reologiska egenskaperna hos MFC-gelen studerats. MFCn har även ytmodiferats med kemiska tekniker, t.ex. ytkarboxymetylering. Under projektet har dessutom olika användningsområden för MFC studerats. Till exempel har bio-nanokompositer av MFC, stärkelse och glycerol samt rena MFC-filmer tillverkats. Slutligen har MFCn använts för att skapa avancerade material (skum och aerogeler) och strukturerade ytor.

11.1 Introduction

11.1.1 Background

Nanotechnology is currently one of the most promising areas of scientific and technological development. Recent developments in measurement techniques (for instance atomic force microscopy (AFM)) have made it possible to understand, control and manipulate materials and structures at the nanoscale. This allows new possibilities to introduce functionality into materials and products, such as high mechanical performance, control of hydrophobicity, unique optical, magnetic and electric properties etc.

The stiff constituent in a plant cell wall is the cellulose microfibril. Microfibrillated cellulose (MFC), see Figure 1, offers unique possibilities due to its fine scale, high stiffness and strength as well as its combination of high crystalline perfection and hydroxyl groups at the surface. Recently, we have developed a procedure where MFC can be obtained from pulp fibres. The processing is carried out by combination of enzymatic hydrolysis and mechanical disintegration followed by a homogenisation. The advantage with the combined enzymatic and mechanical pre-treatment is that a lower number of passes through the homogeniser is needed so that the required energy is reduced. The presence of hydroxyl groups at the surface of fibrils with a diameter in the nanoscopic range is of considerable interest since they provide reaction sites for further chemical modification. The hydroxyl groups may for instance be used as "handles" to tailor attraction and repulsion forces between nanoscale MFC fibrils. MFC is well suited as a template for surface modification, although it has never been used in this way. Modification effects can be both chemical and physical, where the physical mechanisms are based on surface texture changes.

MFC has not previously been used in material products. Although attempts have been published where MFC was intended as a food and rheology additive, this has not been a commercial success. In the present case, the process to obtain MFC is less energy consuming and the intended applications are in high performance and unique functionality applications. MFC fibres are environmentally friendly, renewable and well suited for low temperature processing of materials in water suspension rather than in organic solvents.

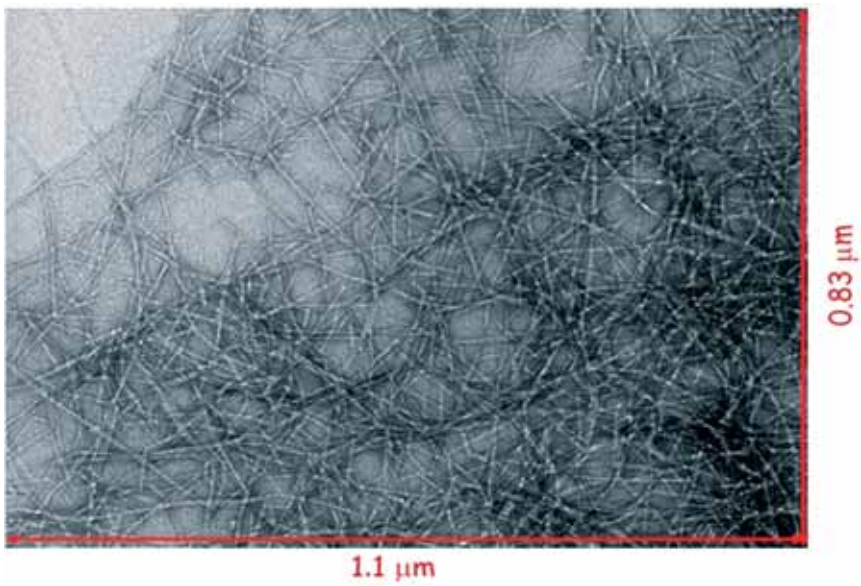

Figure 1. Transmission Electron Micrograph of microfibrillated cellulose.

11.1.2 Objectives

The program has the following three major objectives:

1. Well-characterized microfibrillated cellulose (MFC) from wood.

MFC is currently not available commercially. Turbak's procedure requires many passes through a mechanical homogenizer. It is expected that the present approach, will reduce the required number of passes, as indicated by preliminary results (Sub-project 1, "Nanofacility"). In addition, the MFC needs to be characterized in terms of molar mass, aspect ratio, fibril geometry and surface chemistry (Sub-project 2 "Modified Surfaces and their Characterisation")

2. MFC with tailored surfaces

The hydroxyl groups at the MFC surface will be used to tailor the surface chemistry of MFC. This will change the interaction of MFC with its environment, for instance in water suspension, during drying and in composite materials (Sub-project 1, "Nanofacility"). Such technologies have not previously been applied and can be used to control self-organization (Sub-project 5, "Functional Materials") of individual fibrils as well as polymers interacting with MFC (see also Sub-project 6, "Superhydrophobic surfaces").

3. New materials based on MFC

MFC with tailored surface chemistry is a very exciting constituent for new materials. For instance, one primary goal is to modify the surface so that MFC can be dried without problems of agglomeration. Today, related materials, such as hydrolysed microcrystalline cellulose, immediately agglomerate during drying. This makes further processing very difficult. Examples of applications of surface-modified MFC include high performance nanocomposites (Sub-project 4, "High Performance Nanocomposites") materials and films, materials with superhydrophobic surfaces (Sub-project 6, "Superhydrophobic Materials)" with unique optical, electrical, magnetic or adsorption properties, new wood-based fibres with nanocoatings or MFC-modified surface texture (Sub-project 3 "Functional Fibre Coatings"). Products include filters, textiles, films, packaging materials, coatings and moulded components.

11.2 Results and discussions

The major focus for Sub-project 1 has been to develop a production method for MFC and to manufacture MFC in kilogram quantities in order to supply the other sub-projects with the MFC. The manufacture of MFC has been done using a high pressure homogeniser (Microfluidizer, see figure 2) together with a pre-treatment method. Several pre-treatments methods have been developed during the project, which have lowered the energy consumption from around 30 000 kWh/tonne to with less than 1500 kWh/tonne.

Figure 2. The high-pressure homogeniser (Microfluidizer) used for the production of MFC.

The objective in Sub-project 2 was to characterize MFC and also to modify the fibril surfaces for different nanotechnology applications.

The dimensions of the microfibrils were determined using Atomic Force Microscopy (AFM). Two different size fractions of microfibrils were observed - small individual fibrils and larger fibril bundles. The average height of the individual fibrils was less than 10 nm and the length varied from 70 nm to several micrometers. The fibril bun-

dles were thicker than individual fibrils – their height was in the range of tens of nanometers. Dimensions and homogeneity of the fibrils in aqueous environment was characterized by measuring the particle size of the fibrils. The results showed that MFC forms aggregates in water but these aggregates can be eliminated by efficient stirring and/or centrifugation.

Model surfaces were needed for adsorption studies of MFC. Smooth and uniform model surfaces were prepared by spin-coating of disintegrated and centrifuged MFC dispersions (Figure 3). The model surfaces were used to study the swelling of the fibrils and adsorption of polymers on the fibrils. Modification of MFC was done by adsorbing both cationic and anionic polyelectrolytes (CMC) on the fibril surfaces.

MFC was also used to modify cellulose fibres in paper making. MFC was used together with a cationic polyelectrolyte, poly(amideamine) epihlorohydrin (PAE), to improve strength properties of paper. The adsorption of PAE and MFC on fibre surfaces was studied using QCM-D and AFM. By pre-adsorbing PAE on the cellulose surface a thick and viscous layer of the nanofibrils could be adsorbed. Paper hand sheets were prepared to study the effect of MFC and PAE on paper strength properties. The addition of MFC significantly improved both the wet and dry strength of the sheets compared to using PAE alone.

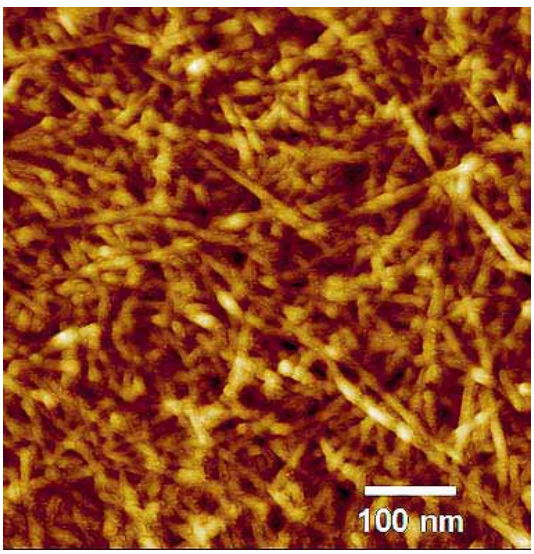

Figure 3. Topographical AFM image of the spin-coated MFC model surface on a silica substrate. The scan size is 600 × 600 nm. The RMS roughness of the MFC film is ~1 nm.

The objective for Sub-project 3 is to prepare multilayered fibre coating films containing instance MFC.

Multilayered thin films of MFC and cationic polyelectrolytes have been used on oxidised silicon wafers in order to prepare nanostructured fibrillar surfaces. The MFC selected was of generation 2 and three different cationic polyelectrolytes were investigated; a polyetyleneimine (PEI), a polyallylamine (PAH) and polyDiAllyl-DiMethylAmmonium-Chloride (p-DADMAC). The results show that multilayer thin films are formed with all the different polyelectrolytes but the 3D PEI show the most rapid increase in thickness of the film with the number of consecutive treatments. It has also been found that the films are so even that they achieve different colours depending on the thickness of the films (see figure 4). An attempt to form structured films by spraying the different components was also successful even though the films were not as even as for the dip-coating technique.

The objective for Sub-project 4 was to prepare MFC films of high mechanical performance as well as prepare novel biodegradable composites of high performance.

For MFC-films it is critical to have MFC which is well disintegrated, to develop the preparation procedure and to learn about the deformation mechanisms in the material. The quality of the MFC has bee improved during the year. The film preparation procedure is also improved so that the organization of the microfibrils most likely is better confined to true two-dimensional, rather than a considerable amount of out-of-plane MFC. The importance of high molar mass MFC has also been recognized. The films now show 16 GPa modulus, 220 MPa strength and up to 12 % strain to failure (see *figure 5*). This is an unusual combination of strength and ductility. For comparison, a strong paper sheet made from softwood kraft pulp typically has a modulus of 8 GPa, a strength of 80 MPa and 3 % strain to failure (this will of course depend on sheet manufacturing procedure etc.). Hence, the MFC-films are the strongest (nano)paper ever made. During deformation of the MFC-films, yielding seems associated with MFC debonding. Then follows a region where pull-out

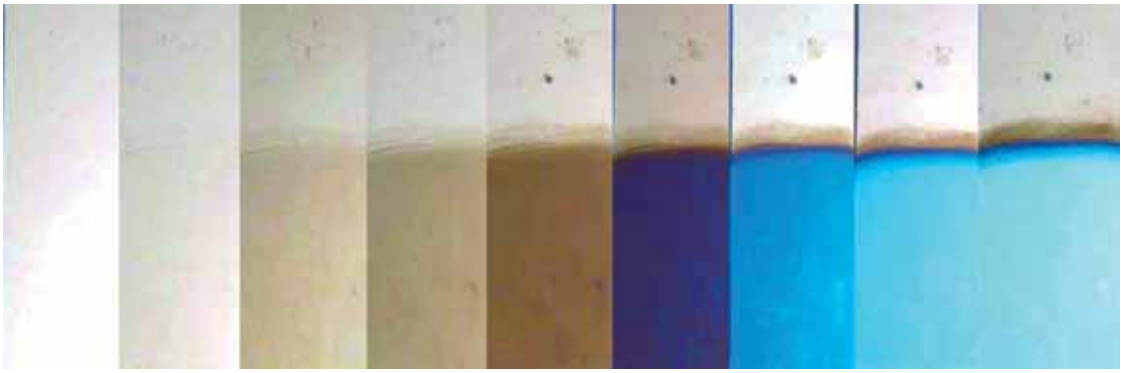

Figure 4. Multilayered films of MFC and polyelectrolytes are so even that they achieve different colours depending on the thickness of the films.

and friction appears important, and ultimate strength is controlled by molar mass.

For the biodegradable composites a procedure for introducing up to 70% of MFC in starch/glycerol was successfully developed. The resulting materials also showed a surprising combination of ductility, modulus and strength (see figure 6). The reason is that MFC acts as a network, so that the matrix can be very soft without compromising nanocomposite properties.

A novel nanocomposite foam based on MFC nanofibres and starch has been successfully prepared. The uniqueness is that for the first time has a foam been prepared where the cellulose fibre reinforcement is present in the cell wall due to its nanoscale diameter. Significant improvements in energy absorption performance were observed with cellulose nanofibres addition to starch foams. The cellulose nanofibres content in the cell wall was as high as 40 percent.

The goal of Sub-project 5 includes three steps: 1) characterization and 2) modification of the properties of MFC, and 3) identification of possible new applications and products (see Figure 7).

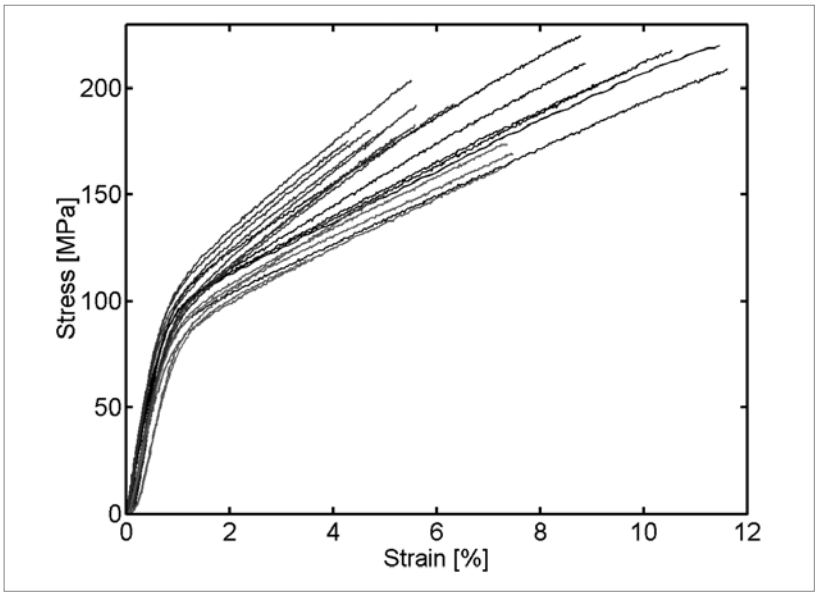

Figure 5. Stress-strain curves for different MFC films.

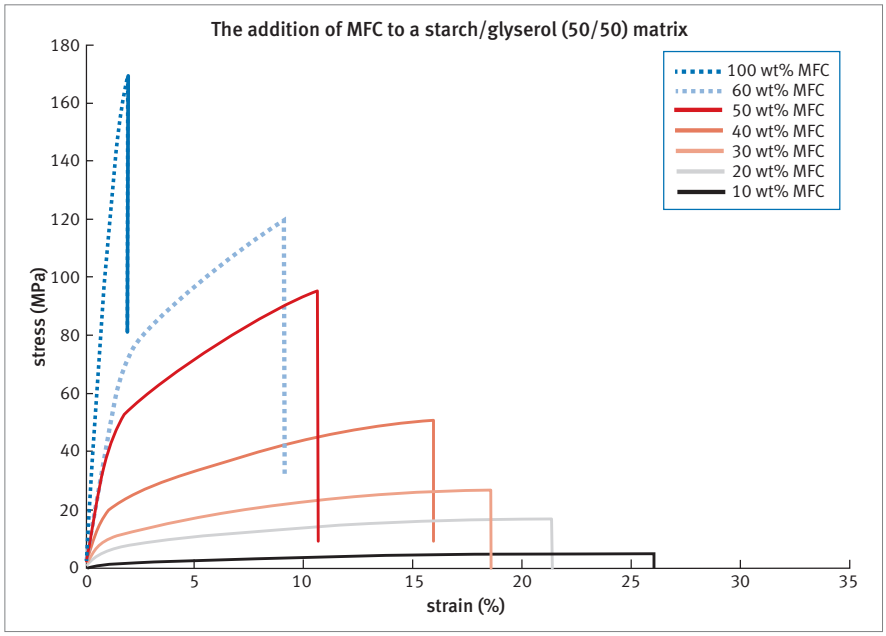

Figure 6. Stress-strain curves for nanocomposites based on MFC and starch-glycerol blends.

First, aqueous gels based on microfibrillated fibres were observed and their structural and viscoelastic properties were analysed using e.g. electron microscopy (TEM, SEM), atomic force microscopy, dynamic rheometer, and dynamic mechanical analysis. The gels were particularly strong even at small weight fractions of cellulose and strong pseudoplastic behaviour was observed.

Methods were developed to dry the network structures of the gels still preserving the highly porous structure which lead to highly porous aerogels. Nitrogen adsorption was used to determine the BET surface area. The dried MFC nanometre porous fibrils were used as a substrate for atomic layer deposition (ALD). The porous sponge-like network acted as a template for chemical vapour deposition techniques, mostly familiar from electronics processing to deposit in a controllable manner thin films of inorganic materials, in aiming towards functional organic/inorganic hybrid nanomaterials. The cellulose template and the inorganic coating are both confined to nanometre dimensions and they are expected to result in various attractive properties upon selecting different inorganic materials. As the first demonstration we prepared TiO2 -coated MFC with photo-catalytic activity (collaboration with Helsinki University). In addition, the dried MFC was used for dip coating to prepare conducting nanofibrillar network. In this modification process, MFC was exposed to conductive commercial polyaniline (PANI).

Finally the mechanical properties of the dried matter were studied. Five different drying methods were studied and aerogels (not collapsed upon drying) and xerogels (collapsed upon drying) with different functional properties were achieved. These aerogels with varying pore size, BET surface area, density, and mechanical properties open novel applications in materials science such as reinforced composites, functional packaging foams, catalyst supports, templates for sensors, functional surfaces, templates for membranes, and as templates for surface modification. The results from this project are presented in papers and reports (se separate paragraph).

The activities in Sub-project 6 were aimed at increasing our general understanding of superhydrophobicity and, to apply this to nanostructured cellulose products. Within this project a number of different approaches have been taken. Firstly a modelling work aimed for general understanding on wetting on superhydrophobic surfaces was con-

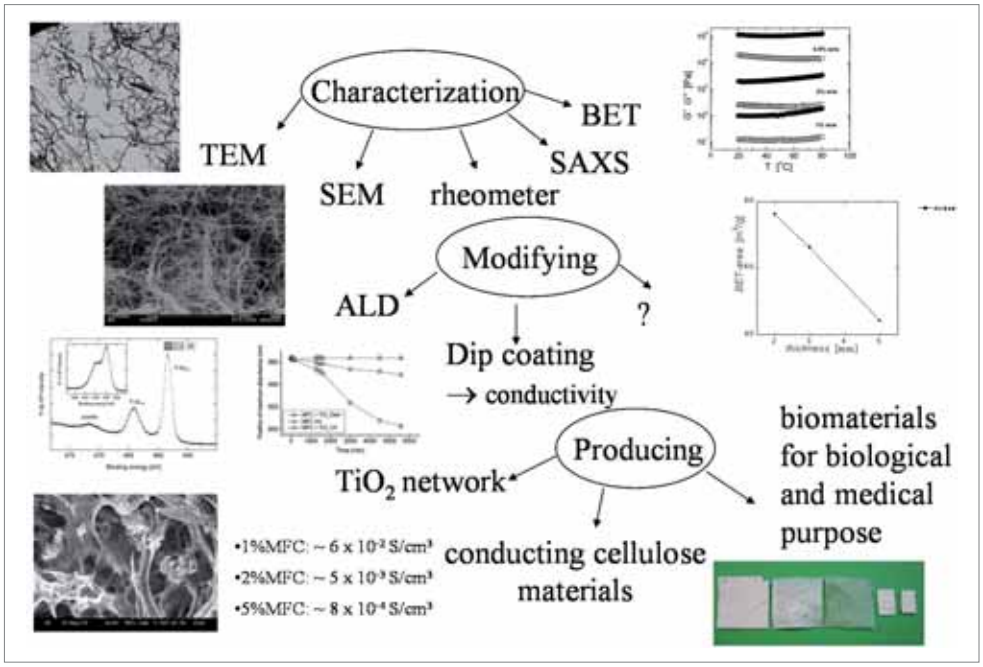

Figure 7. Schematics for new functional materials based on MFC.

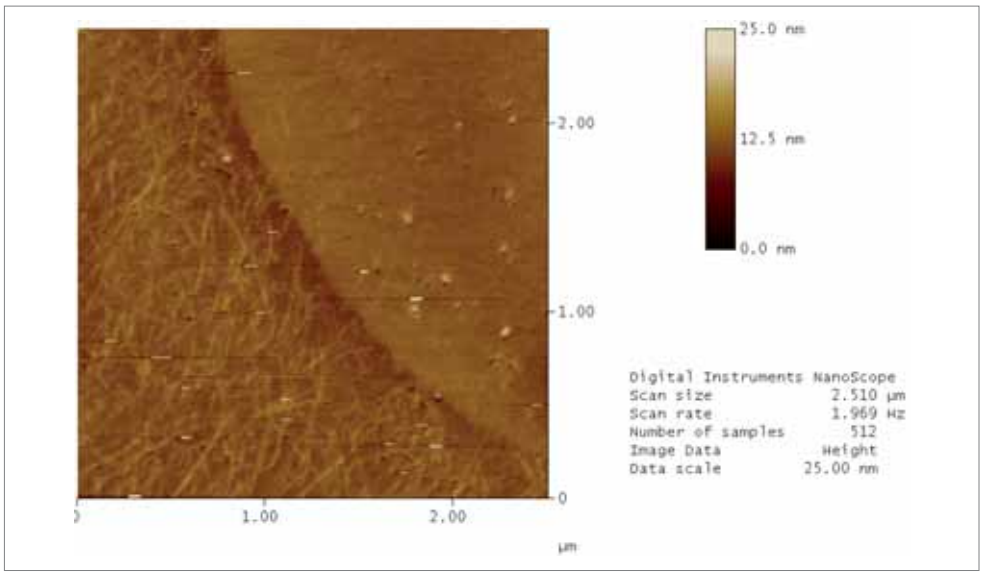

Figure 8: An AFM image taken at the border between a MFC coated silica surface and a non-coated part of the same. The size of these zones can be controlled and with present technique be made from a few micrometers and larger and up. The individual fibrils can be clearly seen in this image.

ducted. It was specially focused on the free energy barriers between the wetting states as a function of the surfaces geometric structure and the surface energy. This has resulted in a published article entitled (Werner et al, 2005). Secondly an experimental method on how to characterise the liquid-vapour interface beneath droplets has been developed. In this method an agar solution is allowed to solidify on the surface to be analysed and after solidification the droplet is removed and analysed for structure on the wetted surface. This is an initial work conducted to reach on a new type of characterisation and more work is needed to finalise this to a standard procedure. The experiences from the above projects have been used in developing a cellulose based superhydrophobic surfaces.

Within sub-project 6 a master thesis project, by Lisa Persson, conducted in order to prepare ordered MFC surfaces with the aid of micro contact printing. Since the work was successful it was continued in project form. MFC deposited on polymer treated silica surfaces giving a few nanometre thick MFC films with circular 5μm wide holes could be prepared as shown in Figure 8. This procedure might have many interesting potentials outside the preparation of superhydrophobic surfaces, for example, to be used together with sacrificial layers to form membranes. In another master thesis work within the project, by Karl Axnäs, it was also shown that it is possible to prepare multilayer with MFC and positively charged polymers (see Subproject 3). This opens up new possibilities for preparing responsive nanomaterials based on basically biological, renewable materials.

11.3 Conclusions

During this project several routes for manufacturing of MFC have been developed and in this process the energy consumption for making the MFC has been lowered from around 30000 kWh/tonne to less than 1500 kWh/tonne. The MFC can now be produced in kilogram quantities in lab scale. The different generations of MFC have been characterised using techniques such as AFM, TEM, SEM, solid state NMR, and rheolog. It has been found that the MFC fibrils are approximately 5-20 nm wide and 500-1000 nm long and can form strong gels at low concentrations.

Furthermore, the performance of MFC in different materials has been investigated. MFC can for instance be mixed into a native starch matrix which gives a strong, stiff and ductile material which can compete with commercial plastics. MFC can also be dried into foams and aerogels which in turn can be functionalised (e.g. electrically conducting). The MFC may also be ordered on a surface either together with polyelectrolytes in multilayers or deposition in ordered patterns.

11.4a Capabilities generated by the project

- Several production methods for MFC which consume much less energy.
- Better understanding of how MFC behaves and looks like.
- Utilisation of existing analysis equipments for MFC characterisation
- Development of entirely new materials containing MFC
- Foams/aerogels
- Bio-nanocomposites
- Polyelectrolyte/MFC multilayered films
- Structured surfaces
- A new network for scientists interested in MFC and its applications.

11.4b Utilisation of results

The breakthroughs concerning new manufacturing methods for MFC has been so successful that the first commercial MFC-plant now is being erected.

11.5 Publications

Ankerfors, M., Lindström, T., Larsson, T., Pääkkö, M., Ruokolainen, J., Ahola, S., Österberg, M. and Henriksson, M. "A Manufacturing Method for Microfibrillated Cellulose". 6th International Paper and Coating Chemistry Symposium 2006, Stockholm, Sweden.

Axnäs, K. "Build-up of polyelectrolyte multilayers with microfibrillated cellulose", Master of Science Thesis, KTH, 2006.

Persson, L. "Design of super hydrophobic cellulose surfaces using micro contact printing", Master of Science Thesis, LiTH, 2006.

Pääkkö, M., Ankerfors, M., Kosonen, H., Nykänen, A., Ahola, S., Österberg, M., Ruokolainen, J., Laine, J., Larsson, P.T., Ikkala, O. and Lindström, T. "Enzymatic Hydrolysis Combined with Mechanical Shearing and High-Pressure Homogenization for Nanoscale Cellulose Fibrils and Strong Gels", revised Biomacromolecules, 2007.

Pääkkö, M., Vapaavuori, J., Sivennoinen, R., Nykänen, A., Ruokolainen, J., Kosonen, H. and Ikkala, O. "Pure Cellulose Nanofibrillar Aerogels and Xerogels". Manuscript in preparation.

Pääkkö, M., Ankerfors, M., Ikkala, O. and Lindström, T. "Practical nano-chemistry: High Modulus Networks Based on Nanoscale Microfibrillated Cellulose". International Symposium on Macro- and Supramolecular Architectures and Materials (MAM-06): Practical Nano-Chemistry and Novel Approaches, poster, 2006.

Silvennoinen, R., Ras, R.H.A, Ankerfors, M., Pääkkö, M., Pore, V., Sainio, J., Ruokolainen, J., Kemell, M., Laine, J., Ritala, M. and Ikkala, O. "Novel Functional Aerogels Based on Cellulose Nanofiber Sheets Coated by Inorganic Thin Films using Chemical Vapor Deposition", revised Chemistry of Materials, 2007.

Vapaavuori, J. "Preparation of Microfibrillated Cellulose Aerogels and Xerogels by Different Freeze-Drying Techniques: Structure and Properties", Special Assigment 2006, TKK.

Werner, O. et al. "Wetting of Structured Hydrophobic Surfaces by Water Droplets". Langmuir 2005, 21, 12235-12243.

Patent applications.

11.6 National and international cooperation

Institutional parties

STFI-Packforsk AB, Sweden

Helsinki University of Technology (TKK), Finland

- Dept. Engineering Physics and Mathematics
- Laboratory of Forest Products Chemistry

Royal Institute of Technology (KTH), Sweden

- Dept. Fibre- and Polymer Technology

Industrial Partners

Finnish industrial partners

- Kemira Oyj
- M-real Oyj
- UPM-Kymmene Oyj

Swedish industrial partners

- Bim Kemi AB
- Domsjö Fabriker AB
- Eka Chemicals AB
- Iggesund Paperboard AB
- Kemira
- M-real
- SCA AB
- StoraEnso AB

Industrial partners participating in the Paper Chemistry Cluster at STFI-Packforsk

- Stora Enso AB
- M-real
- Södra Cell Mörrum
- Billerud
- Korsnäs
- Holmen Paper
- Voith Paper GmbH & Co KG
- Frantschach Pulp & Paper Austria AG (included in Mondi Packaging)
- Norske Skog

Steering committee

SCA AB, *Ulf Carlsson*

Stora Enso AB, *Göran Bengtsson*

Domsjö Fabriker AB, *Kristina Elg Christoffersson*

Bim Kemi AB, *Thord Hassler*

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M-real, *Sune Wännström*

Eka Chemicals, *Jonas Liesen*

KTH, Dept. Fibre- and Polymer Technology, *Lars Wågberg*

KTH, Dept. Fibre- and Polymer Technology, *Lars Berglund*

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TKK, Laboratory of Forest Products Chemistry, *Janne Laine*

UPM-Kymmene, *Leila Pohjola*

M-Real, *Pekka Soukas*

Kemira, *Reijo Aksela*

STFI-Packforsk AB, *Tom Lindström*

STFI-Packforsk AB, *Mikael Ankerfors*

12 New, eco-efficient, durable and high performance wood polymer composites (WPCs) and wood WPC hybrids for joinery products (ECOMBO)

FINAL REPORT	
Name of the research project	**New, eco-efficient, durable and high performance wood polymer composites (WPCs) and wood-WPC hybrids for joinery products**
Coordinator of the project	Anne-Christine Ritschkoff

BASIC SUB-PROJECT DATA	
Name of the sub-project 1	**Material & process development for highly durable WPCs for applications in door and window frame structures**
Project period	1.12.2003–31.3.2007
Organization in charge of research	SP Technical Research Institute of Sweden
Sub-project leader	Mats Westin
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SP Technical Research Institute of Sweden, Material och produkter, Brinellgatan 4 Box 857, SE-501 15 Borås, Sweden Tel. +46 10 516 51 40 mats.westin@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	301 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	72 000
VINNOVA	90 000
Other public funding	
Town of Kemijärvi	4 000
VTT	30 000
Other funding	
Swedish industry partners	90 000
Finnish industry partners	15 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder	Person months
Mats Westin, Ph.D., Assistant Centre Manager	M	SP Trätek		VINNOVA	2
Pia Larsson Brelid	F	SP Trätek		VINNOVA	2
Magnus Wålinder, Ph.D., Centre Manager	M	SP Trätek		VINNOVA	3
Jyrki Mali, Senior Research Scientist	M	VTT		Tekes	2
Johanna Lampinen, Research Scientist	F	VTT		Tekes	3
Riitta Mahlberg, Research Scientist	F	VTT		Tekes	2
Anne-Christine Ritsschkoff, Ph.D., Chief Research Scientist	F	VTT		Tekes	2
Total person-months of work conducted by the research team 16 person-month = full-time work for at least 36 h/week, paid holidays included					

Name of the sub-project 2	**Tailoring of basic raw materials**
Project period	1.12.2003–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Johanna Lampinen
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT Processes, Box 1607, 33101 Tampere, Finland johanna.lampinen@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	174 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	80 000
VINNOVA	20 000
Other public funding	
Town of Kemijärvi	4 400
VTT	34 000
Other funding	
Swedish industry partners	20 000
Finnish industry partners	16 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder	Person months
Johanna Lampinen, Research Scientist	F	VTT		Tekes	3
Riitta Mahlberg, Research Scientist	F	VTT		Tekes	2
Juha Manilla, Research Scientist	M	VTT		Tekes	2
Jyrki Mali, Senior Research Scientist	M	VTT		Tekes	3
Mats Westin, Ph.D., Assistant Centre Manager	M	SP Trätek		VINNOVA	1
Total person-months of work conducted by the research team 11 person-month = full-time work for at least 36 h/week, paid holidays included					

Name of the sub-project 3	Material science and property aspects of WPCs
Project period	1.12.2003–31.3.2007
Organization in charge of research	KTH
Sub-project leader	Magnus Wålinder
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	KTH, Building Materials/SP Trätek P.O. Box 5609 SE-114 86 Stockholm, Sweden Tel. +46 10 516 62 23 Fax +46 8 411 83 35 magnus.walinder@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	254 450
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	8 000
VINNOVA	130 000
Other public funding	
KTH	34 000
Town of Kemijärvi	450
VTT	3 400
Other funding	
Swedish industry partners	76 000
Finnish industry partners	1 600

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder	Person months
Magnus Wålinder, Ph.D., Assistant Professor	M	KTH		VINNOVA	2
Ove Söderström, Docent, Professor	M	KTH			2
Mats Westin, Ph.D., Assistant Centre Manager	M	SP Trätek		VINNOVA	0.5
Pia Larsson Brelid	F	SP Trätek		VINNOVA	0.5
Lars Elof Bryne, M.Sc., Ph.D. student	M	KTH		VINNOVA	20
Kristoffer Segerholm, M.Sc., Ph.D. student	M	KTH		VINNOVA	4
Jyrki Mali, Senior Research Scientist	M	VTT		Tekes	
Johanna Lampinen, Research Scientist	F	VTT		Tekes	
Riitta Mahlberg, Research Scientist	F	VTT		Tekes	0.5
Anne-Christine Ritsschkoff, Ph.D., Chief Research Scientist	F	VTT		Tekes	0.5
Total person-months of work conducted by the research team 30 person-month = full-time work for at least 36 h/week, paid holidays included					

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organisation
2007	Lic.	M	Lars-Elof Bryne, 1969, 1998		Ove Söderström, KTH
2007	Lic.	M	Kristoffer Segerholm, 1979, 2003		Ove Söderström, KTH

Name of the sub-project 4	**Total evaluation and dissemination**
Project period	1.12.2003–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Anne-Christine Ritschkoff
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT Technical Research Centre of Finland P.O.Box 1000, FI-02044 VTT, Finland Tel. +358 20 722 5546 Fax +358 02 722 7027 anne-christine.ritschkoff@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total subproject budget in EUR	103 730
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	32 000
VINNOVA	20 000
Other public funding	
KTH	20 000
Town of Kemijärvi	1 800
VTT	13 500
Other funding	
Swedish industry partners	10 000
Finnish industry partners	6 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder	Person months
Anne-Christine Ritsschkoff, Ph.D., Chief Research Scientist	F	VTT		Tekes	1
Magnus Wålinder, Ph.D., Assistant Professor, Centre Manager	M	KTH/SP Trätek		VINNOVA	1
Mats Westin, Ph.D., Assistant Centre Manager	M	SP Trätek		VINNOVA	1
Pia Larsson Brelid	F	SP Trätek		VINNOVA	0.5
Jyrki Mali, Senior Research Scientist	M	VTT		Tekes	1
Johanna Lampinen, Research Scientist	F	VTT		Tekes	1
Riitta Mahlberg, Research Scientist	F	VTT		Tekes	1
Total person-months of work conducted by the research team 6.5 person-month = full-time work for at least 36 h/week, paid holidays included					

Abstract

The project aimed at development of durable and high-performance wood polymer (or plastic) composites (WPCs) and novel wood-WPC hybrid materials for joinery products. WPC test samples and profiles were manufactured by injection moulding and conical extrusion technology. Assessments of the mechanical performance, moisture exposure effects, biological durability and UV resistance of manufactured samples and profiles were carried out. The results indicate that the UV-resistance of WPCs can be improved with inorganic UV protection additives. The biological durability and moisture resistance of WPCs can be significantly improved by using a modified wood component such as heat treated or furfurylated wood. Further treatments of WPC surfaces has shown to be rather poor. However, this project clearly shows that the surface treatments, paintability and glueability of WPC profiles can be successfully carried out by using an adequate surface pretreatment prior to the surface finishing. The project has also generated valuable information regarding the wood-polymer adhesion mechanisms and the micromorphology or ”inner structure” of these types of wood-thermoplastic composites. By tailoring suitable material performances, the WPC has been shown to be a suitable material for various joinery and building applications such as door and window profiles, kitchen counters, decking and railing products.

Tiivistelmä

Hankkeen tavoitteena on kehittää ruiskuvalettuja ja ekstruusio-tekniikalla valmistettuja kestäviä, korkean suorituskyvyn puukuituvahvisteisia puukomposiitteja puusepän teollisuuden tarpeisiin. Hankkeessa tutkittiin tuotettujen komposiittikappaleiden mekaanisia, säänkesto- ja biologisia ominaisuuksia. Tutkimustulokset osoittavat, että hankkeessa valmistettujen komposiittien mekaaniset ominaisuudet ovat erinomaiset. Jopa 75% puuraaka-ainetta sisältävien komposiittien lujuudet ylittivät puun karakteristisen lujuuden. Komposiittien käytön kannalta keskeisiä haasteita ovat komposiittimateriaalien kestävyys UV-valon vaikusta vastaan, biologinen kestävyys ja pintojen jatkokäsiteltävyys. Hankkeen tulokset osoittavat, että UV-valon aiheuttamia haittoja (esim. värinmuutoksia) voidaan vähentää lisäämällä komposiittien puu- ja polymeerimatriiseihin epäorgaanisia UV-suoja-aineita. Komposiittien biologista kestävyyttä voidaan parantaa merkittävästi käyttämällä esimerkiksi lämpökäsiteltyä puuraaka-ainetta. Käsittelemätöntä puuraaka-ainetta sisältävien komposiittien todettiin olevan erittäin alttiita mikro-organismien aiheuttamalla hajoamiselle. Puu-muovikomposiittien maalaus ja liimaus on käytännössä osoittautunut erittäin hankalaksi. Tämä puute on myös rajoittanut komposiittien käyttökohteita. Hankkeessa osoitettiin, että komposiittien pintojen jatkokäsittely on mahdollista, mutta vaatii onnistuakseen pinnan esikäsittelyn etsaamalla, mekaanisella karhentamisella tai plasmakäsittelyllä.

Sammanfattning

Projektets syfte var att utveckla nya ekoeffektiva, beständiga och högpresterande trä-plast-kompositer (eng. wood plastic composites, WPCs) och trä-WPC-hybridmaterial för snickeritillämpningar. WPC-provmaterial har framställts genom både formsprutning och genom s.k. konextrudering. De tillverkade proverna har utvärderats främst med avseende på mekaniska egenskaper, fuktresistens, beständighet mot mikrobiologisk nedbrytning och UV-exponering. Resultat visar att UV-resistensen hos WPCs kan förbättras genom användning av additiv i form av oorganiska UV-absorbenter. Den mikrobiologiska beständigheten och fuktresistensen hos WPCs kan förbättras markant genom att använda en modifierad träkomponent, i detta fall antingen i form av värmebehandlat eller furfurylerat trä. Vidareförädling av WPC, t.ex. genom limning, ytbehandling och målning har också studerats. Resultat visar att dessa typer av vidareförädling är fullt möjliga främst genom att tillämpa rätt sorts förbehandling av WPC-ytorna. Projektet har även genererat värdefull information gällande s.k. trä-polymer-adhesion och gällande mikromorfologi, eller den ”inre strukturen” hos dessa typer av trä-termoplast-kompositer. Genom att skräddarsy rätt sorts materialegenskaper har projektet visat att WPC är ett mycket lämpligt material för tillämpning som snickeri- och byggnadsmaterial, t.ex. i fönster- och dörrprofiler, köksbänkar, och altanmaterial.

12.1 Introduction

12.1.1 Background

In recent years there has been a remarkable growth world-wide in the production of biomaterial-based composites. In Europe the biocomposite sector has involved mainly natural (or agricultural) fibre-polymer composites (NFPC) for the automotive industry and in North America mainly wood polymer composites (WPC) for the building materials industry. The rapid development in the material science and nanotechnology, provides great opportunities for the development of new knowledge-based material combinations and technical breakthroughs. In order to be good alternatives to existing joinery products the "eco-efficient" concepts have to fulfil material and process requirements for the following properties: good resistance to weathering, resistance to decay (by fungi, bacteria and insects), good paintability and gluability, possibility for nailing, screwing and cutting with conventional tools, direction designed stiffness, shape stability, low relative creep, and low cost.

12.1.2 Objectives

The overall objective of this research is to develop durable and high-performance wood polymer composites (WPCs) and novel wood-WPC hybrid materials for joinery products. These WPCs and hybrids represent new, innovative and eco-efficient biocomposites for targeted product profiles with:

- tailored long-term durability properties for joinery products
- modified surface properties for improved mechanical performance of joinery products.

12.2 Results and discussion

WPC samples have been produced by both injection moulding and conical extrusion technology. The thermoplastic used were in both cases polypropylene and for the conical extrusion additives of about 2% coupling agent and 3% lubricants were used. The wood raw materials were conifer (50% pine and 50% spruce), birch, heat treated pine (Thermowood D, 212 °C peak temperature), furfurylated solid pine sapwood and heat treated spruce (Thermowood D, 212 °C peak temperature). These wood materials were either in the form of pure cutter chips or prepared from solid wood by a two-step grinding process allowing aspects ratios of 5–10 to be obtained. The major particle size distribution varied approximately between 20–120 mesh depending on wood species and modification type. In addition, conifer pellets from Vapo Oy were used for manufacturing of test samples using the conical extrusion process. The weight percent of wood in the injection moulded samples varied between 40–60 % and for the conical extruded samples between 60% and 80%. Additional injection moulded samples for evaluation of thin film coatings were prepared with 60, 40 and 0 % wood-content.

The conical extruded WPC samples with furfurylated pine sapwood and heat treated spruce initially involved a one-step (or direct) extrusion process, i.e. the modified wood particle types were directly fed into the conical extruder together with polypropylene pellets and certain processing additives. Initial results from durability tests of the conical extrude WPC samples showed that for a high wood content formulation of about 70–75%, a homogeneous feeding of the wood/polypropylene preblend must be achieved for an efficient processing of the WPC profiles. Therefore the WPC manufacturing procedure for the main trials in this case involved a pre-compounding process where a wood-polymer blend (granulates) were produced which then easily and more homogeneous could be fed into the conical extruder.

Strength properties

The bending strength and the bending modulus of elasticity of injection moulded and conical extruded samples were determined by a 4-point bending test (EN 408). Test specimens were conditioned in an atmosphere with a mean relative humidity of 65 % and a temperature of 20 °C. The results showed that type of wood species, wood modification and the wood-content level have a significant effect on the bending strength results. An example of these results is presented in Figure 1.

For injection moulded WPC samples, the addition of UV-pigments and nanomers, in order to improve the UV- and abrasion resistance, were studied with regard to functionality and mechanical properties. In general, the addition of UV-pig-

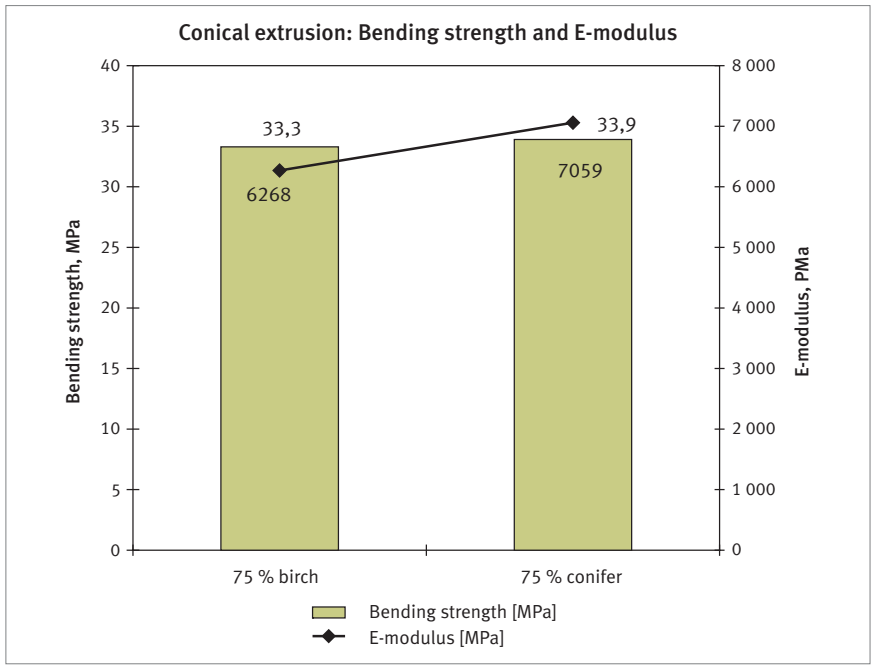

Figure 1. The bending strength and the bending MOE (EN 408, 4-point bending) of extruded samples. Raw materials: 75% wood pellets, 20% polypropylene, 3% lubricant and 2 % coupling agent.

ments slightly improved the mechanical properties compared with untreated control samples. However, the selection of the type of nanomer is critical, i.e. the chemically most suitable nanomers improved the strength properties of WPCs, whereas the chemically unsuitable nanomers decreased the strength properties up to 35-40% compared with untreated control samples.

Resistance against UV- and biological factors

The wood-polymer components are rather susceptible to environmental strain, such as weather, moisture, UV-radiation and biological organisms. Exposure to UV-light leads damage formation both to polymer and wood components in WPCs. Wood and polymeric components of WPC materials were modified with the addition of inorganic and organic UV activates in order to improve the durability of the WPC material against UV radiation. The selected UV-protection agents were added into the polymer component and/or wood component of WPCs. The assessment of the UV durability properties of WPCs were carried out in accelerated test set-up by exposing the samples to UV-light (UV-A and UV-B) and in field test conditions. The results indicate that UV durability of WPCs was clearly enhanced with inorganic UV activates. In order to achieve sufficient effect both main components, polymer and wood, should be treated with UV-agents. The long-term experiences from field test are, however, needed for final evaluation of the practical efficacy of inorganic UV protectants. Results of 3 months' outdoor exposure are shown in Figure 2.

The assessment of microbiological durability properties of WPCs included laboratory-scale soil box tests of wood-based materials (extended ENV 807 and AWPA E10), accelerated laboratory-scale mould resistance test (VTT BioBuild) as well as in-ground (EN 252) and above ground field tests (Horizontal double layer test, hazard class 3). The principles of the test setups and pictures from the field tests are shown in Figures 3–6.

The results indicated that the biological resistance of WPCs with untreated wood is not sufficient for outdoor exposure conditions. Especially this is valid for WPC samples with high wood con-

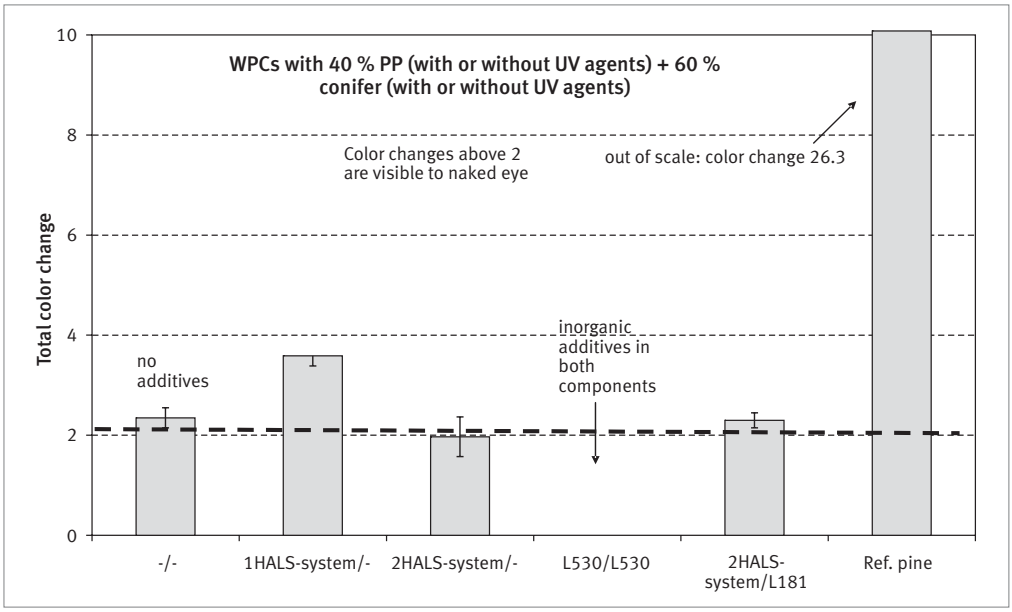

Figure 2. The effect of inorganic UV protection agents on the colour changes of WPCs during 3 months' outdoor exposure. The information on the left side of slash (/) indicates the UV agents used for PP and that on the right side refers to the UV agents used for wood.

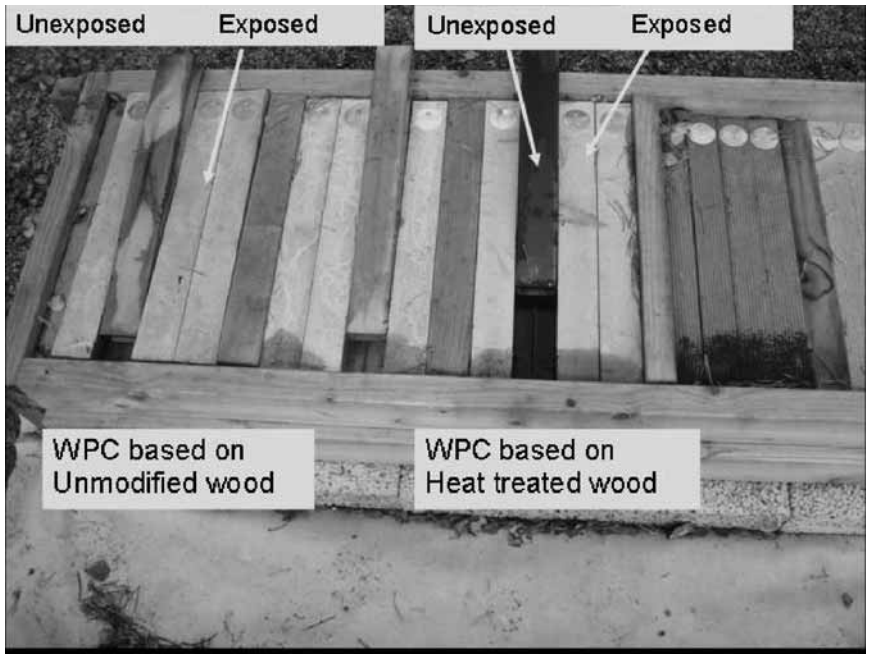

Figure 3. WPC test sample from unmodified and heat treated wood exposed above ground for 2 years ("Horizontal Double layer test").

Figure 4. In ground field testing according to EN 252 of WPCs.

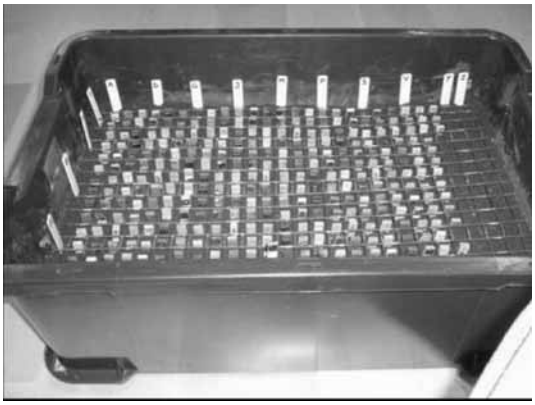

Figure 5. Laboratory TMC test set-up of WPCs according to an extended version of ENV 807.

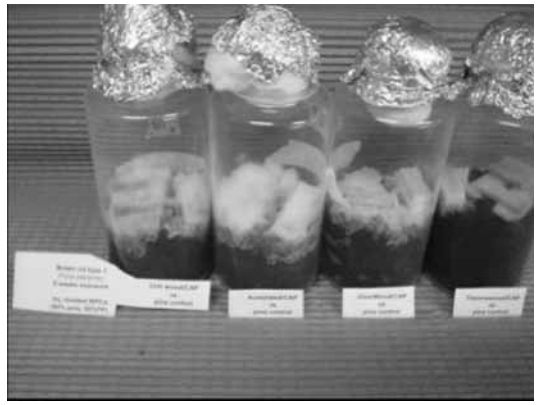

Figure 6. Laboratory test of WPCs according to AWPA E 10.

tent, see Figure 7 showing examples of the results from the laboratory TMC tests in compost and forest soils. In the samples taken out from the in ground field tests, initial decay was detected in all untreated samples, see example in Figure 8. Also unacceptable swelling and distortion occurred in the untreated samples exposed in the above ground Horizontal double layer test, see Figure 9. The biological resistance of WPC materials against fungal damages (losses in mass and strength) can be improved by using a chemically modified wood component. Rather promising results have been obtained with heat treated wood. In the WPC samples with modified wood no indication of decay could be detected in any test specimens. Also the swelling and distortion of the specimens with modified wood is minimal, see Figure 9.

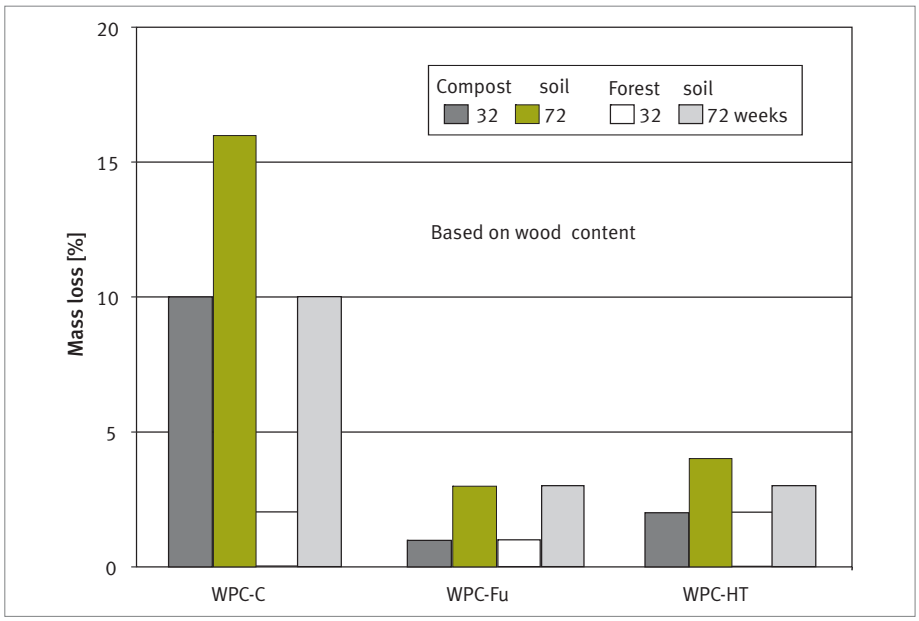

Figure 7. Results from laboratory TMC soil box tests according to an extended version of ENV 807 of WPCs (70% wood, 25 % PP, 3% lubricants and 2% coupling agent) presented as mass loss in two different types of unsterilised soils after 32 and 72 weeks.

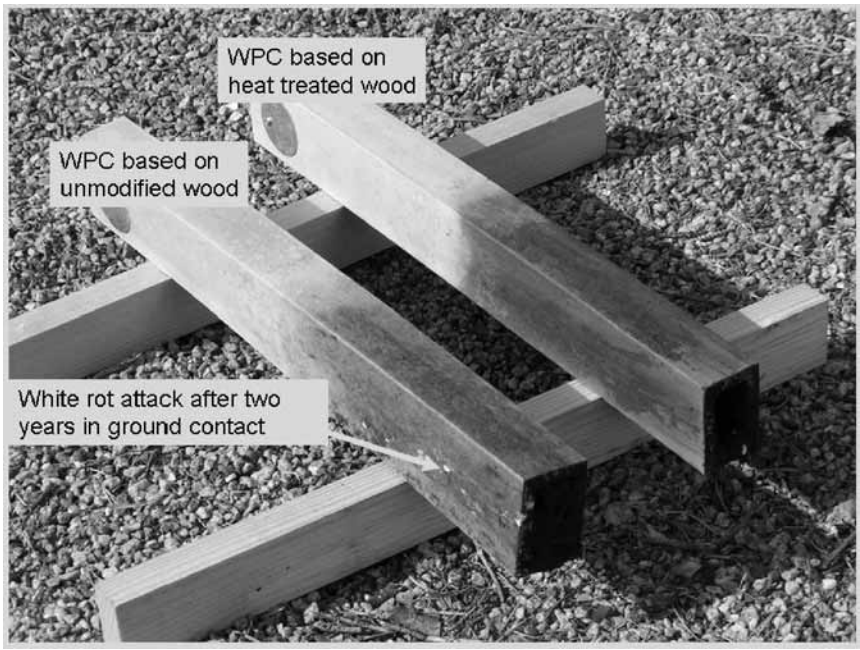

Figure 8. WPCs from in ground field tests showing white rot fungi attacked on samples with untreated wood after two years exposure while the heat treated samples showed no signs of fungal attack.

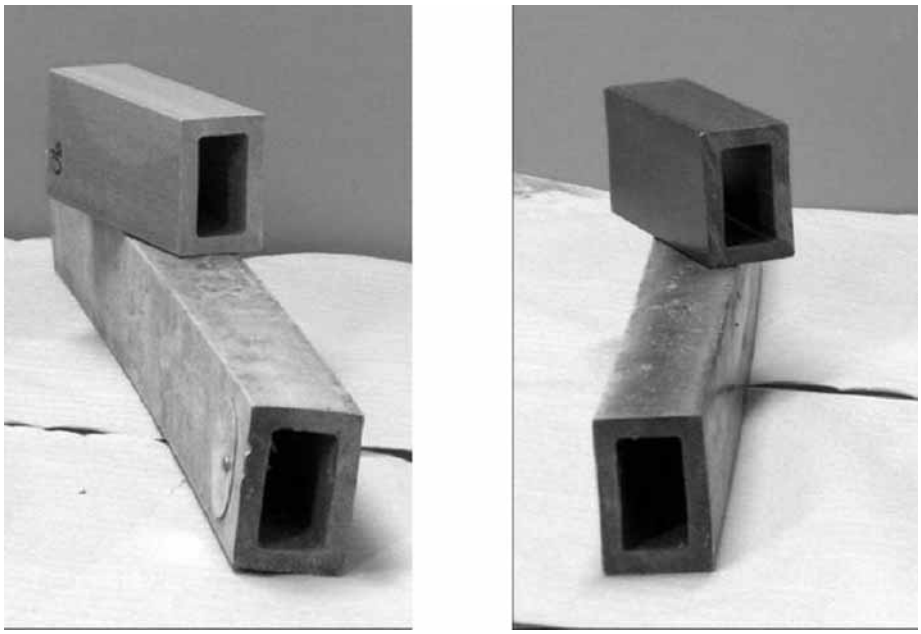

Figure 9. Dimensional changes in the extruded WPC profiles after exposure two years in the Horizontal Double layer test. The left (a) and right (b) pictures show samples with unmodified wood and heat treated wood respectively. Note the severe distortion of the samples in a).

The assessment of microbiological durability properties of WPCs included laboratory-scale soil box tests of wood-based materials (extended ENV 807 and AWPA E10), accelerated laboratory-scale mould resistance test (VTT BioBuild) as well as in-ground and above ground field tests (hazard class 3). The results indicated that the biological resistance of WPC with untreated wood is not at sufficient level for outdoor exposure condition. Especially this is valid for WPC samples with high wood content. In the samples taken out from the field test initial decay was detected in all untreated samples. Also unacceptable swelling and distortion occurred in the untreated samples. The biological resistance of WPC materials against fungal damages (losses in mass and strength) can be improved by using a chemically modified wood component. Rather promising results have been obtained with heat treated wood. In the WPC samples with modified wood no indication of decay could be detected in any test specimens. Also the swelling and distortion of the specimens with modified wood is minimal.

WPC materials with multilayer structures

Thin coating solutions based on sol-gel technology is one of the most promising method to improve material properties as well as to provide new properties to the products. The sol-gel coating solutions are used widely e.g. for hydrophobic and soil-repellent surfaces, scratch resistant surfaces and as adhesion promoters between chemically different components. The injection moulded WPC samples were coated with three different sol-gel-mediated thin coating systems. The coating systems were consisted of organically modified ceramic components to obtain repellence properties with optimal durability against mechanical strain. Best results were obtained with ceramic sol-gel coating with modified surface energy properties. However, to obtain adequate adhesion between coating and WPC material pre-treatment either by chemical

etching (acid) or plasma treatment is needed. The plasma treatment was noticed to be the most efficient pre-treatment of WPC surfaces prior the sol-gel coating deposition.

The paintability and gluability of WPCs

Surface finishing of WPC materials with non-transparent pigmented paints for aesthetic reasons are often needed. Surface treatments of extruded confer and birch WPC surfaces were carried out with four different paint systems which included alkyd oil primer/alkyd oil top coat systems (two different ones), a system with 2-component polyurethane epoxy primer with a 2-component urethane acryl top coat and a system with one layer of a 2-component polyurethane acrylic paint. Torque wrench tests showed that adhesion of the paint systems to non-pre-treated WPC surfaces is poor. Therefore, sanding, chemical etching or plasma treatments were carried out to see whether adhesion values can be increased by the pre-treatment processes. Adhesion values of all the paint systems were at least doubled by the pre-treatments. However, the systems still failed mainly at the interface of the substrate and the paints. Best results were obtained when pre-treated composite surfaces were coated with the PUR topcoat (Figure 10).

The gluing studies revealed that gluing of WPC surfaces can not be successfully carried out without pre-treatments of the surfaces. Sanding, chemical etching or plasma treatments enhance the outcome of the gluing. Acceptable gluing results are achieved by using epoxy and polyvinyl acetate adhesives on pre-treated surfaces. Cohesion of the WPC material itself will be the critical factor with these adhesives.

Cold resistance of WPC materials

The cold resistance of the WPC materials was evaluated by means of the V313 test (EN 321), which comprises of water soaking, freezing and drying (70 °C) cycles of the samples for three weeks. The exposure did not affect the bending strength of the WPC materials. However, the colour of the specimens after the exposure was somewhat lighter compared to the original colour.

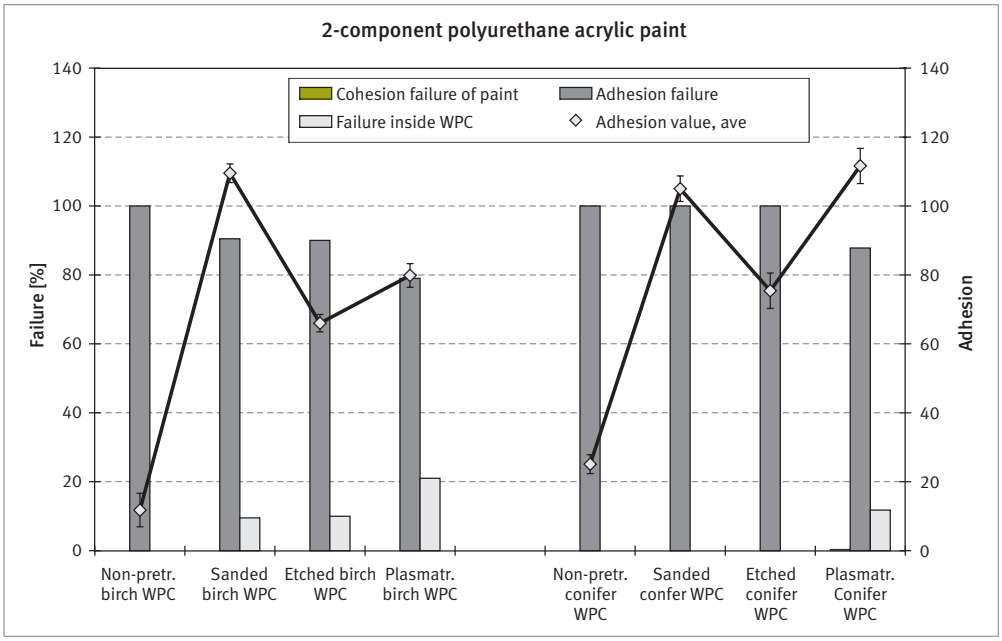

Figure 10. The effect of pre-teatments on the paintability of WPC surfaces with the polyurethane acrylic paint.

Wood-polymer-water interactions

One primary objective of this part of the work was to achieve a better insight about wood-polymer-water interactions related to WPCs emphasising on interfacial and moisture sorption problems. An effort was also made to study the effects of wood modification approaches on such wood-polymer adhesion and moisture sorption behaviour. The chemical treatments in focus were heat treatment using the Thermowood process and so-called furfurylation. Methods used were based on wetting analysis (contact angle analysis), spectroscopic techniques for surface chemical composition, and water vapour sorption experiments.

Apparent contact angles on unmodified, heat treated and furfurylated wood veneers for a series of polar and non-polar probe liquids were determined by using the Wilhelmy plate technique. These wetting parameters were then used to predict various wood-thermoplastic interaction parameters (or simply the work of adhesion) which in turn were compared with measured wood-water interaction parameters, see Figure 4. Somewhat unexpected, a reduced hygroscopicity of the bulk wood, as obtained for the included modification routes, is not equivalent with an increased hydrophobicity of the wood surface. The effects of ageing of the wood component, i.e. the ageing time after its preparation by milling, grinding etc, on the wood surface characteristics were evaluated by wetting analysis as well as spectroscopic techniques based on X-ray photoelectron spectroscopy (XPS or ECSA) and time-of-flight secondary ion mass spectrometry (TOF-SIMS) The results indicated that the surface characteristics of the unmodified wood are supposedly more influenced by the presence of wood extractives and ageing effects compared with the modified wood types.

The sorption experiments involved measurements of unsteady state water vapour sorption where dry (approximately corresponding to the state after the WPC processing) and thin "veneers" of injection moulded wood-polypropylene (50/50 weight percent proportion) composites were exposed to a climate of approx. 80 % relative humid-

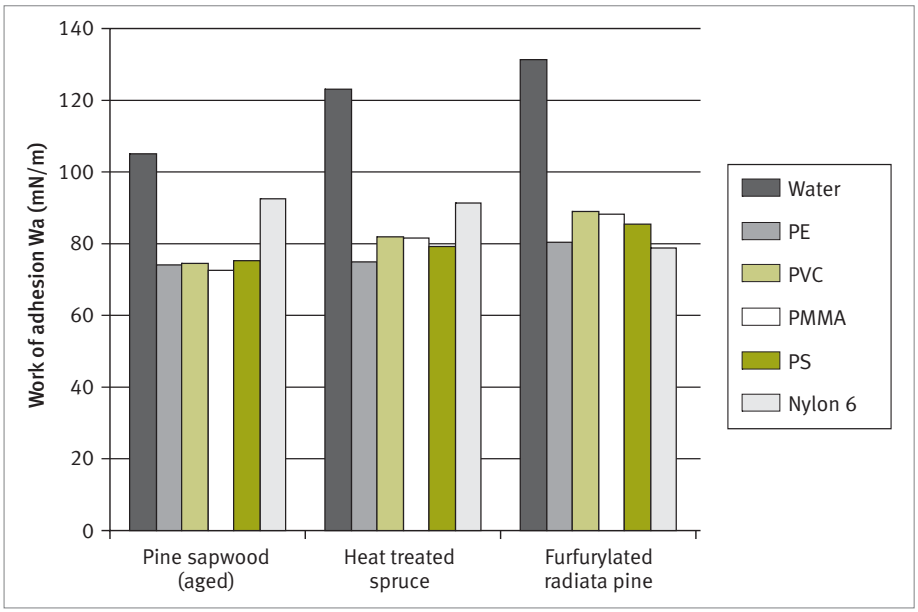

Figure 11. Predicted wood-thermoplastic-water interactions. PE = polyethylene; PVC = polyvinyl chloride; PMMA = Poly(methyl methacrylate); PS = polystyrene.

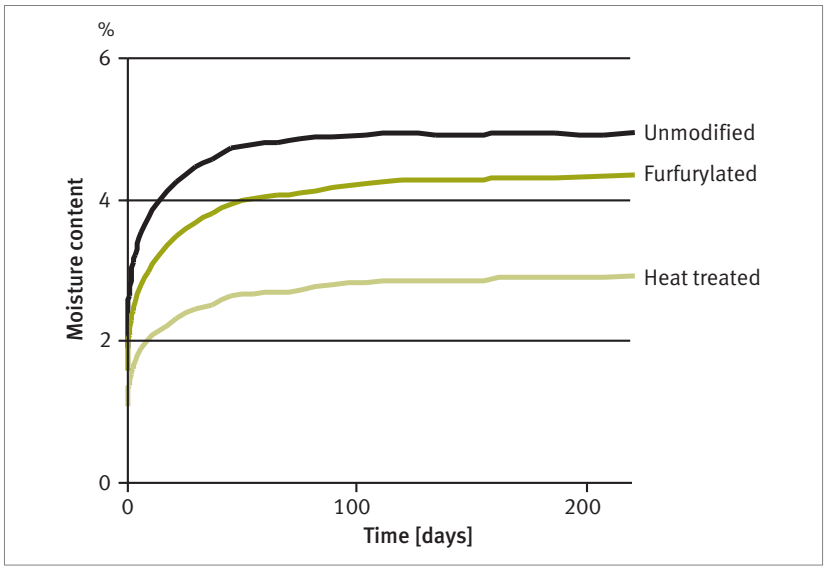

Figure 12. Moisture content versus exposure time in a climate of 80% relative humidity for injection moulded wood-polypropylene composites with an unmodified, furfurylated and heat treated wood component respectively.

ity. The measurements were carried out until an equilibrium state was reach. (Figure 12 shows the moisture content of the different WPC samples versus exposure time. In this case, the samples have also been artificial weathered in a weather-o-meter before the water vapour exposure. Results showed that, in particular the WPCs with a heat treated wood component had significantly better moisture resistance compared with controls containing an untreated wood component.

WPC micromorphology and micromechanics

This part has involved studies of the micromorphology or "inner structure" of conical extruded wood-polypropylene (70/30 weight percent proportion) composites. The effects of moisture exposure and the use of a modified wood component on the micromorphology were also studied. A low-vacuum scanning electron microscope (LV-SEM) was used to study the micro structure of the materials before and after being subjected to a moisture saturation (by immersion in liquid water) and drying cycle. In order to avoid the formation of mechanically induced microdefects, a UV-laser irradiation technique was applied as a surface preparation method for the microscopy analysis. Figure 13 shows a micrograph of the microstructure of such extruded WPCs. As can be seen, a good dispersion exists between the wood component and the polypropylene matrix, i.e. a matrix filled wood lumen and a very low over-all porosity of the WPC. Figure 14 shows a micrographs of a WPC with an unmodified wood component after subjected to such a moisture saturation and drying cycle. Results showed that, due to this moisture stress cycle a severe damage occurred within the composites based on the unmodified wood component with a frequent separation between the wood particles and the polypropylene matrix, whereas the composites based on modified wood (heat treated or furfurylated) were only slightly affected or not affected at all by the moisture stress.

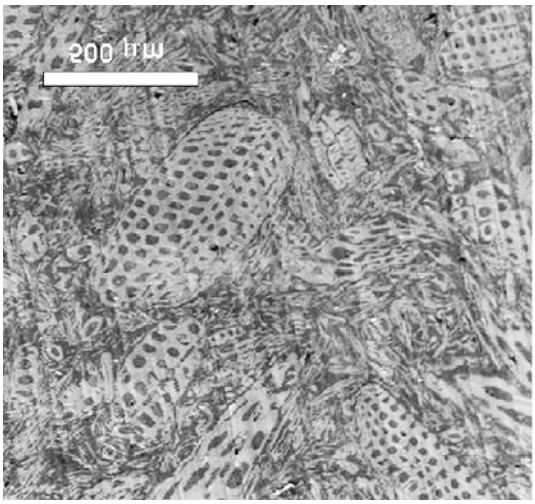

Figure 13. Micromorphology of a wood polypropylene composite with approximately 70% wood (by weight). The surface of the sample was prepared using a UV laser ablation technique.

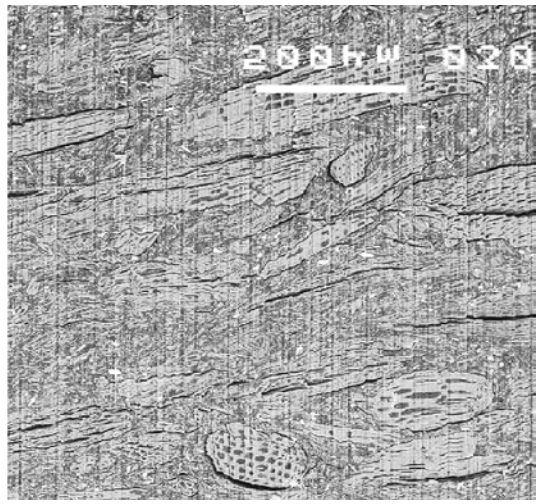

Figure 14. Micrograph of the micromorphology of a wood polypropylene composite with an unmodified wood component after subjected to a moisture saturation and drying cycle. Note the frequent interfacial cracks between the wood particles and the polymer matrix. The surface of the sample was initially prepared using a UV laser ablation technique.

12.3 Conclusions

The improvement of durability of WPC materials against UV and biological strain was one of the main objectives of the project. It was clearly shown that the UV-resistance of WPCs can be improved with inorganic UV protection agents combined with suitable radical scavengers. However, for the sufficient efficacy the active ingredients should be added both polymeric and wood components of WPCs. Unexpectedly, widely used organic UV-protection agents failed to give sufficient protection against UV-radiation. The estimation of long-term efficiency of the inorganic UV protection agents could not carried out during the project due to the unaccomplished field tests. The results from laboratory and field tests show that WPC materials with non-modified wood are highly susceptible to biological deterioration. However, the use of modified wood material improves significantly the fungal resistance of WPCs. Outstandingly good results have been obtained with heat treated wood material.

The bending strength of the conical extruded WPC profiles with wood content of 75% was shown to be better than characteristic strength of sawn timber. Addition of UV-protection agents and special chemicals (nanoceramics) did not diminish on the mechanical properties of WPCs. It was also demonstrated that temperature fluctuations from minus degrees of to plus degrees did not affect on the mechanical strength of WPC profiles.

The surface properties on wood-polymer composite materials have some disadvantageous properties. In general, the polymeric surfaces are susceptible to wearing, abrasion and soiling. In addition the adhesion on paints and glues on surfaces is shown to be rather poor, thus reducing the usability of WPCs in building and joinery applications. The abrasion resistance and anti-soiling properties of WPC surfaces can be improved with design sol-gel mediated, functional thin coating technology. Nanohybrid thin coatings with functional properties designed for WPC materials gave good resistance against scratches and soiling. Special pretreatment was, however, needed for adequate adhesion of sol-gel coatings on the WPC surfaces.

Sol-gel coatings provide an excellent tool also for future UV-protection of surfaces. The ceramic part of sol-gels itself can act as UV protection agent. In addition the sol-gel matrix can be modified with UV-protection agents. Among the pretreatments used (etching, mechanical roughening and plasma) plasma treatment gave best results regarding to adhesion and functional properties. It was also demonstrated that painting and gluing of pretreated WPC profile surfaces can be done successfully. Mechanical roughening, chemical etching or plasma treatments were efficient pretreatments for good paint and glue adhesion on the WPC surfaces. The adequate surface finishing needs, however, careful selection of paints and/or glues.

Wetting and spectroscopic analysis of wood components used for WPCs showed that unmodified wood was supposedly more influenced by the presence of wood extractives and ageing effects compared with the modified wood types. Based on the wetting analysis, a prediction of wood-thermoplastic interactions compared with measured wood-water interaction parameters indicated that a reduced hygroscopicity of the bulk wood, as obtained for the included modification routes, did not result in an increased hydrophobicity of the wood surface. It was also shown that, in particular the WPCs with a heat treated wood component had significantly better moisture resistance compared with controls containing an untreated wood component. Micromorphology studies of WPCs showed that a moisture saturation and drying cycle resulted in severe micro cracks and interfacial damage within the composites based on the unmodified wood component, whereas the composites based on modified wood were only slightly affected by the moisture stress.

In general, the given objectives for the projects were obtained in adequate manner. Exceptionally, due to the manufacturing aspects the objectives concerning the polymer matrix diversity and multilayer extrusion technology could not be met during the project.

12.4a Capabilities generated by the project

Two Swedish so-called Licentiate Thesis Degrees are expected in August or September 2007 by Lars-Elof Bryne and Kristoffer Segerholm at KTH Building Materials.

12.4b Utilisation of results

The markets for WPCs are predicted to increase. This project has provided fundamental and practical knowhow on the improvement of durability properties of WPCs against weather, UV-radiation, micro-organisms, wearing and other external strain. The project has also provided valuable knowledge on the treatability and finishing of the WPC surfaces. The results have been transferred to Finnish and Swedish polymer, wood and equipment supplying industries and they will be exploited in the product development of WPCs for Nordic and European markets.

12.5 Publications and communication

a) Scientific publications

1. Articles in international scientific journals with referee practice

To be submitted

Mali, J., Mahlberg, R., Lampinen J., Ritschkoff, A-C. (2007): Improved mechanical properties with wood fiber reinforced polymer composites technology for joinery products. Manuscript.

Westin, M., Larson-Brelid, P., Ritschkoff, A-C., Mahlberg, R., Mali, J., Lampinen, J.(2007): Biological resistance of wood polymer composites. Manuscript.

Mahlberg, R., Mali, J., Suomi-Lindberg, L., Mannila, J., Lampinen, J., Ritschkoff, A-C. (2007): UV-resistance , glueabiliy and surface treatments of wood plastic composites. Manuscript.

Bryne, L.E., Lausmaa, J., Englund, F., Wålinder, M.E.P. (2007). Surface characteristics of modified wood. Part 1. Chemical composition. Manuscript.

Bryne, L.E., Wålinder, M.E.P. (2007). Surface characteristics of modified wood. Part 2. Wettability. Manuscript.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Wålinder, M.E.P., L.-E. Bryne. 2006. Wood Adhesion Mechanisms: Prediction of Wood Thermoplastic-Water Interactions. In: Wood Adhesives 2005. C.R. Frihart (Ed.). Forest Products Society. Proceedings No. 7230. ISBN 1-892529-45-9, Madison WI, US, pp 385–392.

Mali, J., Lampinen, J., Mahlberg, R., Mannila, J. & Ritschkoff, A-C. (2006): Improved mehanical properties with wood fibre reinforced polymer composite technology. In: Ritschkoff, A-C., Koskinen, J. & Paajanen, M. (2006): Applied Material Research at VTT. VTT Symposium 244. Espoo 2006. pp. 333.

Larsson-Brelid, P., Segerholm, B-K., Westin, M. & Wålinder, M., (2006): Wood plastic composites from modified wood; Part 1 - Conceptual idea, mechanical and physical properties. The International Research Group on Wood Preservation, IRG. 37th Annual Meeting TromsØ, Norway. 18-22 June 2006.

Westin, M., Larsson-Brelid, P., Edlund, M-L. & Alfredsen, G. (2006): Wood plastic composites from modified wood; Part 2 – Durability in laboratory decay tests. The International Research Group on Wood Preservation, IRG. 37th Annual Meeting TromsØ, Norway. 18-22 June 2006.

Larsson Brelid, P. Segerholm, B, K. Alfredsen, G. Westin, M. Wålinder, M.E.P. 2006. Wood Plastic Composites with Improved Dimensional Stability and Biological Resistance. In: Proceedings of the 2nd International Conference on Environmentally-Compatible Forest Products, Ecowood. Porto, Portugal. 20-22 September. P 385-391.

Wålinder, M. (presenting author) Westin, M., Larsson-Brelid, P., Segerholm, K., Bryne, L.-E. 2005. Use of modified wood and biobased themoplastics in WPC. Invited presentation at SWST annual convention, June 19, Quebec City, Canada.

Segerholm, K. (presenting author), L.-E. Bryne, M.E.P. Wålinder, P. Larsson-Brelid, M. Westin, O. Söderström. 2005. WPCs made from modified wood - Water vapour sorption behaviour and effects on the wood-polymer interface morphology. Poster presentation at 8th International Conference on Woodfiber-Plastic Composites. May 23–25, Madison, WI, USA.

Bryne, L.-E. (presenting author), K. Segerholm, M.E.P. Wålinder, P. Larsson-Brelid, M. Westin. 2005. WPCs made from modified wood - Estimation of wood-polymer and wood-water interaction parameters. Poster presentation at 8th International Conference on Woodfiber-Plastic Composites. May 23–25, Madison, WI, USA.

Wålinder, M. (presenting author), K. Segerholm, L.-E. Bryne. 2004. On-going Research about the Use of Modified Wood in WPC. 1st Nordic Workshop on Future Trends in Wood and Bio-composite Products. December 10, Trondheim, Norway.

b) Other dissemination

Such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results.

Lampinen, J. 2005. Nordic Co-operation project Ecombo: New Eco-efficient, Durable and High Performance Wood Plastic Composites. Wood-plastic Composites 2005. Growth, Profits, Benefits and Propositions, 5-7 December, Austria Trend Eventhotel Pyramide, Vienna.

Forskning utvecklar kompositer. NTT No. 24/2005, 18–19.

Kompositer av trä och plast framtidens byggmaterial. Provning och Forskning No. 2/2005, 10–11.

IRIS Nyhetsbrev, Nr. 1 2005.

12.6 National and international cooperation

Advisory board of the project

Conenor Oy, *Markku Vilkki*

Primo AB, *Sven-Axel Nordberg*

Kemira Pigments Oy, *Esa Latva-Nirva*

Virtain Muovityö Oy, *Olavi Mäkinen*

Metsäpuu Oy, *Jorma Tiiri/Matti Sairanen*

Town of Kemijärvi, *Kari Ruokonen*

OFK Plast AB, *Olof Frisk*

Sveaskog Förvalting AB, *Urban Nordmark*

Svenska Fönsterproducenter, *Kent Wahlén*

Tanum Fönster AB, *Kjell Olsson*

Swedoor AB/Vest-Wood AS, *Jonas Bresman*

Tekes, *Juha Vaajoensuu*

Vinnova, *Eva Esping/Bengt Larsson*

WoodWisdom programme, *Kristiina Poppius-Levlin*

VTT, *Jari Koskinen*

KTH, *Magnus Wålinder*

13 Eco-efficient Modified Wood Products (ECOMOD)

FINAL REPORT

Name of the research project	**Eco-efficient Modified Wood Products**
Coordinator of the project	Mats Westin

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Novel tall oil derivatives**
Project period	1.3.2004–28.2.2007
Organization in charge of research	VTT
Sub-project leader	Salme Koskimies
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O.Box 1000, FI-02044 VTT, Finland Tel.+358207225278 Fax +358207227026 salme.koskimies @vtt.fi
URL of the project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	316 500
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	180 000
VINNOVA	6 500
Other public funding	
VTT	85 000
Other funding	
Tikkurila Coatings	15 000
Skogsindustrierna	30 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Salme Koskimies	F	VTT		Tekes/ind
Nina Heiskanen	F	VTT		Tekes/ind
Sirkka-Liisa Maunu	F	UH		Tekes
Pirita Ushanov	F	UH		Tekes
Jan Ekstedt	M	SP Trätek		VINNOVA

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 2	Coating based on tall oil alkyds and coating systems for modified wood products
Project period	1.3.2004–28.2.2007
Organization in charge of research	SP Trätek
Sub-project leader	Jan Ekstedt
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SP Trätek, Box 5609 SE-114 86 Stockholm, Sweden Tel. +46 (0)8 762 1823 Fax +46 (0)8 762 1801 jan.ekstedt@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	216 600
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	37 500
VINNOVA	70 800
Other public funding	
VTT	22 500
Other funding in cash	
Wood Focus	15 000
Other funding in-kind	
Swe/Nor industrial counter-finance	70 800

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Mats Westin	M	SP Trätek		VINNOVA
Tommy Sebring	M	SP Trätek		VINNOVA
Saila Jämse	F	VTT		Tekes
Leena Paajanen	F	VTT		Tekes
Mia Löija	F	SP Trätek		Tekes
Lone Ross Gobakken	F	NFLI/UMB		VINNOVA/WPT
Stig Lande	M	WPT		WPT

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 3	Development of quality control systems for production of modified wood
Project period	1.3.2004–31.12.2006
Organization in charge of research	SP Trätek
Sub-project leader	Mats Westin
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URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	313 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	58 400
Other public funding	
Research Council of Norway (NFR)	98 500
Other funding in cash	
WPT ASA	49 300
Other funding in-kind	
Swe/Nor industrial counter-finance	107 700

RESEARCH TEAM

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Gry Alfredsen	F	NFLI		NFR
Erik Larnøy	M	NFLI		NFR
Andreas Rapp	M		BFH, Germany	VINNOVA
Stig Lande	M	NFLI		WPT

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 4	Evaluation of specific building engineering properties
Project period	1.1.2004–31.12.2006
Organization in charge of research	Chalmers Univ. of Technology
Sub-project leader	Robert Kliger
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URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	78 900
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	17 800
Other public funding	
Faculty fund, Chalmers Univ. of Technol.	43 200
Other funding in cash	
FoU vast (part of Swedish ind. Counter-finance)	5 400
Skogsindustrierna (part of Swedish ind counter-fin.)	3 800
WPT ASA (part of Swedish ind. counter-finance)	3 200
Other funding in-kind	
Swe/Nor industrial counter-finance	5 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Hannah Epmeier	F	Chalmers		VINNOVA/CTH
Mats Westin	M	SP Trätek		VINNOVA
Charlotte Bengtsson	F	SP Trätek		VINNOVA

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 5	Evaluation of durability and "eco-efficiency"
Project period	1.3.2004–28.2.2007
Organization in charge of research	SP Trätek
Sub-project leader	Marie-Louise Edlund
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SP Trätek Box 5609, SE-114 86 Stockholm, Sweden Tel. +46 8 762 1842 Fax +46 8 762 1801 marie-louise.edlund@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	447 900
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	187 000
Other public funding	
Research Council of Norway (NFR)	73 900
Other funding in cash	
Skogsindustrierna (part of Swedish ind counter-fin.)	74 300
WPT ASA (part of Swedish ind. counter-finance)	56 200
Other funding in-kind	
Swe/Nor industrial counter-finance	56 500

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
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Pia Larsson Brelid	F	SP Trätek		VINNOVA
Susanne Ekendahl	F	SP Biolab		VINNOVA
Gry Alfredsen	F	NFLI		NFR
Julia Paravicini	F	SP/Uni-Göttingen	Uni-Gött, Germany	VINNOVA
Pawel Domagalski	M	SP/AU Poznan	AU Poznan, POL	VINNOVA
Stig Lande	M	WPT		WPT

Total person-months of work conducted by the research team 36
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	Ph.D.	F	Hannah Epmeier, 1971, 2000	Chalmers Univ. of Tech.	Robert Kliger, CTH
2006	M.Sc.	F	Julia Paravicini, 1981, 2006	Uni-Göttingen	Mats Westin, SP Trätek
2006	M.Sc.	M	Pawel Domagalski, 1982, 2006	AU Poznan	Bartek Mazela

Abstract

New durable products from modified wood, e.g. furfurylated and thermally modified wood, have emerged on the market during the last few years. This development has partly been driven by increased environmental awareness and need for reduced maintenance of joinery and garden wood products. The reasons for the claimed eco-efficiency of the new products are reduced need of maintenance and that toxic preservatives are avoided. However, at the beginning of this project, many building engineering properties of the modified woods were unknown, specifically designed surface coating systems for these products did not exist and neither did quality control systems for the production of many modified wood products. Therefore these aspects were covered through the ECOMOD Finnish-Swedish research cooperation.

Furthermore, for coating applications tall oil is an alkyd raw material of much greater importance in a Nordic perspective than alternatives such as linseed and soybean oil. The commercial alkyds based on tall oil derivatives are generally used in solvent-borne coating systems and a further challenge was therefore to develop water-borne coating systems based on tall oil derivatives.

The project was divided into five sub-projects:

1. Novel tall oil derivatives
2. Coating systems based on sub-project 1 and coating systems for modified wood
3. Development of quality control systems for production of modified wood
4. Evaluation of building engineering properties
5. Evaluation of durability and "eco-efficiency"

All sub-projects were sucessful: 1) Novel water-borne alkyd and alkyd-acrylic hybrid binders were developed, resulting in two patent applications; 2) Laboratory scale coating formulations based on these binders had promising properties and suitable coating types for differently modified wood products were identified; 3) Important steps have been taken towards fully implemented new quality control systems; 4) A lot of new data for modified timber, important in building application, has been generated and resulted in a Ph.D. degree; 5) Durability testing in laboratory and field, emission analysis and ecotoxicological testing has in a conclusive way strengthened the hypothesis that both furfurylated and thermally modified wood products are indeed eco-efficient.

Tiivistelmä

Viime vuosina markkinoille on tullut uusia kestävämpiä modifioituja puutuotteita. Keskeiset syyt kehitykseen ovat kasvanut ympäristötietoisuus, tarve vähentää puutuotteiden huoltotoimenpiteitä ja välttää myrkyllisiä lisäaineita. Esimerkkejä modifioiduista puutuotteista ovat mm. furfuryloitu ja lämpökäsitelty puu. Aloitettaessa tätä projektia monia modifioidun puun rakennusteknisiä ominaisuuksia ei tunnettu, yhtenäisiä laaduntarkkailujärjestelmiä sekä räätälöityjä pinnoitesysteemiä ei myöskään ollut olemassa. ECOMOD projekti käynnistettiin erityisesti em. seikkojen tutkimimiseksi ja kehitämiseksi suomalais-ruotsalaisena yhteistyönä.

Lisäksi on huomattava, että pohjoismaissa mahdollisuus hyödyntää mäntyöljyä alkydipinnoitteiden raaka-aineena soija- ja pellavaöljyn asemasta on liiketaloudellista merkitystä. Koska tavoitteena oli kehittää ympäristönormit täyttäviä vesipohjaisia pinnoitteita puutuotteille, lisähaasteen tutkimukselle asetti myös se, että kaupalliset alkydimaalit ovat edelleen suurelta osin luotinpojaisia.

Projekti jakaantui viiteen osaprojektiin;

1. Innovatiiviset mäntyöljyjohdannaiset
2. Osaprojektiin 1 perustuvat pinnoitesysteemit ja modifioidulle puulle räätäölöidyt pinnoitteet
3. Laatujärjestelmän kehittäminen modifioidun puun tuotannolle
4. Rakennusteknisten ominaisuuksien arviointi
5. Kestävyyden ja ekotehokkuuden arviointi.

Kaikki osaprojektit onnistuivat varsin hyvin: 1) Innovatiivisten alkydi- ja alkydi-akryyli hyprisideaineiden osalta jätettiin sisään kaksi patenttihakemusta; 2) Lupaavia pinnoiteformulaatioita perustuen uusiin sideaineisiin kehitettin laboratoriomittakaavassa ja modifioidulle puulle sopivia pinnoitetyyppejä identifioitiin; 3) Modifioidun puun laatujärjestelmän kehittämisessä edistyttiin merkittävästi; 4) Tuotettiin paljon uutta tutkimustietoa modifioidun puun rakennussovelutuksien kannalta tärkeiden ominaisuuksien osalta; 5) Laboratorio- että koekenttä olosuhteissa suoritetut kestävyystestit, emissioanalyysit ja ekotoksisuustestit osoittivat kattavasti, että sekä furfuryloitu puu että lämpökäsitelty puu ovat ekotekokkaita tuotteita.

Sammanfattning

Nya beständiga produkter från modifierat trä, exempelvis furfurylerat och värmebehandlat trä, har marknadsförts under de senaste åren. Drivkraften bakom denna utveckling har delvis varit ökad miljömedvetenhet och behov av minskad underhållsfrekvens för snickeri- och trädgårdsprodukter. Grunden till att dessa produkter marknadsförs som ”miljövänliga” är just minskat underhållsbehov och att de inte innehåller toxiska träskyddsmedel. Vid detta projekts början var många byggtekniskt viktiga egenskaper för modifierat trä okända, ytbehandlingssystem specifikt för dessa produkter existerade ej och inte heller fanns fullt fungerande kvalitetskontrollsystem för produktion av modifierat trä. Dessa aspekter ingick därför i det finsk-svenska samarbetet.

Vidare är tallolja, ur nordiskt perspektiv, en viktigare råvara för alkydfärg än alternativ såsom linolja och soyaolja. Kommersiella alkyder baserade på talloljederivat används generellt i lösningsmedelsburna ytbehandlingssystem och en ytterligare utmaning är därför att utveckla vattenburna system baserade på talloljederivat.

Projektet var uppdelat i fem delprojekt:

1. Nya talloljederivat
2. Nya ytbehandlingssystem baserade på 1 och ytbehandlingssystem för modifierat trä
3. Utveckling av kvalitetskontrollsystem för produktion av modifierat trä
4. Utvärdering av, ur byggteknisk synpunkt, viktiga egenskaper
5. Utvärdering av beständighet och ”ekoeffektivitet”

Alla delprojekt har varit framgångsrika: 1) Nya vattenburna alkyd- och alkyd-acrylathybrid-bindemedel har tagits fram, vilket resulterat i två patentansökningar; 2) Ytbehandlingssystem baserade på dessa bindemedel har visat lovande egenskaper och lämpliga ytbehandlingssystem för modifierade träprodukter har identifierats; 3) Viktiga steg har tagits mot nya fullt implementerade kvalitetskontrollsystem; 4) En mängd data för modifierat trävirke, viktiga för byggtillämpning, har genererats och resulterat i en teknologie doktorsexamen; 5) Beständighetsutvärdering på laboratorium och i fält, emissionsanalyser och ekotoxikologisk provning har på ett övertygande sätt styrkt hypotesen att såväl furfurylerade som värmebehandlade träprodukter är ekoeffektiva.

13.1 Introduction

13.1.1 Background

Wood is an excellent building material with a high strength/density ratio, it is a renewable resource and has been used successfully for centuries. However, the swelling and shrinkage movements due to variations in moisture loading (repeated wetting and drying and/or variations in relative air humidity) because of climatic variations is higher than for

most alternative building materials, e.g. concrete, plastics and steel. Furthermore, like other organic material, wood is susceptible to decaying organisms.

Durability aspects of wood products for outdoor use – Traditionally, toxic preservatives have been used to prevent microbial decay of wood. The most common preservatives have been CCA (chromated copper arsenate) and creosote (coal tar distillate). However, the use of creosote and heavy metals is nowadays highly restricted and CCA is banned for residential use in USA and Norway. In the EU a similar ban of CCA will come into force in July 2004. Alternative and more environmentally acceptable preservative treatments, e.g. heat treatment and furfurylation are emerging on the market. Furfurylation is based on use of derivatives of hydrolysis products from biomass waste (e.g. bagasse and corn cobs) and provides high resistance to microbial decay, insect and marine borer attack. However, the documentation concerning ecotoxicological data and decay resistance of modified wood from commercial production is scarce and unsatisfactory.

Wooden joinery and building products – In western and southern part of Europe, wooden windows have lost most of the market to PVC and aluminium windows, mainly due to decreased maintenance need. For the building industry, wall studs, floor beams in structures above crawl spaces and sills could be interesting products for modified wood since many methods of wood modification are known to reduce dimensional movements caused by varying moisture conditions and reduce the mechano-sorptive creep deformation. However, more research is needed before the best technical and economic solution can be proposed.

Coating systems, based on tall oil alkyds, for both untreated and modified wood – Alkyd resins are crosslinked polyesters synthesised from polyfunctional monomers. The principal raw materials involved in the manufacturing of alkyd resins are polyhydric alcohols (polyols) and dibasic acids (or corresponding anhydrides) together with the modifying oil (or corresponding fatty acids), e g linseed oil, tall oil and soybeen oil. The properties of alkyd resins depends to a large extent on the nature and amount of modifying oil incorporated in the polymer. There is an urgent need to develop water borne alkyd coating formulations to meet the present requirements concerning limitations on volatile organic compounds in paints. In this project the main challenge is to develop tailored water-borne alkyd binders and coating formulations mostly for modified wood products. These are known to have different surface properties compared to normal wood.

13.1.2 Objectives

The overall objective is to develop eco-efficient tall oil alkyd coating systems and modified wood products for use in building, joinery and outdoor applications.

In order to achieve truly eco-efficient tall oil alkyd coating systems:

- The raw materials and production processes will be chosen so that they cause minimum environmental impact.
- The systems will preferably be water-borne or of high-solids type
- The system components will have minimised content of additives causing environmental impact.
- The service life will be long.

In order to achieve truly eco-efficient modified wood products:

- The modification processes will be optimised with a minimised environmental impact in mind and quality control systems will be developed.
- The building engineering properties of modified wood will be evaluated, in order to determine the optimal use in building and joinery application and outdoor wooden constructions
- New eco-efficient coating systems, e.g. based on tall oil alkyds, will be developed specifically for modified wood
- The weathering performance of coated products and the durability against microbial decay will be assessed and thereby the expected prolonged service life of the products calculated
- The durability of modified wood in lab and field will be thoroughly assessed
- The emissions and ecotoxicological impact will be tested and analysed
- The overall environmental impacts in a life cycle perspective will be assessed.

13.2 Results and discussion

13.2.1 Novel tall oil derivatives

Several literature studies and screening trials have been carried out in order to map feasible organic synthesis routes for obtaining tall oil derivatives suitable for alkyd binders in coating systems.

Two main types of binders were developed using tall oil fatty acid (TOFA) with conjugated double bonds: self-stabilising water-borne alkyd emulsions and alkyd-acrylic hybrid binders. The latter were produced by modification of alkyds in micro-emulsion polymerisation using acrylic monomers. The binder emulsions were optimised with regard to technical properties, e g storage stability, viscosity and drying properties. The TOFA derivatives were characterised by e g NMR analysis (see figure 1).

Structures of linoleic acid and one isomer of conjugated linoleic acid are presented.

The alkyds prepared had high fatty acid content, i.e. long oil length as the air-drying alkyds were desired. The backbone alkyd resins had in general relative low content of free hydroxyl and carboxyl groups. The binders were characterized regarding their viscosity, free acid content and molecular weight distribution (by GPC). The alkyds had molecular weights generally in the range of about 1000-10000.

For the alkyd-acrylic hybrids the alkyd:acrylic ratio and emulsion solid content was analysed. Furthermore, the hybrid products were analyzed for their monomer conversion, degree of grafting, emulsion stability, average particle size and size distribution, and molecular weight range.

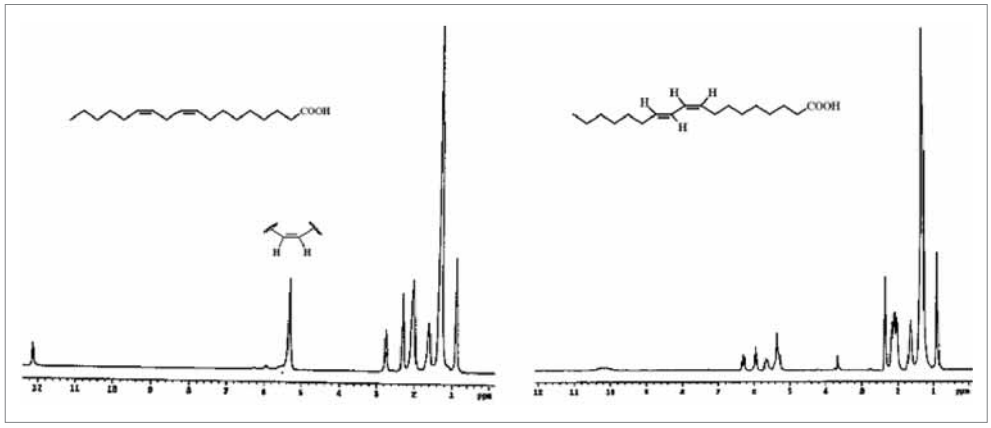

Figure 1. 1H NMR spectrum of TOFA (tall oil fatty acid) and a representative isomerisation product.

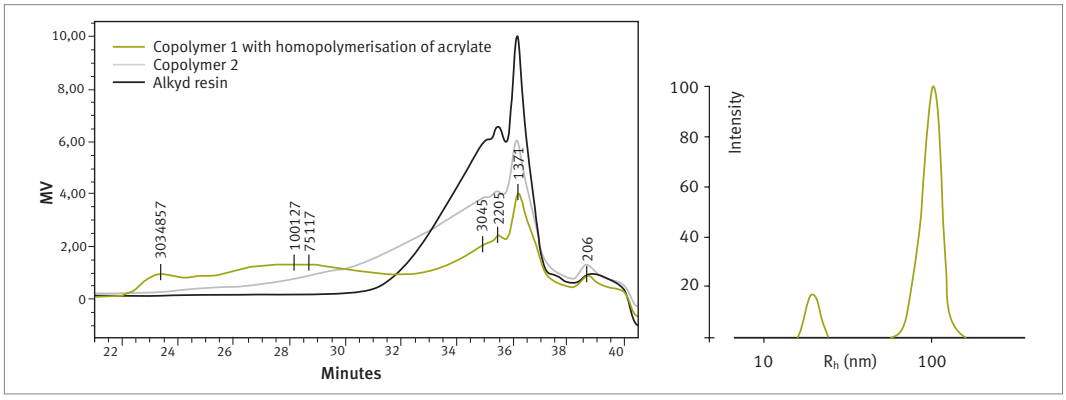

Figure 2. SEC chromatogram of an alkyd resin and copolymers. Size distribution of a copolymer product obtained by dynamic light scattering.

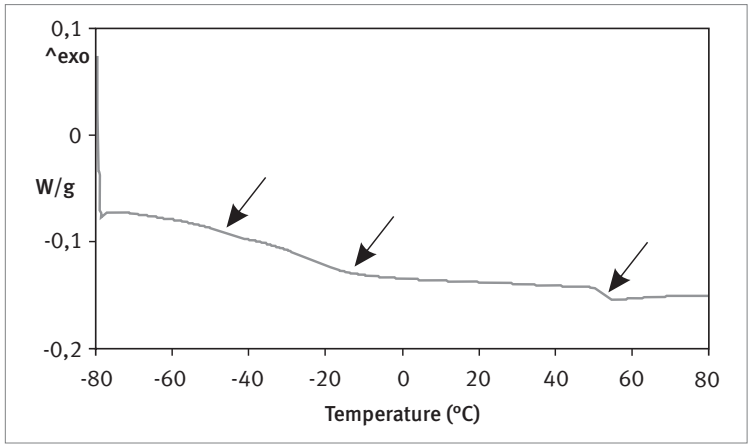

Figure 3. DSC thermogram of an alkyd-acrylic hybrid product.

13.2.2 Coating systems for modified wood and coatings from sub-project 1 binders

Coating systems for modified wood

Surface characteristics of unmodified, heat treated, acetylated and furfurylated wood veneers have been estimated by contact angle analysis. The Wilhelmy plate technique was used for determination of apparent contact angles for a series of polar and non-polar standard probe liquids. Somewhat unexpected, the results demonstrate that aged unmodified wood has less affinity to water than aged modified wood. This indicates that the surface characteristics of modified wood, which is of outmost importance for the adhesion and molecular interaction with a coating, is not necessarily unfavorable with regards to the surface energetic characteristics as compared with unmodified wood. In other words, a reduced hygroscopicity of the bulk wood, as obtained for the included modification routes, is not equivalent with an increased hydrophobicity of the wood surface.

Evaluation of a EN 927-3 field test of coated modified wood has lead to a number of conclusions, e g ThermoWood and VisorWood should be coated, in order to achieve an acceptable appearance of the materials after outdoor exposure and a semi-transparent acrylic coating performs best on all modified substrates.

Furthermore, several coating systems from Jotun have been tested on furfurylated wood in accelerated weathering tests and field tests. The results indicate that the alkyd-acrylic hybrid top coat (Demidekk Optimal) performs well on furfurylated wood without need for a primer coating.

Coatings based on binders from sub-project 1

The work has been concentrated on penetration studies of reference coatings. Penetration of reference priming oil and wood oil into the wood surface and film thickness of the coating has been monitored with fluorescence microscopy. The wood substrates included were heat treated and untreated spruce and pine heartwood and sapwood and two types of furfurylated wood (Kebony and VisorWood). Face dressing of samples before analysis was made with razor blade and for comparison with UV-laser (SP Trätek / KTH). Penetration depth was normally 1 cell layer. Film thickness varied between 0-160 micron. Penetration into furfurylated wood was poor because cell lumina contain polymers of furfuryl alcohol. The studied water borne wood oil and priming oil cannot be recommended for furfurylated wood.

13.2.3 Development of quality control systems for modified wood

Feasible analytical methods for determination of wood content of grafted furan polymer have been explored. FTIR and NIR (near infra-red) spectroscopy has been abandoned as non-destructive meth-

ods of analyses for large planks from the industrial production due to the low robustness of the models. This was explained by variation in treatment level in both longitudinal and thickness direction and to interference from cracks (matrix scatter corrections needed). However, on powdered cross section samples from the planks good estimation of average treatment level could be achieved by both these methods. It was concluded that FTIR and NIR analysis may be suitable for external quality control but not necessarily for on-line measurement. Trials with Pyrolysis GC / Thermoextraction GC were not successful and this type of analysis was dropped. However, thermogravimetric analysis (TGA) proved to be a promising method for cheap and simple estimation of treatment level.

The Thermowood companies (mainly StoraEnso and Finnforest) have developed and implemented a system for internal quality control of the Thermowood production. This system specifies requirements on incoming timber, control of process equipment and documentation. Andreas Rapp at BFH has developed a system for quality control of produced thermally modified timber through strength indicator value (SIV) and durability indicator value (DIV). The SIV is based on analysis of resistance to impact milling and the DIV is based on resistance to acid hydrolysis. The correlation between SIV and strength properties is high and so is the correlation between DIV and durability. Both the SIV and DIV can be calculated with low deviation after testing very small amounts of wood.

Finally, suggestions have been made for changes in the Nordic Wood Preservation Council documents for approval, documentation, internal and external control of preservative treated timber so that these could also include modified wood such as furfurylated.

13.2.4 Building engineering properties of modified wood

Firstly, the basic mechanical and physical properties of modified wood (Licentiate work of Hannah Epmeier) have been produced. Types of modified wood were furfurylated, oil-heat-treated (OHT), acetylated, MMF-resin treated, succinylated, maleoylated, NMA-modified and UZA-treated.

Equilibrium moisture content (EMC) in dry and humid climate was evaluated. EMC was reduced by all modifications, especially by furfurylation (more than half) and acetylation (more than 2/3). However, regarding furfurylation, the EMC is expected to be less reduced by the use of water borne formulations because of the surfactants added.

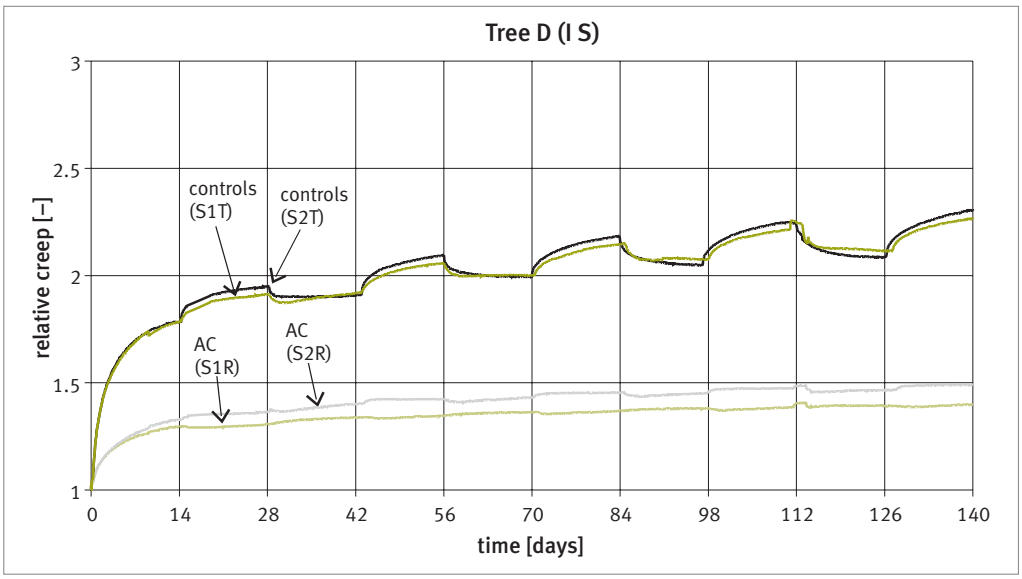

Figure 4. Creep curves due to cycling of relative humidity between 30% and 90% of unmodified and modified timber.

Property change vs. modification level was evaluated (change in comparison with unmodified pine). Impact strength, stiffness stabilisation efficiency (SSE), anti-swelling efficiency (ASE) and Brinell hardness were considered. Difference between modified and unmodified wood and also difference in dry and wet state was observed. Impact strength is reduced by all treatments, the other parameters are increased compared to unmodified pine, especially for furfurylated and acetylated wood.

Bending creep tests when cycling between 30% and 90% RH have been performed on oil heat treated (Thermex-type), melamine resin treated, acetylated and furfurylated pine. The relative creep was reduced by all modification types included (see Figure 4 as an example).

This work has been summarised in the Ph.D. thesis of Hannah Epmeier.

Furthermore, the general properties of modified woods important for building application have been summarized in a report to SBI (see publication list).

Another Ph.D. student (Kristoffer Segerholm) has also looked at moisture sorption properties of modified wood. Results from his initial trials are shown in figure 5.

13.2.5 Evaluation of durability and eco-efficiency

Durability evaluation in laboratory and field

Testing according to an extended version of the European standard ENV 807 (3 types of soil) and the American standard AWPA E10 have been performed on all modified wood types and a few reference treatments. Furfurylated wood has also been tested according to EN 113. All test specimens were pre-aged according to EN 84 before the actual tests. The standard EN 350-1 was then used to calculate the natural durability class for each group. The results were: durability class 1 (very durable) in tests with all types of fungi and in all soil types for Thermowood D (pine), Thermowood D+ (spruce), Thermex (pine heartwood), moderately furfurylated pine (WPG>30) and the reference acetylated pine. Thermex from pine sapwood varied in durability class between 1 and 3 (moderately durable), tall oil treated pine varied between 3 and 2 (durable), linseed oil treated pine had durability class 3 and Thermowood S+ (spruce) varied between 1 and 4 (slightly durable). As an example of results see Table 1.

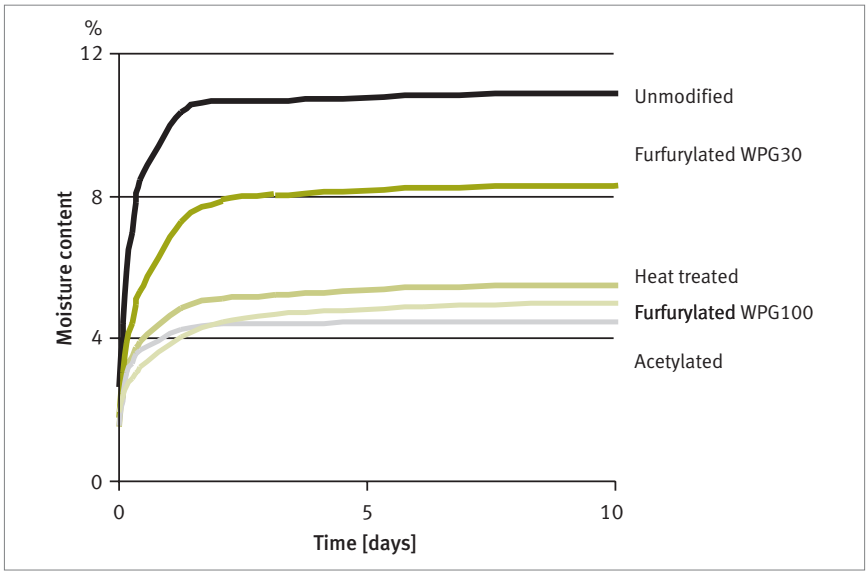

Figure 5. Moisture content curves for thin veneers modified wood in 85% RH

Table 1. Results from EN 113 tests of furfurylated wood at different treatment levels (WPG).

Test material type		Corrected mass loss (%) in EN 113 test								
Treating solution	**WPG**	**Brown rot fungus 1 *Coniophora puteana***			**Brown rot fungus 1 *Gloeophyllum trabeum***			**White rot fungus *Coriolus versicolor***		
		mass loss	*x-value*	Durab. class	mass loss	*x-value*	Durab. class	mass loss	*x-value*	Durab. class
None (Control)		54	1,00	5	45	1,00	5	39	1,00	5
VW FA 0	7	53	0,99	5	40	0,899	5	26	0,68	5
VW 1	20	3	0,05	1	12	0,267	2	4	0,11	1
VW 2	25	2	0,03	1	7	0,149	1	4	0,10	1
VW 3	40	2	0,04	1	5	0,10	1	4	0,10	1
VW 4	65	1	0,02	1	2	0,055	1	2	0,05	1

Some test specimens have been installed in an EN 275 field test of resistance against marine borers (shipworms and gribbles). All test stakes for field trials according to EN 252 (stakes in ground) and the Horizontal Double Layer test, were installed in June 2005 in Sweden (Borås and Simlångsdalen), Norway (Ås) and Poland (Poznan). Results from the first year in soil contact shows that pine controls are moderately decayed whereas the modified wood specimens are sound.

Emissions to air and water and Ecotox tests

Emission tests of furfurylated wood (VisorWood) specimens show that the emissions to air were below 1% of the permitted threshold limits for all compounds analysed. Emission tests of thermally modified wood (Thermex) show similar results. Water leachates of furfurylated wood were used for aquatic Ecotox tests with algae, bacteria, *Daphnia magna* and fish. The toxic effect on these aquatic systems was in the same order as the effect of leachates from untreated pine, which is very reassuring results. These tests have already been used in two publications. Ecotox tests with bacteria have been performed on leachates from all modified wood types and the results were similar to the results found for furfurylated wood.

Environmental impact in a life cycle perspective

LCA were made for furfurylated wood and preservative treated (CCA, Chromated Copper Arsenate) wood with a floating boat jetty as a functional unit. In this LCA, furfurylated wood came out much better than CCA treated.

Evaluation of eco-efficiency

Results for both furfurylated and thermally modified wood show that: 1. Durability is high => long service life, 2. Emissions to air, soil and water are low, 3. Ecotoxicity of emitted substances is low, 4. LCA shows that modified wood seems to be better than alternative durable material. Therefore these materials could indeed be labelled eco-efficient.

13.3 Conclusions

Promising new wood coating binder types based on tall oil derivatives have been developed. Characterization of modified wood surfaces has been done and commercial coating types have been screened leading to recommendations for coating types. Possible analysis techniques for internal and external quality control of modified wood have

been identified and tried. The work on assessment of specific building engineering properties has generated a lot of useful data. Durability, emission and ecotoxicity testing of modified wood has given results indicating long service lives for modified wood products and strengthened the hypothysis that these material could be called eco-efficient.

13.4a Capabilities generated by the project

Patent applications

Koskimies, S, Heiskanen, N., Wikstedt, M., Ahola, P., Jämsä, S., Paajanen, L. & Laamanen, S. (2006) FI.20065150, 06.03.2006, VTT.

Koskimies, S, Heiskanen, N, Willberg, P., Hulkko, J., Uschanov, P. & Maunu, S. L. (2006). FI.20065151, 06.03.2006, VTT.

Westin, M. (inventor) (2005): Furan polymer impregnated wood. European patent application No: 03771506.7-2113-NO0300248.

13.4b Utilisation of results

13.5 Publications and communication

The most important publications are indicated with an asterisk.

a) Scientific publications

1. Articles in international scientific journals with referee practice,

Epmeier, H. and Kliger, R. (2005): Experimental study on material properties of modified Scots pine. *Holz als Roh- und Werkstoff* 63:430-436.

Epmeier, H., Johansson, M., Kliger, R. and Westin, M. (2006): Properties of modified Scots pine – Material properties and their interrelation in chemically modified clear wood of Scots pine. *Holzforschung* 61(1):34-42.

Epmeier, H., Westin, M. and Rapp, A.O. (2004): Differently Modified Wood: Comparison of Some Selected Properties. *Scand. J. For. Res.,* 19(suppl. 5):31-37.

*Epmeier, H., Johansson, M., Kliger, R., and Westin, M. (2006): Bending creep performance of modified timber. Accepted for publication in *Holz als Roh- und Werkstoff*.

Epmeier, H. (2006): Modified timber as structural material – Bending creep behaviour and deformation factors. Submitted for publication in *Wood Science and Technology.*

Hadi, Y.S., Westin, M. and Rasyid, E. (2005): Resistance of furfurylated wood to termite attack. *Forest Products Journal*, 55(6): 85-88.

*Lande, S., Eikenes, M. and Westin, M. (2004): Chemistry and Ecotoxicology of Furfurylated Wood. *Scand. J. For. Res*., 19(suppl. 5):14-21.

*Lande, S., Westin, M. and Schneider, M.H. 2004: Properties of Furfurylated Wood. *Scand. J. For. Res*., 19(suppl. 5):22-30.

Lande, S., Westin, M. and Schneider, M.H. 2004: Ecoefficient wood protection – Furfurylated wood as alternative to traditional wood preservation. *Management of Environmental Quality: An International Journal,* 15(5):529-540.

*Westin, M., Rapp, A.O. and Nilsson, T. 2006: Field Test of Resistance of Modified Wood to Marine Borers. *Wood Material Science and Engineering* 1(1):34-38.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice,

Gobakken, L.R. and Westin, M. 2005: Field performance of coated modified wood panels. In proceeding of the 2nd European Conference on Wood Modification, 6-7 October, Göttingen, Germany. Page 319-330.

Koskimies, S., Heiskanen, N., Hulkko, J., Willberg, P., New fatty acid based binders and applications, VTT Symposium on Applied Materials, Espoo, June 2006

Lande, S., Eikenes, M., Westin, M. and Schneider, M. 2005: Changes in mechanical properties of furfurylated wood. In proceeding of the 2nd European Conference on Wood Modification, 6-7 October, Göttingen, Germany. Page 283-288.

Uschanov, P., Maunu, S.L., Heiskanen, N., Koskimies, S. 2006: Synthesis and Characterization of Tall Oil Fatty Acid Based Alkyd–Acrylate Hybrid Polymers Used in Coatings s from carbon dioxide. Poster at the 3rd Waterborne and High Solids Coatings - PRA Conference, Brussels, Belgium.

Uschanov, P., Maunu, S.L., Heiskanen, N., Koskimies, S. 2006: Synthesis and Characterization of Tall Oil Fatty Acid Based Alkyd–Acrylate Hybrid Polymers Used in Coatings. Poster at Nordic Polymer Days 2006, Copenhagen, Denmark

Westin, M. and Alfredsen, G. 2005: Detection of decay in modified wood using ultrasound. In proceeding of the 2nd European Conference on Wood Modification, 6-7 October, Göttingen, Germany. Page 376-380.

Westin, M., Lande, S. and Schneider, M.H. 2004: Wood furfurylation process and properties of furfurylated wood. International Research Group on Wood Protection document IRG/WP 04-40289. 13 p.

Westin, M. and Alfredsen, G. 2005: Detection of decay in modified wood using ultrasound. In proceeding of the 2nd European Conference on Wood Modification, 6-7 October, Göttingen, Germany. Page 376-380.

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Epmeier, H. (2006): Moisture-related properties of modified timber – an experimental study. Doctoral thesis. Dept of Civil and Environmental Engineering, Chalmers University of Technology. ISBN 91-7291-851-9.

B) Other dissemination (such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results).

Epmeier, H. Kliger, R., and Westin, M. 2004. Egenskaper hos modifierat virke. 50 pages. FoU-Väst Rapport 0403, Sveriges Byggindustrier, ISSN 1302-7410.

13.6 National and international cooperation

13.6.1 Steering Committe of the project

Chairman: *Stig Lande*, R&D Manager, Wood Polymer Technology ASA, Norway

Vice chairman: *Jukka Ala-Viikari*, Wood Focus and Thermowood Association

Project coordinator: *Mats Westin*, SP Trätek

Project deputy coordinator: *Salme Koskimies*, VTT

Jonas Bresman, Vest-wood AS/Swedoor AB

Pirjo Ahola, Tikkurila Oy

Marie-Louise Edlund, SP Trätek

Sirkka-Liisa Maunu, University of Helsinki

Bengt Larsson, VINNOVA

Juha Vaajoensuu, Tekes

13.6.2 Research partners in the project consortium

SP Trätek (Technical Research Institute of Sweden, Section for Wood Technology)

Subcontractor to SP Trätek: BFH (Bundesforschungsanstalt für Forst- und Holzwirtschaft), Germany

VTT (Finnish Technical Research Centre)

University of Helsinki, dept of Polymer Chemistry

Norwegian Forest and Landscape Institute (Skogforsk), Ås, Norway

Research partners from Industry: AB Bitus, Ekopine Oy, Forchem Oy, Ingarps Träskydd AB, Jotun AS, Swedoor/Vest-wood AS, Tanum Fönster AB, Tikkurila Coatings Oy, Wood Focus Finland, Wood Polymer Technologies ASA

13.6.3 Other contacts outside the project

University of New Brunswick, Fredricton, Canada

University of Toronto, Toronto, Canada

Toxicon AB, Landskrona, Sweden

14 Innovative eco-efficient high fire performance wood products for demanding applications (InnoFireWood)

FINAL REPORT

Name of the research project	**InnoFireWood – Innovative eco-efficient high fire performance wood products for demanding applications**
Coordinator of the project	Dr Birgit Östman

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Chemical and biochemical modification**
Project period	1.1.2004–30.6.2006
Organization in charge of research	SP Trätek / Wood Technology
Sub-project leader	Dr. Birgit Östman
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SP Trätek, Box 5604 SE-114 86 Stockholm Tel. +46 10 516 6224 Fax +46 8 411 8335 birgit.ostman@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	250 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	120 000
Other funding	
KTH Biotechnology	35 000
SP Trätek	5 000
Industry	90 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Dr. Birgit Östman, Research leader	F	SP Trätek		
Dr. Lazaros Tsantaridis, Research Scientist	M	SP Trätek		

Dr. Harry Brumer, Docent	M	KTH Biotechnology
Dr. Peter Piispanen, Post-Doctoral Scientist	M	KTH Biotechnology
Dr. Esko Mikkola, Senior Research Scientist	M	VTT
Dr. Tuula Hakkarainen, Senior Research Scientist	F	VTT
Total person-months of work conducted by the research team 12 person-month = full-time work for at least 36 h/week, paid holidays included		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
			No academic degrees		

Name of the sub-project 2	Physical modification and structural means
Project period	1.1.2004–30.6.2006
Organization in charge of research	VTT
Sub-project leader	Dr. Esko Mikkola
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O. Box 1803 FI-02044 VTT, Finland Tel. +358 20 722 4825 Fax +358 20 722 4815 esko.mikkola@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	80 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	50 000
Other funding	
VTT	10 000
Industry	20 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Dr. Esko Mikkola, Senior Research Scientist	M	VTT		
Dr. Tuula Hakkarainen, Senior Research Scientist	F	VTT		

Dr. Jukka Hietaniemi, Senior Research Scientist	M	VTT
Dr. Birgit Östman, Research leader	F	SP Trätek
Dr. Lazaros Tsantaridis, Research Scientist	M	SP Trätek
Total person-months of work conducted by the research team 6 person-month = full-time work for at least 36 h/week, paid holidays included		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
			No academic degrees		

Name of the sub-project 3	**Fire performance and classification**
Project period	1.1.2004–30.6.2006
Organization in charge of research	VTT
Sub-project leader	Dr. Esko Mikkola
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O. Box 1803 FI-02044 VTT, Finland Tel. +358 20 722 4825 Fax +358 20 722 4815 esko.mikkola@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	100 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	58 000
Other funding	
VTT	19 000
Industry	23 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Dr. Esko Mikkola, Senior Research Scientist	M	VTT		
Dr. Tuula Hakkarainen, Senior Research Scientist	F	VTT		
Dr. Jukka Hietaniemi, Senior Research Scientist	M	VTT		

Dr. Kaisa Belloni, Senior Research Scientist	F	VTT
Dr. Birgit Östman, Research leader	F	SP Trätek
Dr. Lazaros Tsantaridis, Research Scientist	M	SP Trätek
Total person-months of work conducted by the research team 6 person-month = full-time work for at least 36 h/week, paid holidays included		

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
			No academic degrees		

Name of the sub-project 4	**Eco-efficiency and durability in different end uses**
Project period	1.1.2004–30.6.2006
Organization in charge of research	SP Trätek / Wood Technology
Sub-project leader	Dr. Birgit Östman
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	SP Trätek, Box 5604 SE-114 86 Stockholm Tel. +46 10 516 6224 Fax +46 8 411 8335 birgit.ostman@sp.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	140 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	75 000
Other funding	
SP Trätek	5 000
Industry	60 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Dr. Birgit Östman, Research leader	F	SP Trätek		
Dr. Lazaros Tsantaridis, Research Scientist	M	SP Trätek		

Dr. Esko Mikkola, Senior Research Scientist	M	VTT
Dr. Tuula Hakkarainen, Senior Research Scientist	F	VTT

Total person-months of work conducted by the research team 6
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
			No academic degrees		

Abstract

The basis for a new generation of innovative wood products has been developed in the InnoFireWood project. The main characteristics of such products are substantial improvements in fire performance with maintained eco-efficiency, durability, and other properties of the original wood product. Industrial relevance, sustainable production, and end-use applications were in main focus. This type of products will increase the competitiveness of wood products in relation to other building products.

A new Nordic system with classes for the Durability of the Reaction to Fire performance (DRF classes) at interior and exterior building applications has been implemented in order to assess the overall sustainable performance of the new products.

Tiivistelmä

Tuotekehityksen perusteita uuden sukupolven innovatiivisille ja kestäville vaativiin kohteisiin tarkoitetuille palosuojatuille puutuotteille kehitettiin suomalais-ruotsalaisessa tutkimuksessa.

Kehitettyjen tuotteiden palo-ominaisuudet paranivat, mutta niiden työstettävyys ja ulkonäkö säilyivät ennallaan. Menetelminä käytettiin kemiallisia, biokemiallisia ja fysikaalisia modifiointimenetelmiä sekä näiden yhdistelmiä. Palosuojattu puu ei tuota palaessaan lämpöä niin paljoa, että se edistäisi palon leviämistä.

Sammanfattning

Basen för en ny generation av innovativa, beständiga och miljöanpassade brandskyddade träprodukter för krävande tillämpningar har utvecklats i ett finsk-svenskt projekt. Ett nytt nordiskt system (NT Fire 054) som klassificerar brandskyddets beständighet i s k bruksklasser (eng. DRF classes) för inomhus- och utomhusbruk i byggnader har tillämpats. Flera nya produktidéer har tagits fram genom kemiska, biokemiska och fysikaliska modifieringar. Brandklassning, simulering och brandskyddets beständighet och miljöbedömning har ingått.

14.1 Introduction

14.1.1 Background

Fire performance is one of the main obstacles to an increased use of wood in buildings in most countries. New European classification systems for fire performance have recently been agreed, but the national safety levels will remain, causing continued limitations in the use of wood products. Chemical and physical modifications together with composite solutions are efficient ways to produce high fire performance wood products. However, the durability of the fire performance as well as the overall performance of new innovations have been unsolved problems for a long time. Now, new systems have become available.

New products will enlarge the possibilities to use wood on macro-scale in buildings and other demanding applications such as ships and vehicles in general. The results of the project will enhance the competitiveness of wood products compared to other non-renewable building materials that do not function as a CO_2-storage.

Fire retardant (FR) treatments may improve the fire performance of wood products considerably through reducing ignitability, rate of heat release and flame spread. Wood products treated with fire retardants can readily meet the highest requirements of combustible materials, i.e., their reaction-to-fire performance is as high as e.g. that of coated gypsum boards, coated mineral wool and steel plates with coatings.

Improving the fire performance is in principle a straightforward task comprising the generation of new chemicals and treatment procedures and testing the fire performance of the resulting products. In practise, however, the costs of such work are so high that any systematic R&D efforts may constitute a severe, if not excessive, burden to the mainly small and medium-sized companies in the field.

In addition, the excellent fire performance of the virgin FR wood products may degrade with time, especially in outdoor applications. This is due to the fact that the chemicals used to generate the FR properties are water-soluble compounds most often based on phosphorous and/or boron containing compounds or mixtures of compounds. Thus, when exposed to high humidity, the FR chemicals may migrate in the wood towards the surface and may ultimately be leached out. Even at moderate outdoor humidities and indoors, the fire performance may deteriorate because the FR chemicals migrate away from the surface towards lower concentration regions deeper inside the material thus increasing flammability of the product.

The durability of fire retardant treatments at humid conditions is closely linked to the water solubility of the chemicals used and may therefore be improved by chemical techniques. Improved humidity resistant fire retardant compounds are obtained by introducing chemicals lowering water solubility. A further improvement can be achieved with compounds based on polymeric resin systems, which can be used in all indoor and outdoor situations. Physical and structural modification methods may be less sensitive to the exposure conditions and thus more durable in different end-use conditions.

14.1.2 Objectives

The project will develop the basis for a new generation of innovative wood products whose main characteristics are substantial improvements in the fire performance with maintained eco-efficiency, durability and other properties of the original wood product. These new products will be obtained by modifying the wood by chemical, biochemical and physical means. Industrial relevance, sustainable production and end-use applications are in main focus. Such products will increase the competitiveness of wood products in relation to other building products.

The project will thus contribute to the frontier of knowledge in the field of high fire performance wood products with maintained eco-efficiency and sustainability.

The project will also build a knowledge base by promoting core competence and multidisciplinary research. The transfer of new knowledge will be enhanced by networking between research and industry.

14.2 Results and discussion

Summaries of the different sub-projects and key results are presented below.

14.2.1 Chemical modification

Several types of chemicals have been applied by vacuum pressure impregnation into solid wood panelling products, thermally modified timber and furfurylated wood products. For plywood two routes have been employed, addition of fire retardants to the glue and FRT top veneers, separately and in combination.

The fire performance of the new products has been studied by testing in the cone calorimeter (ISO 5660). Full scale performance and fire classification have been predicted by four models. The new Nordic system with Durability of Reaction to Fire performance (DRF) classes has been implemented in order to obtain products suitable for sustainable end use in buildings. Key results include

- The new FR products had either improved fire performance (predicted Euroclass B or C) or maintained low moisture content (< 30 %) at high RH. Few products fulfilled both criteria, which is needed for reaching DRF class INT for interior building applications. An optimization matrix for evaluating the fire and moisture sensitivity properties of FR wood products has been developed.
- Two FR treatments have been superior: Dequest at all retention levels studied and Novaflam at the higher retention levels studied. Thermally modified timber may be FR treated after, but not before, the heat treatment and Euroclass B may be reached. Furfurylated wood may be FR treated either before or after the furfurylation and Euroclass B may be reached.
- Plywood obtained improved fire performance only when the surface veneer is FR treated and only with a few FR treatments. FR additions in the glue did not obtain sufficient effect, not even if the veneer is very thin, about 0,5 mm.
- Fire rated building panels with wood surface may consist of a wooden veneer on a non-combustible substrate. The choice of a high density substrate is most important for the fire classification. With very thin veneers and limited combustibles from top lacquer and adhesive, even Euroclass A2 might be reached, which is the classification for paper-faced gypsum boards.
- The models used to predict the full scale fire behaviour and fire classification of chemically modified wood have different features, the Trätek model being most conservative and the new Rule of thumb approach most liberal. However, narrow peaks sometimes make predicting difficult.

14.2.2 Biochemical modification

The possible use of xyloglucan (XG) as a novel component in FR chemicals to provide permanently FR solid wood products has been explored for the first time. The ability of XG to act as a molecular anchor for functional chemistry on cellulose fibers had been previously established in pulp and paper chemistry. This work allowed us to extend the technology to solid wood materials, which are inherently more difficult to modify due to a higher level of structural complexity.

It has been shown that XG conjugates can be adsorbed to wood and that conjugation of chemicals to XG results in improved retention of those chemicals. These effects parallel those observed for wood pulp fibers and highlight the generality of the XET/XG method, in addition to pointing out certain limitations. In particular, the three-dimensional structure of wood and the limited porosity of the wood structure limit the rate at which wood fibers can be modified relative to pulped fibers. The addition of XG failed to improve fire properties to any notable extent, at maximum Euroclass C might be reached, but the presence of XG was not detrimental to fire performance in the situations studied. With respect to improving fire retardancy, clearly more work is needed.

14.2.3 Physical modification

Based on the general principles of physical modification, the following cases were studied: Effect of weather resistance varnish on FR treated wood products, Colour effects, Al foil protecting plywood veneers and FR glue used in okume plywood with UV lacquer. Key results include

- FR treatment may lose its effectiveness when the surface is covered with a varnish. The reaction-to-fire behaviour can be of the same order or even inferior to the performance of untreated specimens.
- The differences between wood products of different colours are relatively small, having no effect on the European reaction-to-fire classification of the product.
- Al foil with a minimum thickness of 50 µm can effectively protect plywood veneers underneath and improve the classification of the product.

The Al foil should be placed as close to the surface of the product as possible. The thickness of the whole product can be crucial for classification due to the increasing heat release towards the burn-through of the specimen.

- FR glue in plywood can improve the classification of plywood. However, the results may vary due to the narrow heat release peak caused by the topmost veneer in the beginning of the test. It is also noted that fire retardants may increase smoke production, leading to smoke class s2.

14.2.4 Fire classifcation and simulation

Two relevant systems for the reaction to fire classification of FR wood products have been presented: the European system for construction products and the maritime regulations. Since major test methods in these systems are in medium scale, the need for predictive tools including fire simulations has been emphasised. Special attention has been on predicting the Euroclasses based on small scale data from the cone calorimeter test (ISO 5660).

Available tools have been reviewed and a new simple Rule of thumb tool especially for FR wood products has been proposed. The comparisons of experimental and predicted Euroclasses show that the rules of thumb predict the classification too pessimistically in some cases, whereas the estimates based on modelling calculations tend to be too optimistic. It is noted, however, that a sharp heat release peak in the beginning of a fire test makes the results very sensitive to variations.

A progressive and illustrative tool for simulating fire development is the Fire Dynamics Simulator (FDS) program. It can be used for simulating systems of varying complexity, ranging from a simple small-scale test for a single material to whole buildings including different materials and structures. Two FR treated prototype products and untreated spruce were simulated in different room fire conditions using FDS. The results clearly show the improvements in flashover times when FR wood products are being used as wall coverings. According to the results increase in the room height usually seems to delay the flashover time more than increase in the floor area.

14.2.5 Durability of fire performance in end-uses

The durability of the fire performance at exterior applications has been studied by accelerated ageing according to NT Fire 053 Method A, i e exposure to 12 one-week cycles of simulated rain and drying. Some of the most promising FR treatments with chemical modification have been included. Two paint systems (based on alkyd and linseed oil) have been used to maintain the fire performance properties of FR wood products at exterior applications. The same products are being exposed to natural weathering at a test field close to Stockholm. Key results include

- Most fire retardant treatments with surface coats have maintained the initial fire performance after accelerated ageing and are thus DRF class EXT.
- Without the surface coat the fire performance after ageing may be as low as for untreated wood.
- Three FR wood products maintained their initial fire performance after accelerated ageing: Dequest (at two retention levels) and Bayhibit with both paint systems and BSM 2000 only with the alkyd paint.
- A compatible combination of FR treatment and surface coat system has to be chosen in each case.
- Mass loss may be used as an indicator for loss of FR chemicals and failure to maintain the initial fire performance during weather exposure.

14.2.6 Eco-efficiency in end-uses

The choice of fire retardant chemicals is most important for the environmental profile of the final wood product. In this study, the basic principle has been to choose chemicals with an environmentally safe profile and to check all available information. Halogenated compounds have been completely avoided. Environmental risks are also associated with degradation products during normal product use and during fire conditions. Key results include

- The importance of choosing environmentally safe fire retardants, e g non-halogenated, has been emphasised in order not to loose the environmentally safe profile of the original wood product.

- The need for standardisation of methods and models for evaluating degradation has been emphasised.
- Options to develop product and environmental declarations are described.
- The principles for a systematic design tool have been proposed to assess important fire properties in combination with essential life cycle and eco-toxic parameters in end-use applications.

14.3 Conclusions

Main conclusions are:

- Euroclass B (i.e. highest possible class for wood products) can be reached for several types of treatments.
- The possible use of xyloglucan as a novel component in FR chemicals to provide permanently FR solid wood products has been explored for the first time with promising results.
- A new Nordic system for the Durability of Reaction to Fire (DRF) performance of FR wood products in interior and exterior building applications has been implemented for the new products.
- Several FR treatments have low hygroscopicity similar to untreated wood and are thus DRF class INT. Most FR treatments with surface coats have maintained their initial fire performance after accelerated ageing and are thus DRF class EXT. Without the surface coat the fire performance after ageing may be as low as for untreated wood.
- A new simple Rule of thumb tool to predict the full scale fire performance based on small scale fire testing especially for FR wood products has been proposed.
- A progressive and illustrative tool for simulating fire development is the Fire Dynamics Simulator (FDS) program has been developed.
- The importance of choosing environmentally safe fire retardants, e g non-halogenated, has been emphasised in order not to loose the environmentally safe profile of the original wood product.
- The principles for a systematic design tool have been proposed to assess important fire properties in combination with essential life cycle and eco-toxic parameters in end-use applications.
- The methodologies used for fire testing, prediction of fire classification, durability of fire performance and fire simulations have been successful and it is recommended that these methodologies are used in further studies.

14.4a Capabilities generated by the project

The project results form the technological basis for new innovative, durable and eco-efficient wood based products with high fire performance for demanding applications to be primarily utilised by the project partners and later on by others as well. Capabilities also include an increased awareness by different players that such products are being useful tools to an increased use of wood.

Industrial views on the InnoFireWood project have been compiled by the end of the project:

- Has the project contributed or changed your opinion on possibilities for using or producing fire retardant wood products?
- What is the most valuable project result for your company?
- Have you got any new product ideas?
- Do you have any new products being considered for commercialisation? If so, how many?
- How do you see the future possibilities of high fire performance wood products?

Totally seven answers from the industry partners were received. The industrial comments are mainly positive in terms of increased industrial awareness, insight and competence in possibilities and problems with fire retardant wood products and in terms of new ideas on how to proceed within the company. It is obvious that much work remains to be done by industries themselves. Companies participating actively in the project have expressed a positive attitude, while those being less active or not participating are more neutral or have not responded at all.

14.4b Utilisation of results

Companies will utilise the results in their further product development and in producing background information for new market promotions. Results will also be used in international scientific and industrial networking.

Target groups for dissemination of results outside the core project group are those influencing the choice of building materials, national authorities and relevant scientific spheres, being approached by presentations in professional magazines and at international and national conferences and seminars.

14.5 Publications and communication

a) Scientific publications

The following scientific publications based on the InnoFireWood project results have been issued so far.

Hakkarinen, Mikkola, Östman, Tsantaridis, Brumer, Piispanen: *InnoFireWood – Innovative eco-efficient high fire performance wood products for demanding applications.* State-of-the-art Report, May 2005.

Östman B, Tsantaridis L: *New innovative eco-efficient high fire performance wood products.* FRPM'05, 10th European Conference on Fire Retardant Polymers, Berlin, September 2005.

Hakkarainen T and Mikkola E: *Palosuojattujen puutuotteiden palokäyttäytymisen (The estimation of fire retardant treated wood products)* Pelastustieto, vol 55, pp 83-87, 2005. (In Finnish)

Östman B A-L, Tsantaridis L D: *New innovative eco-efficient high fire performance wood products*, submitted to FRPM '05, 10th European Book on Fire Retardancy and Protection of Materials, 2006.

Hakkarainen T and Mikkola E: *Improved fire performance for wood-based products*, VTT Symposium on applied materials, Espoo, Finland, 2006.

Hakkarainen et al: *Test and modelling results of VTT*, Internal report of the InnoFireWood Project, www.vtt.fi/proj/innofirewood. June 2006.

Östman, Tsantaridis, Mikkola, Hakkarainen, Belloni, Brumer and Piispanen: *Innovative eco-efficient high fire performance wood products for demanding applications.* Final report for Vinnova-Tekes project InnoFireWood. SP Rapport 2006:30, 2006.

Some further scientific papers are planned based on the InnoFireWood project results:

- Chemical modification (SP Trätek)
- Biochemical modification (KTH and SP Trätek)
- Physical modification (VTT and SP Trätek)
- Fire modelling (VTT)
- Euroclass prediction based on limiting values (VTT and SP Trätek)

b) Other dissemination

Similä T: *Proper fire engineering is more important than materials.* UPM Magazine Griffin Wood, 2004.

InnoFireWood. Input to yearbooks 2004-2007 of the Wood Material Science Research Programme.

InnoFireWood – Innovative eco-efficient high fire performance wood products for demanding applications. Poster at the Annual Seminar for the Wood Material Science Finnish-Swedish Research programme, Helsinki 2004 and Stockholm, 2005

The InnoFireWood project. Presentations by SP Trätek, KTH and VTT. Workshop at UPM R&D Centre in Lahti, Finland, 22 June 2005

Östman B: *Eco-efficient wood products offering high-fire performance.* Wood Material Science and Engineering Research Programme Annual Seminar, Helsinki April 2006

Durability of Reaction to Fire Performance of fire-retardant treated wood products in different end use conditions. Nordtest Method NT FIRE 054, 2006

Östman B: *Brandskyddat trä som håller.* Miljöforskning, juni 2006

Eco-efficient wood products offering high-fire performance. Leaflet on InnoFireWood project results, 2007.

14.6 National and international cooperation

The steering group for the InnoFireWood project has had the following members.

Keijo Kolu, chairman, UPM Kymmene
Jouni Hakkarainen, Finnforest
Bernt Hoffrén, Interenergy PressoCenter
Johanna Kairi, Stora Enso Timber
Pekka Nurro, Woodfocus
Corné van Hamont, Wacker-Chemie
Ulrik Lindgren, Ingarps Träskydd
Bernt-Åke Sultan, Borealis
Juha Vaajoensuu, adj., Tekes
Bengt Larsson, adj., VINNOVA

The steering group has had six meetings, alternating in Finland and Sweden.

The InnoFireWood project has close connection with the Nordtest project *Service classes for fire retardant wood products*, which has been running 2005-06. The project partners have partly been the same and the Nordtest results have been implemented in the InnoFireWood project. The goal of the Nordtest project was to establish a Nordic standard for requirements based on earlier test methodologies. It has resulted in a new Nordtest method with classes for the Durability of Reaction to Fire (DRF) performance. The new Nordtest method will be transformed to a European EN method.

The project has kept close cooperation with the European network, *FireRetard.com*, which originates in an earlier European Thematic Network *High fire performance wood products*, HIFI coordinated by VTT and with active participation from Trätek. The network includes industrial and research partners from several countries with experience or interest in fire retardant wood products.

The project has also close cooperation with a quite new European network, *Fire Safe Use of Wood*, FSUW, initiated by Finland and Sweden and with participants from at least eight European countries. The partners are from industry, research and building and fire authorities. The FSUW network has a wide scope and several objectives, e g

- New knowledge will lead to wider and more similar acceptance of wood building products in different countries which means less barriers to trade in Europe
- Increased use of fire safe wood products will provide new opportunities to increased use of products from renewable resources that will stabilize the CO_2 balance.

Further information on the network is available at www.fsuw.com.

15 Innovative design, a new strength paradigm for joints, QA and reliability for long-span wood construction (InnoLongSpan)

FINAL REPORT

Name of the research project	**Innovative design, a new strength paradigm for joints, QA and reliability for long-span wood construction (InnoLongSpan)**
Coordinator of the project	Dr. Antti Hanhijärvi, VTT

BASIC SUB-PROJECT DATA

Name of the sub-project 1	**Performance of high capacity dowel type joints – Effects of long-term loading and moisture**
Project period	1.1.2004–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Dr. Antti Hanhijärvi
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O.Box 1000, FI-02044 VTT Tel. 020-722 5980 Fax 020-722 7007 Antti.Hanhijärvi@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	260 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	156 000
Other funding	
Industry	52 000
VTT	52 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Antti Hanhijärvi, Senior Research Scientist	M	VTT		
Ari Kevarinmäki, Senior Research Scientist	M	VTT		

Alpo Ranta-Maunus, Research Professor	M	VTT
Rainer Yli-Koski, Research Scientist	M	VTT

Total person-months of work conducted by the research team 15
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 2	Ductility and influence of moisture variations on high capacity dowel joints
Project period	1.1.2003–31.3.2007
Organization in charge of research	Växjö University
Sub-project leader	Hans Petersson
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	School of Tecnology and Design Växjö University SE-35195 Växjö, Sweden Tel. ++46 470 70 81 76 hans.petersson@vxu.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	88 800
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	42 600
Other funding	
Industry	46 200

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Johan Sjödin	M	Skanska Teknik Växjö University	Växjö University	
Hans Petersson	M	Växjö University	Växjö University	
Erik Serrano	M	Växjö University	Växjö University	

Total person-months of work conducted by the research team 10
person-month = full-time work for at least 36 h/week, paid holidays included

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	Licentiate in engineereing	M	Johan Sjödin, 1977 M.Sc.	Växjö University	

Name of the sub-project 3	Drilling type dowel joints
Project period	1.1.2004–31.3.2007
Organization in charge of research	Div. of Structural Mechanics, Lund University
Sub-project leader	Erik Serrano (Initially Per Johan Gustafsson)
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Växjö University School of Technology and Design SE-351 95 VÄXJÖ, Sweden Tel. +46 470 70 89 90 Mobile +46 703 15 52 63 erik.serrano@vxu.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	47 500
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	22 800
Other funding	
Industry	24 700

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Erik Serrano, Ph.D., Professor	M	Växjö University / Lund University Växjö University / Lund University		See "Funding"
Per Johan Gustafsson, Ph.D., Professor	M	Lund University, Lund University		See "Funding"

Total person-months of work conducted by the research team 3
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 4	High capacity rubber type joints
Project period	1.1.2004–31.3.2007
Organization in charge of research	Div. of Structural Mechanics, Lund University
Sub-project leader	Per Johan Gustafsson
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Structural Mechanics, LTH, Lund University Box 118 ,221 00 Lund, Sweden Tel. +46 46 222 49 22 Per_J.Gustafsson@byggmek.lth.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	244 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	107 300
Other funding	
Industry	116 400

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Per Johan Gustafsson, Ph.D., Professor	M	Lund University, Lund University		See "Funding"
Magnus Wikström, M.Sc., Research Engineer	M	Casco Products AB, Royal Institute of Technology, Stockholm		See "Funding"
Total person-months of work conducted by the research team 20 person-month = full-time work for at least 36 h/week, paid holidays included				

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2007	M.Sc.	M	Magnus Wikström, 1982, 2007, M.Sc.	Royal Institute of Technology, Stochkolm	Peter Herder, Casco Products AB
2006	M.Sc.	M	Peter Björnsson, 1980, 2006, M.Sc.	Lund University	Per Johan Gustafsson, Lund University
2006	M.Sc.	M	Henrik Danielsson, 1980, 2006, M.Sc.	Lund University	Per Johan Gustafsson, Lund University

Name of the sub-project 5	**Reliability and competence in timber construction**
Project period	1.1.2003–31.3.2007
Organization in charge of research	Lund University
Sub-project leader	Sven Thelandersson
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Div. of Structural Engineering Lund University Box 118, SE-22100 Lund, Sweden Tel. +46 46 2227000 Fax +46 46 222 4212, Sven.Thelandersson@kstr.lth.se
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	111 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
VINNOVA	53 000
Other funding	
Industry	58 000

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sven Thelandersson, Prof.	M	Lund University	VINNOVA, Skogsindustrierna	
Eva Fruhwald, M.Sc.	F	Lund University	VINNOVA, Skogsindustrierna	
Arne Emilsson, M.Sc.	M	Limträteknik AB	Limträteknik AB	
Erik Serrano, Ph.D.	M	Swedish Testing and Research Institute, SP	VINNOVA, Skogsindustrierna	

Total person-months of work conducted by the research team 16
person-month = full-time work for at least 36 h/week, paid holidays included

Name of the sub-project 6

Project period	1.1.2004–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Prof. Alpo Ranta-Maunus
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT, P.O.Box 1000, FI-02044 VTT Tel. +358 20 722 5536 Fax +358 20 722 7007 Alpo.Ranta-Maunus@vtt.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING

Total sub-project budget in EUR	94 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	56 400
Other funding	
Industry	38 000
VTT	9 600

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Tomi Toratti, Senior Research Scientist	M	VTT		
Ari Kevarinmäki, Senior Research Scientist	M	VTT		
Alpo Ranta-Maunus, Research Professor	M	VTT		
Total person-months of work conducted by the research team 7 person-month = full-time work for at least 36 h/week, paid holidays included				

Abstract

The objective of the project was to enhance the use of timber in long-span structures.

Based on some 150 tension tests of large dowelled joints, a new design method for the timber failure mechanisms in dowelled joints was developed. The new method is based on a new concept of failure criterion. It clarifies the design and shows more accurate correspondence to experimental data than before.

The effect of moisture changes on the load-bearing capacity of large dowelled joints was investigated experimentally and with numerical simulations. The results showed that the load-bearing capacity is reduced by decreasing moisture. Highest reduction was found in joints initially exposed to restrained shrinkage deformations in the joint area.

As a new type of joint, rubber foil adhesive joining technology was shown to give the possibility for lap joints with extremely high load bearing capacity. They performed well as medium size and large joints. They are also of particular interest in situations with impact loading or enforced deformations e.g. due to moisture changes and showed good characteristics when applied to joining of both wood-to-wood and wood-to-steel parts.

As a special non-traditional joint type, a special self-drilling type of dowel joint was evaluated by means of finite element analyses (FEA). The numerical approach showed the capability of capturing the fundamental behaviour of the joint in terms of maximum load bearing capacity and deformation mode of the dowels, which allows to optimize the joint parameters by numerical simulation.

A survey of failures in buildings with timber as a primary structural material was made including a total of 127 failure cases. In total, about half of the failures are related to building design and about one fourth of the failures are caused at the building site (on-site alterations, poor principles during erection). This means that wood quality, production methods and principles only cause a small part (together about 11%) of the failures. Thus, vast majority of failures occur due to human errors,which can not be counteracted by increased safety factors or safety levels in structural codes.

As part of the development of quality assurance procedures, quality requirements were set for the design and construction of timber buildings, so that sufficient reliability, durability and overall usefulness of the building are ensured. These results are particularly meant for the design, construction, use and maintenance of long span or otherwise demanding timber structures and joints. However, the procedures developed may be used also for other structures.

Tiivistelmä

Projektin tavoitteena oli edistää puun käyttömahdollisuuksia suuren jännevälin rakenteissa.

Projektissa tehtiin noin 150 vetokoetta suurilla tappivaarnaliitoksilla. Näiden kokeiden perusteella luotiin uusi mitoitusmenetelmä tappavaarnaliitosten puustamurron mukaisen lujuuden mitoitukseen. Uusi menetelmä perustuu uudentyyppiseen

lujuuskriteeriin. Se selkeyttää mitoitusta ja on entistä tarkempi verrattaessa koetuloksiin.

Kosteusvaihteluiden vaikutusta suurten tappivaarnaliitosten lujuuteen tutkittiin sekä kokeellisesti että laskennallisesti simuloimalla. Tulokset osoittivat, että aleneva kosteus aiheuttaa lujuuden pienenemisen. Suurin pieneneminen havaittiin tapauksessa, jossa alun perin kosteana valmistettu liitos kuivui ja näin aiheutui estetystä kutistumisesta johtuvia jännityksiä liitosalueelle.

Uutena liitostyyppinä tutkittiin kumikalvoliimaliitosta. Osoittautui, että tämän teknologian avulla on mahdollista saavuttaa erittäin korkeita lujuuksia jatkosliitoksissa. Liitos toimi hyvin keskikokoisissa ja suurissa liitoksissa sekä tilanteissa, joissa liitos joutuu iskukuormituksen alaiseksi tai pakotettujen muodonmuutosten alaiseksi, esim. kosteusvaihtelun takia. Liitos osoittautui ominaisuuksiltaan hyväksi sovellettaessa sekä puu-puu että puu-teräs liitoksiin.

Erikoisliitoksena tutkittiin laskennallisesti elementtimenetelmällä myös itseporautuviin tappivaarnoihin perustuvaa liitosta. Laskennallinen menetelmä kykeni kuvaamaan liitoksen käyttäytymisen, eli maksimilujuuden ja tappien muodonmuutokset, mikä mahdollistaa liitosparametrien optimoimisen laskennallisesti.

Projektin osana tehtiin myös pääasiallisesti puurakenteisten rakennusten vauriotapausten tutkimus. Se käsitti 127 vauriotapausta. Noin puolet vaurioista johtui rakennesuunnittelun virheistä ja noin neljännes toteutuksen virheistä (paikalla tehdyt muutokset, huonot rakennuskäytännöt). Puu materiaalin laatu, tuotantomenetelmät ja -tavat aiheuttivat vain pienen osan (n. 11%) vaurioista. Tämä osoittaa, että suurin osa vaurioista johtuu inhimillisistä virheistä, eikä niistä johtuvia vaurioita voida ehkäistä kasvattamalla varmuusmarginaalia tai suurentaen varmuuskertoimia.

Projektissa kehitettiin laadunvarmistusohje ja menettelytapoja laadun varmistamiseksi puurakenteita sisältävissä vaativissa rakennuskohteissa. Ohje sisältää laatuvaatimuksia rakennuttamiselle, suunnittelulle, rakentamiselle ja itse lopputuotteelle. Ohje koskee erityisesti AA-vaativuusluokan (RakMK A2) puurakenteiden suunnittelua, valmistusta, asennusta, käyttöönottoa ja huoltoa. Tuloksia suositellaan käytettäväksi soveltuvin osin myös muissa vaativuusluokkissa.

Sammanfattning

Målet med projektet var att öka konkurrensförmågan för trä som material i konstruktioner med stora spännvidder.

I projektet utfördes ca 150 dragprovningar med stora dymlingsförband. Med utgångspunkt från de experimentella observationerna av brottlaster och brottförlopp utvecklades en ny metod för dimensionering och beräkning av bärförmåga. Metoden baseras på en beskrivning av möjliga träbrottförlopp i dymlingsförband och på en ny typ av hållfasthetskriterium. Den nya metoden gör dimensioneringen lättare att förstå och ger bättre överenstämmelse med provningsresultaten.

Fuktvariationers inverkan på hållfastheten i de stora dymlingsförbanden undersöktes både experimentellt och genom numeriska simuleringar. Resultaten visade att minskad fuktkvot ger minskad bärförmåga. Den största effekten konstaterades vid förhindrad krympning åstadkommen genom att tillverka förbandet vid hög fuktkvot och sedan utsätta det för uttorkning före provning.

En ny typ av förband, gummifolielimförband, utvecklades och undersöktes. Experimentella provningar visade att denna nya teknologi gör det möjligt att uppnå synnerligen hög bärförmåga hos limmade överlappförband. Den goda bärförmågan är framträdande särskilt för stora förband. Två andra mycket intressanta tillämpningar är förband utsatta för stötbelastning och förband utsatta för påtvingade deformationer orsakade av t.ex. fuktvariationer. Gummifolielimförband studerades experimentellt och med goda resultat både för fallet förbindning trä-till-trä och för fallet förbindning trä-till-stål.

Som specialförband studerades även förband med självborrande ståldymlingar. Dessa studier gjordes som numeriska simuleringar med finita elementmetoden. Simuleringarna gav information om spänningar och deformationer vid olika last, och om förbandens styvhet och bärförmåga, vilket möjliggör numerisk optimering av förbandsparametrar.

Som en del av projektet gjordes också en skadefallsundersökning huvudsakligen i byggnader av träkonstruktioner. Undersökningen omfattades av 127 skadefall. Cirka hälften av skadorna berodde på fel i projektering och cirka en fjärdedel på fel i utförande (ändringar gjorda på platsen, dålig bygg-

nadspraxis). Trämaterialets kvalitet förorsakar endast en liten del (ca 11 %) av skadorna. Detta visar att den största delen av skadorna beror på mänskliga fel, som inte kan förebyggas genom ökade säkerhetsnivåer eller höjda säkerhetsfaktorer i gällande konstruktionsnormer.

I projektet utvecklades anvisningar för kvalitetssäkring och metoder att försäkra kvaliteten för träkonstruktioner i byggnader. Anvisningen innehåller kvalitetskrav för byggherre, projektörer, byggare och själva slutprodukten. Anvisningarna gäller speciellt för planering, tillverkning, montering, ibruktagande och underhåll av träkonstruktioner enligt AA-kompetensklassificeringen (RakMK A2). Resultaten rekommenderas för användning i tillämpliga delar också i andra typer av konstruktioner.

15.1 Introduction

15.1.1 Background

Recent failures of long-span roof structures made of timber (Jyväskylä exhibition centre, Ballerup sports arena) have threatened the competitiveness of timber especially in large public buildings.. These failures were mainly caused by gross errors in design and/or production techniques, and a widespread opinion among building professionals is that wood is more risky to use than the competing materials. After this project started in 2003, a number of new cases have occured where roof structures have collapsed, especially in Central Europe during the winter 2005-2006. Some collapses drew a lot of public attention. These events do emphasize the importance for failure evaluation and that the observations from failure causes are transmitted to adequate quality assurance measures.

The performance and competitiveness of timber in large (= long span) structures is to a great extent dependent on the potential to join timber members together with reliable heavy-duty joints. Sufficiently high capacities can be achieved by using the so-called dowel-type fasteners (dowels and bolts). The present design practice of these joints is largely based on a rather old theory that assumes plastic behaviour of timber at fastener–timber interface. However, in joints involving perpendicular-to-grain tension and shear stresses, the failure is often brittle and not explicable with plasticity. In large and high capacity joints, the relative importance of perpendicular-to-grain tension and shear stresses increases. Use of an ill-fitting theory as a design criterion may lead to loss in competitiveness because of inaccuracy.

Due to the importance of joints in the large timber structures, the development of improved design methods as well as new joint types with high capacity can be very advantegeous for the use of timber.

15.1.2 Objectives

The objective of the project is to enhance the competitiveness of timber in long-span structures by improving the design methods used for the joints and development of completely new joints. Furthermore, the competitiveness is ensured by increasing confidence in timber as a building material among professionals in the construction sector by documenting reliability and developing quality assurances (QA) procedures.

15.2 Results and discussion

15.2.1 Traditional dowel type joints in large timber structures

Effect of timber failure mechanisms on capacity

Alltogether more than 150 tension tests of heavy-duty dowelled joints were made with glulam and Kerto-LVL specimens. The experimental program contained tests of both double-shear and multiple-shear-plane joints.

A surprising feature observed is that in large proportion the failure mode was different than what the design calculation is currently based on (EN1995-1-1:2004). Viz., in a large part of the outer timber members, the design capacity is based on the plug-shear failure mechanisms, whereas the observed failure was block shear. In fact, in no cases was plug shear observed. For the cross-laminated Kerto-Q LVL the plug shear design value is in some cases very low, due to the low shear strength of the cross-veneers (rolling shear). How-

ever, in no cases was the plug shear observed for the Kerto-Q specimens, either. The plug shear failure does not occur, because the dowels remain straight or bend very little before failure and failure occurs as block shear.

Based on the large amount of experimental results, a new method for designing dowel type steel-to-timber joints against timber failure mechanisms was developed (Hanhijärvi and Kevarinmäki 2007a,b).

Effect of moisture variations on capacity

Experimental tests were also carried out concerning the influence of initial moisture-induced stresses in the joint area in dowel-type joints loaded in tension parallel to the grain. The moisture induced stresses were caused by shrinkage deformations that were restrained perpendicular to the grain by the fasteners. In small-scale joints, no major influence on the load-bearing capacity was found. Contact-free measurements and supplementary numerical results (Sjödin 2006) provided an explanation to this by showing the moisture-induced stresses to be very local around the dowels.

In contrast, a significant decrease of the load-bearing capacity was detected for the large-scale joints (Sjödin and Johansson 2007). The configuration of the joints strongly affected the influence of moisture. An increase in spacing between the dowel rows perpendicular to the grain led to a lower load-bearing capacity and to an increased occurrence of moisture-induced cracks.

The effect of moisture gradients on dowel-type joints was also numerically analysed based on experimentally observed results, (Sjödin et Serrano 2006, 2007).

15.2.2 Novel type joints

Rubber type joints

Rubber type joints of both timber-to-timber and wood-to-steel type were investigated (Wikström 2006, 2007, Gustafsson 2007). In the case of timber-to-steel the steel parts were vulcanized with a thin rubber layer, which were then glued to the wooden part. It was found that several different qualities of rubber and glue would be useful. Pretreatment of the rubber surfaces by rinsing with a strong acid is required for good adhesion.

Test of six types of lap joints glulam-glulam gave good results in the sense that failure in the majority of the tests developed in the glulam away from the bond line. For a joint in simple shear along grain, a strength of 4.4 MPa was found, governed by the shear failure in the wood. This high strength indicated that the entire bond area was active in carrying the load. Corresponding deformation is elastic and of the same order as the (plastic) deformation of a nailed joint. The tests of three types of glued and nailed joints LVL-glulam gave similar and good results, as well as tests of four types of lap-joints glulam-to-steel plates.

Tests of steel rods vulcanized with a thin rubber layer and glued into holes drilled in glulam along the grain showed less strength than expected. Failure developed within the rubber or in the rubber to steel interface. This might perhaps be due to the manufacture difficulties in vulcanization of rods with a very thin rubber layer.

Work on theoretical modeling of lap joints gave a computer code for general 3D lap joints and simple equations for 2D lap joints with a flexible bond layer (Gustafsson 2006, 2007).

Selfdrilling type joints

The selfdrilling type joints were investigated by numerical simulations. To restrict the number of necessary material parameters, the following main features were kept: orthotropic linear elastic material, orthotropic plastic behaviour, different tensile and compressive yield strengths. Due to the application (joints), the material model for wood was also restricted so that it describes in a reasonable way the behaviour in compression, but a linear elastic behaviour is assumed up to the point of failure in tension. For crack formation and propagation analysis, the current material model must be supplemented with a fracture mechanics model. (Serrano 2007)

The nonlinearity of the joints is caused by the plastic response of the dowels and the plastic response of the wood in compression along the grain. The latter is highly localised. Striving for a simpler modelling approach, as opposed to the use of the above mentioned plasticity model, a nonlinear

contact algorithm was tested. The contact algorithm defines a nonlinear contact-pressure versus overclosure relation, which was defined such as to simulate the elasto-plastic response of the wood in compression parallel to the grain.

Using the simplified approach with the nonlinear contact algorithm, results were obtained in terms of load bearing capacity, which were close to the values obtained in earlier tests. The stiffness of the joints was overestimated, however. The plasticity model gave results close to the ones obtained with the simplified approach. A parameter study showed that the load bearing capacity per dowel decreases slightly with the number of dowels in a row. This decrease was small, however, which is contributed to the highly ductile nature of the joint.

15.2.3 Failure survey

The concept of failure was considered mainly as related to the ultimate limit state (collapse) and is defined as events which had or could have implied risk for human lives. A total of 127 failure cases were included in the survey. The case reports were analysed and causes behind the failure event were classified into nine categories. In total, about half of the failures are related to errors in design. About one fourth of the failures are caused at the building site (on-site alterations, poor principles during erection). Wood quality, production methods and principles only cause a small part (together about 11%) of the failures. The problem is therefore not the wood material, but the actions of engineers and workers in the building process. (Frühwald et al. 2007)

Among the studied cases, instability is a very dominant failure mode. This means that the collapse or failure was caused by insufficient or completely lacking bracing, which led to buckling or material failure. Bending failures and tension perpendicular to grain failures are also common.

15.2.4 Quality assurance of challenging timber structures

As part of this project a quality assurance (QA) guide (Anon. 2006) for challenging timber structures was created. The new guide book is intended for professionals in the construction industry, and it contains comprehensive, detailed information and tools for management of challenging construction projects with a high standard of quality. The guide describes the construction project, starting from the competitive bidding and ending in commissioning and maintenance. The responsibilities of the parties involved, the documentation of all plans, decisions and other important information are covered. The guide also provides checklists for the designers, managers and other key persons.

15.3 Conclusions

15.3.1 Joints in large scale timber structures

Traditional dowel type joints

Based on the large experimental data the following conclusion can be made:

- In large portion of the tested joints, the plug shear failure mechanism did not occur in the joint area contrarily to what the design equations in EC5 (EN 1995-1-1:2004; Annex A) suggest. The new results give good grounds for improving the design.
- The reduction effect of the number of dowels in a row is too conservative for these joints, because it does not take into account the slenderness of the dowels.
- Initial moisture changes reduced the load-bearing capacity of steel-to-timber dowel joints. The size of the joint and the joint configuration set-up affected the moisture influence. Moisture gradients strongly affect large dowel-type joints.
- Highest decrease in the load-bearing capacity was found for the joints where the steel dowels restrained the moisture movements

The new design method developed for the design of dowel type joints against timber failure mechanisms shows clear advantages:

- clarity compared to previous methods
- more accurate according to the experimental verification
- the separation of different failure modes makes the design transparent and combination of other mechanisms easier.

Novel type joints

Rubber foil adhesive joining gives a possibility for lap joints with extremely high load bearing capacity. They are of particular interest for medium size and large joints. and in situations with impact loading or enforced deformations e.g. due to moisture changes. They can be applied to joining of wood to wood and wood to steel parts.

The numerical analyses performed for the self-drilling type joints showed that it is a demanding challenge to fully model the response of this type of joint. However, since the joints are very ductile, showing a response characterised by a plateau value reached after initial yielding, it should suffice to model the initial yielding of the joint, thus capturing the load bearing capacity. The parameter studies showed the influence of the friction between the dowel and the wood to be an important factor. The influence of increasing the number of dowels in a row was moderate. As regards the design of the joint type analysed, both the experimental tests and the numerical analyses support the fact that, for many cases, the choice of a design approach based on plastic behaviour of the joint is appropriate. To be able to use such a criterion, the dowel and timber geometry must be balanced such that the plastic hinges in the dowel can develop.

15.3.2 Reliability and QA in large timber structures

The failure survey confirms the conclusion that for structures of all types of materials, the vast majority of failures occur due to human errors. This is in line with results of similar studies made by other researchers. Failures due to human errors can not be counteracted by increased safety factors or safety levels in structural codes. As also found in many other investigations, almost no failures were caused by unfavourable combinations of random events. Thus, there is no evidence from the present investigation that the chosen safety level for timber in structural codes would be inadequate.

It may not be possible to eliminate the risk of human errors completely but their frequency can be reduced by improving building process management, where an important element is to assign or commission personnel with adequate experience and education as well as with the right attitude to the tasks at hand. Training, education and control measures should be especially focussed on those technical aspects found to be the most common causes of failures. The developed quality assurance guidelines give the necessary tools for the practitioning engineers to ensure that all necessary aspects are taken into account during the construction project and a good, safe and high quality result is achieved.

15.4a Capabilities generated by the project

- New design method for the timber failure mechanisms in dowelled high capacity joints. The new method improves the design by making it clearer and more accurate, enabling better use of timber, especially in large structures.
- New, important knowledge about moisture effects in joints of large scale timber structures. The new knowledge provides basis for considering the effect of moisture changes to the load carrying capacity of dowelled joints.
- A patent on the rubber foil adhesive joints has been approved in several countries including the USA. The patent is hold by Casco Products AB.
- Improved knowledge about and new tools for the application of numerical analysis on mechanical behaviour of dowel type joints. The analyses enable the improvement of joint patterns and enhance the development of new types of joints.
- In-depth knowledge about the causes and frequency of failures of timber structures. This information provides more rational bases for safety issues concerning timber structures. Furthermore the knowledge is applicable in many kinds of risk analyses and also gives input for development programs on the national and international level.
- Quality assurance procedures readily applicable to challenging timber constructions projects.

15.4b Utilisation of results

- The new design method is proposed to the European design code (EC5).
- Design recommendations for taking into account the influence of moisture changes on dowel-type joints.
- The numerical analysis (FE) tools are used e.g. to evaluate the use of design formulae based on plastic behaviour of the joint (slender dowels).
- Discussions on possible practical/commercial implementations of the rubber-type adhesive joint are on-going with a manufacturer of fastener devices, glulam industry, building companies and a timber structural design company.
- The knowledge of structural failures will be used in education and training of structural engineers (courses on the national and European levels).
- The QA guidelines given in the guide book have already drawn much positive attention and have been adopted by many organizations. For example the city of Helsinki has announced that it will use the guide in its projects.

15.5 Publications and communication

a) Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice,

* Sjödin, J and Johansson, C. J. "Influence of moisture induced stresses in multiple steel-to-timber dowel joints". Holz als Roh- und Werkstoff, Vol 65, pp 71-77, 2007.

Sjödin, J., Serrano, E., A numerical study of the effects of stresses induced by moisture gradients in steel-to-timber dowel joints. Holzforschung, Vol 60, pp 694-697, 2006.

Sjödin, J and Serrano, E. "An experimental study of the effects of moisture induced stresses in steel-timber dowel joints". Submitted to Holzforschung, March 2007.

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice

Hanhijärvi A, Kevarinmäki A and Yli-Koski R. (2006). "Block shear failure at dowelled steel-to-timber connections". Proc. CIB-W18 Meeting, Florence Aug, 28-31, 2006.

* Hanhijärvi A, Kevarinmäki A (2007b). "Design of dowelled steel-to-timber connections with timber failure mechanisms". Proc. CIB-W18 Meeting, Bled Aug, 2007.

Sjödin, J., Johansson, C. J. and Petersson, H. (2004) "Influence of moisture induced stresses in steel-to-timber dowel joints". Proceedings of the 8th World Conference on Timber Engineering, Vol. 1, Lahti, Finland.

Sjödin, (2006) "Moisture induced stresses in multiple dowel-type joints loaded parallel to the grain". Proceedings of the 9th World Conference on Timber Engineering, Portland, USA.

Sjödin, J. "An experimental study on methods to increase the ductility of steel-timber dowel joints loaded parallel to the grain" Paper in preparation to the World Conference on Timber Engineering, Japan, 2008.*

3. Articles in Finnish and Swedish journals with referee practice

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice,

5. Scientific monographs,

* Frühwald, E., Serrano, E., Toratti, T., Emilsson, A., Thelandersson, S. (2007). Design of safe timber structures – How can we learn from structural failures in concrete, steel and timber? Report TVBK-3053. Div. of Struct. Eng., Lund University. 270 p.

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Hanhijärvi, A, Kevarinmäki, A (2007a). "Timber failure mechanisms of large dowel type connections". Draft VTT Publications Report

Sjödin, J (2006) "Steel-to-timber dowel joints - Influence of moisture induced stresses" Licentiate thesis, Växjö University, Växjö, Sweden.*

Clase, M. and Nilsson, P., (2006) "Improvement of the Ductility in Doweled Steel-to-Timber Joints", Master´s Thesis, 2006:2, Chalmers University of Technology, Göteborg, Sweden, 2006.

Björnsson, Peter and Danielsson, Henrik, 2006: "Strength and Creep Analysis of Glued Rubber Foil Timber Joints", Master's thesis, Division of Structuiral Mechanics, Lund University

Wikström, Magnus, 2007: Testing of glueability and mechanical properties various wood/glue/rubber bonding-systems, Draft Master thesis, KTH and Casco Products AB

Wikström, Magnus, 2006: "High Capacity Rubber Type Type Joints - Manufacturing of Full Scale Joints", Report, Casco Products AB, Stockholm

Gustafsson, PJ, 2006: "A structural joint and support finite element" , Report TVSM 7143, Division of Structural Mechanics, Lund University

* Gustafsson, PJ, 2007: "Tests of full size rubber foil adhesive joints", Draft report, Division of Structural Mechanics, Lund University

Gustafsson, PJ, 2007: "Stress equations for 2D lap joints with a flexible bond layer", Draft report, Division of Structural Mechanics, Lund University

Serrano, E. Timber joints based on self-drilling dowels – Numerical analyses of nonlinear behaviour. Report TVSM 7146, Division of Structural Mechanics, Lund University, Sweden, 2007 (In press),

b) Other dissemination

Such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results.

* Anon. 2006. RIL 240-2006: "Puurakenteiden laadunvarmistus – Suunnittelu, valmistus työmaatoteutus käyttö" ("Quality assurance of timber structures, design, manufacture, construction and use") (In Finnish, this publication was also translated into English but not published yet).

A number of lectures have been given from the project mainly targeted at practicing engineers, but also for researchers and students.

15.6 National and international cooperation

Advisory board (Steering committee)

Jan Lagerström, Skogsindustrierna, Chairman
Ilmari Absetz, Tekes
Eva Esping, VINNOVA
Bengt Larsson, VINNOVA
Tapani Tuominen, SPU-Systems Oy
Veijo Lehtonen, LATE-Rakenteet Oy
Jukka Juselius, Exel Oyj
Jouni Hakkarainen, Metsäliitto
Unto Hyytiä, Versowood Oy
Jan-Inge Bengtsson, SFS-Intec AB, Fastening Systems
Arne Emilsson, Limträteknik i Falun AB
Johan Fröbel, Svenskt Limträ AB
Peter Herder, Casco Products AB
Sigurd Karlsson, Skanska Teknik AB
Per Johan Gustafsson, Lund University/ Division of Structural Mechanics
Sven Thelandersson, Lund University/ Division of Structural Engineering
Hans Petersson, Växjö University/School of Technology and Design
Erik Serrano, SP Swedish National Testing and Research Institute
Heikki Kukko, VTT
Antti Hanhijärvi, VTT

Direct co-operation in the development of rubber-type adhesive joints with:
Magnus Wikström and *Peter Herder,* Casco Products AB
Roberto Crocetti, Moelven Töreboda AB and Svenskt Limträ AB
Jan-Inge Bengtsson, SFS-Intec AB
Per-Olof Rosenkvist, Thord Lundgren and Per-Erik Austrell, Lund University
Sture Persson, Metso Minerals AB
Arne Emilson, Limträteknik AB
Anders Clang, SP

Discussion with colleagues in the international research community, who also have delivered reports from failure cases included in the study.

National advisory board for the development of the QA-guide of challenging timber structure (Task C2, 15 meetings during two years):

Mikko Mäkinen, SPU SystemsOy
Antero Jarvenpää, Late-Rakenteet Oy
Unto Hyytia, Versowood Oy
Jouni Hakkarainen, Finnforest Oyj
Juha Elomaa, Ramboll Oy
Gunnar Åstrom, RIL
Veijo Lehtonen, Late-Rakenteet Oy
Alpo Ranta-Maunus, VTT
Ari Kevarinmäki, VTT
Tomi Toratti, VTT

16 Multi-sectorial database, model system and case studies, supporting innovative use of wood and fibers (Innovood)

FINAL REPORT	
Name of research project	**Multi-sectorial database, model system and case studies, supporting innovative use of wood and fibres (Innovood)**
Coordinator of the project	Sven-Olof Lundqvist

BASIC PROJECT DATA	
Name of the sub-project 1	**Common practices, database and dissemination of results**
Project period	1.4.2004–31.3.2007
Organization in charge of research	STFI-Packforsk AB
Sub-project leader	Sven-Olof Lundqvist
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	STFI-Packforsk Box 5604, S-114 86 Stockholm, Sweden Phone +46 8 676 71 51 Mobile +46 768 76 71 51 Fax +46 8 411 55 18 svenolof.lundqvist@stfi.se
URL of the project	http://stfi-packforsk.se http://www.woodwisdom.fi/en/
Name of the sub-project 2	**Integrated model system for properties of stems**
Project period	1.6.2004–31.3.2007
Organization in charge of research	University of Helsinki
Sub-project leader	Annikki Mäkelä
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Department of Forest Ecology P.O.Box 27, 00014 University of Helsinki Finland Phone +358 9 19158108 Fax +358 9 19158100 annikki.makela@helsinki.fi
URL of the project	http://www.mm.helsinki.fi http://www.woodwisdom.fi/en/

Name of the sub-project 3	**Data and models for wood and fiber properties**
Project period	1.7.2004–31.3.2007
Organization in charge of research	STFI-Packforsk AB
Sub-project leader	Sven-Olof Lundqvist
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URL of the project	http://stfi-packforsk.se http://www.woodwisdom.fi/en/

Name of the sub-project 4	**Data and models for chemical composition of wood**
Project period	1.4.2004–30.9.2006
Organization in charge of research	Södra Cell
Sub-project leader	Dag Molteberg
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Södra Cell AB Research and Development SE-43024 Väröbacka, Sweden Phone +46 340 62 80 00 Mobile +47 90 57 19 71 Fax +46 340 62 81 87 dag.molteberg@sodra.com
URL of the project	http://www.sodra.com http://www.woodwisdom.fi/en/

Name of the sub-project 5	**Advanced solid wood products and manufacturing processess**
Project period	1.4.2004–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Arto Usenius
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT Visiting address: Lämpömiehenkuja 2, Espoo P.O.Box 1000, FI-02044 VTT, Finland Phone +358 20 722 5540 Mobile +358 40 504 43 44 Fax +358 20 722 6251 Arto.Usenius@vtt.fi
URL of the project	http://www.vtt.fi http://www.woodwisdom.fi/en/

Name of the sub-project 6	Case study 1 **Wood-based composite flooring**
Project period	1.4.2004–31.1.2007
Organization in charge of research	Pergo (Europe) AB
Sub-project leader	Tomas Stjernberg
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Pergo (Europe) AB Box 1010, SE-231 25 Trelleborg, Sweden Phone +46 410 363 100 Mobile +46 70 570 32 98 Fax +46 410 455 29 tomas.stjernberg@pergo.com
URL of the project	http://www.pergo.com http://www.woodwisdom.fi/en/
Name of the sub-project 7	Case study 2 **Customer designed fibres**
Project period	1.6.2004–31.8.2004; 1.10.2005–31.1.2007
Organization in charge of research	Södra Cell AB
Sub-project leader	Dag Molteberg
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Södra Cell AB Research and Development SE-43024 Väröbacka, Sweden Phone +46 340 62 80 00 Mobile +47 90 57 19 71 Fax +46 340 62 81 87 dag.molteberg@sodra.com
URL of the project	http:// www.sodra.com http://www.woodwisdom.fi/en/
Name of the sub-project 8	Case study 3 **Selection of suitable raw material for customer-specified products**
Project period	1.3.2005–28.2.2007
Organization in charge of research	University of Helsinki
Sub-project leader	Annikki Mäkelä
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Department of Forest Ecology P.O.Box 27, 00014 University of Helsinki Finland Phone +358 9 19158108 Fax +358 9 19158100 annikki.makela@helsinki.fi
URL of the project	http:// www.mm.helsinki.fi http://www.woodwisdom.fi/en/

Name of the sub-project 9	Case study 4 **Profitable secondary conversion concepts for end user mill**
Project period	1.6.2005–31.3.2007
Organization in charge of research	VTT
Sub-project leader	Arto Usenius
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	VTT Visiting address: Lämpömiehenkuja 2, Espoo P.O.Box 1000, FI-02044 VTT, Finland Phone +358 20 722 5540 Mobile +358 40 504 43 44 Fax +358 20 722 6251 Arto.Usenius@vtt.fi
URL of the project	http://www.vtt.fi http://www.woodwisdom.fi/en/
Name of the sub-project 10	**Modelling and microscopy**
Project period	1.4.2004–30.9.2006
Organization in charge of research	Metla
Sub-project leader	Pekka Saranpää
Contact information of sub-project leader (institute/unit, address, telephone, fax, e-mail)	Finnish Forest Research Institute (Metla) Vantaa Research Centre Pl 18 (Jokiniemenkuja 1) FIN-01301 Vantaa, Finland Phone +358 10 211 2340 Mobile +358 10 211 2203 Fax +358 01 0211 2203 Pekka.Saranpää@metla.fi
URL of the project	http://www.metla.fi http://www.woodwisdom.fi/en/

FUNDING

(It has been agreed that the budget for Innovood -project will only be specified for the project as a whole)

Total sub-project budget in EUR	1 032 700
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	332 800
VINNOVA	247 312
Other public funding	
VTT	28 998
Metla	48 343

Other funding	
Södra Cell	177 419
Pergo	106 452
Vapo Oy	30 000
Raunion Saha Oy	15 000
Koskisen Oy	10 000
Heinolan Sahakoneet Oy	30 000
Ponsse Oy	6 000
Rovaseutu Kuntayhtymä	376

RESEARCH TEAMS

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sub-project 1	**Common practices, database and dissemination of results**			
Sven-Olof Lundqvist, Tekn.Lic,/ Major Project Manager "Optimal Wood and Fibre Utilazation"	M	STFI-Packforsk AB		
Örjan Hedenberg, Senior reseach associate	M	STFI-Packforsk AB		
Thomas Grahn, Senior reseach associate	M	STFI-Packforsk AB		
Arto Usenius, D.Sc. (Tech), Research Professor	M	VTT		
Harri Mäkinen, Ph.D.	M	Metla		
Annikki Mäkelä, Ph.D., Professor	F	University of Helsinki		
Dag Molterberg, D.Sc., Wood Raw Material Specialist	M	Södra Cell		
Sub-project 2	**Integrated model system for properties of stems**			
Annikki Mäkelä, Ph.D., Professor	F	University of Helsinki		
Anu Kantola, M.Sc., Ph.D. student	F	University of Helsinki		
Harri Mäkinen, Ph.D.	M	Metla		
Arto Usenius, D.Sc. (Tech), Research Professor	M	VTT		
Sanna Härkönen, M.Sc., Designer	F	University of Joensuu		
Åke Hansson, M.Sc., Senior research associate	M	STFI-Packforsk AB		
Sven-Olof Lundqvist, Tekn.Lic., Major Project Manager "Optimal Wood and Fibre Utilazation"	M	STFI-Packforsk AB		

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sub-project 3	**Data and models for wood and fiber properties**			
Sven-Olof Lundqvist, Tekn.Lic., Major Project Manager "Optimal Wood and Fibre Utilazation"	M	STFI-Packforsk AB		
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Åke Hansson, M.Sc., Senior research associate	M	STFI-Packforsk AB		
Harri Mäkinen, Ph.D.	M	Metla		
Pekka Saranpää, Ph.D., Docent	M	Metla		
Tuula Jaakkola, M.Sc.	F	Metla		
Olli Räsänen, M.Sc.	M	Metla		
Arto Usenius, D.Sc. (Tech), Research Professor	M	VTT		
Isabel Pinto Seppä, D.Sc. (Tech), Research Scientist	F	VTT		
Jorma Fröblom, M.Sc. (Tech), Senior Research Scientist	M	VTT		
Sub-project 4	**Data and models for chemical composition of wood**			
Dag Molterberg, D.Sc., Wood Raw Material Specialist	M	Södra Cell		
Alexandra Wigell, M.Sc., Project leader	F	Södra Cell		
Örjan Hedenberg, Tekn.Lic., Senior reseach associate	M	STFI-Packforsk AB		
Sub-project 5	**Advanced solid wood products and manufacturing processess**			
Arto Usenius, D.Sc (Tech), Research Professor	M	VTT		
Tiecheng Song, Lic.Sc. (Tech), Research Scientist	M	VTT		
Antti Heikkilä, M.Sc. (Tech) Research Scientist	M	VTT		
Jorma Fröblom, M.Sc. (Tech), Senior Research Scientist	M	VTT		
Harri Mäkinen, Ph.D.	M	Metla		
Annikki Mäkelä, Ph.D., Professor	F	University of Helsinki		
Sven-Olof Lundqvist, Tekn.Lic, Major Project Manager "Optimal Wood and Fibre Utilazation"	M	STFI-Packforsk AB		

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder
Sub-project 6	**Case study 1 - Wood-based composite flooring**			
Håkan Wernersson, Ph.D., Head of R&D	M	Pergo		
Tomas Stjernberg	M	Pergo		
Michael Lindström, Ph.D., Docent, Reaserch leader	M	STFI-Packforsk		
Kristofer Gamstedt, Ph.D. student	M	STFI-Packforsk		
Sub-project 7	**Case study 2 - Customer designed fibres**			
Dag Molterberg, D.Sc., Wood Raw Material Specialist	M	Södra Cell		
Catharina Kockmann, M.Sc., Project leader	F	Södra Cell		
Maria Edberg, M.Sc., Project leader	F	Södra Cell		
Sven-Olof Lundqvist, Tekn.Lic., Major Project Manager "Optimal Wood and Fibre Utilazation"	M	STFI-Packforsk AB		
Sub-project 8	**Case study 3 - Selection of suitable raw material for customer-specified**			
Annikki Mäkelä, Ph.D., Professor	F	University of Helsinki		
Anu Kantola, M.Sc., Ph.D., student	F	University of Helsinki		
Arto Usenius, D.Sc. (Tech), Research Professor	M	VTT		
Antti Heikkilä, M.Sc. (Tech), Research Scientist	M	VTT		
Tiecheng Song, Lic.Sc. (Tech), Research Scientist	M	VTT		
Sub-project 9	**Case study 4 - Profitable secondary conversion concepts for end user mill**			
Arto Usenius, D.Sc. (Tech), Research Professor	M	VTT		
Antti Heikkilä, M.Sc. (Tech), Research Scientist	M	VTT		
Tiecheng Song, Lic.Sc. (Tech), Research Scientist	M	VTT		

DEGREES

Degrees earned or to be earned within this project

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
2006	M.Sc.	M	Olli Räsänen	University of Helsinki	Pekka Saranpää, Metla, Marketta Sipi, University of Helsinki
2007	Ph.D.	F	Tuula Jaakkola, 1979, M.Sc. 2003	University of Helsinki	Pekka Saranpää, Metla, Marketta Sipi, University of Helsinki

Abstract

The general project objective was to build an improved knowledge-base and new tools, which will support the development and production of new innovative, eco-efficient products with a high content of further converted wood or wood fibres. For this purpose, a database with measurement data and an integrated system of models for growth and properties of trees have been developed. Selected stands of Norway spruce and Scots pine, representing different growth conditions and ages of trees, have been sampled in Sweden and Finland. Samples have been analysed for a wide range of properties, including knots and other stem structures with X-ray tomography, wood and fibre properties with SilviScan, fibres with microscopy, chemical composition of wood and pulp properties. The database is structured to provide related data on properties of stands, trees, logs, wood, knots, fibres and important properties of products from the different sectors of the forest-based industries. It has been extensively used as a basis for modelling of growth and properties. A tool has been developed to facilitate the use of the models for simulation and visualisation of growth and properties and how they are influenced by growth conditions. The data, models and tools have been used in three case studies; addressing production of both pulps and products from solid wood, with the objectives was to optimise the allocation of wood to suitable products and also the production in the mill. The improved knowledge-base and new tools proved to be useful. They are now used in further research and development by the partners.

Tiivistelmä

Hankkeen tavoitteena oli lisätä puumateriaalin tuntemusta ja kehittää uusia välineitä tukemaan innovatiivisten ja ekotehokkaiden puu- ja puukuitutuotteiden kehittämistä. Tavoitteen saavuttamiseksi koostettiin tietokanta tarkkaan mitatuista puista ja puuaineen ominaisuuksista sekä laadittiin integroitu mallijärjestelmä ominaisuuksien ja teollisuuden tuotantoprosessien ennustamiseksi. Aineisto koottiin eri ikävaiheita edustavista ja erilaisissa olosuhteissa kasvaneista kuusikoista ja männiköistä sekä Ruotsista että Suomesta. Koepuista mitattiin lukuisia ominaisuuksia, kuten runkojen geometria ja sisäoksien ominaisuudet röntgentomografilla, puuaineen ja kuitujen ominaisuudet sekä SilviScanilla että mikroskooppikuvista kuvankäsittelyohjelmalla, ja puuaineen sekä sellun kemialliset ominaisuudet. Tietokanta on muodostettu hierarkkisesti metsiköistä, puista, tukeista, oksista, puuaineesta, kuiduista ja lopputuotteiden ominaisuuksista siten, että tietoja voidaan yhdistellä joustavasti puuta jalostavan teollisuuden tarpeiden mukaan. Tietokannan perusteella laadittiin malleja, jotka kuvaavat puiden kasvua ja runkojen ominaisuuksia sekä niiden riippuvuutta puiden kasvuolosuhteista. Mallit on yhdistetty simulointijärjestelmäksi, jolla simuloitujen runkojen ominaisuuksia voidaan tarkastella myös visuaalisesti. Hankkeessa koottua aineistoa, malleja ja simuloin-

tijärjestelmää käytettiin tapaustutkimuksissa, joissa tarkasteltiin sekä mekaanisen että kemiallisen metsäteollisuuden tuotantoprosesseja. Tapaustutkimusten tavoitteena oli optimoida puuraaka-aineen ohjaus eri tuotantoprosesseihin ja lopputuotteiksi. Esimerkkilaskelmien perusteella hankkeessa kehitetyt menetelmät paransivat tarkasteltujen yritysten tuotantoprosessien kannattavuutta. Hankkeessa koottua tietokantaa ja uusia menetelmiä on tarkoitus hyödyntää jatkotutkimuksissa sekä kehittää edelleen.

Sammanfattning

Det övergripande projektmålet var att skapa en bättre kunskapsgrund och nya verktyg till stöd för utveckling och produktion av nya, innovativa och miljöeffektiva produkter med ökat innehåll av mer förädlade träkomponenter eller fibrer. Med detta mål har projektet utvecklat en databas med mätdata om och ett integrerat system av modeller för träds tillväxt och egenskaper. Prover har tagits i Sverige och Finland från utvalda bestånd av gran (*Picea abies*) och tall (*Pinus sylvestris*), som representerar olika växtbetingelser och trädåldrar. Prover har analyserats med avseende på ett stort antal egenskaper, bland annat kvistar och andra stamstrukturer med röntgentomografi, ved- och fiberegenskaper med SilviScan, fibrer med mikroskopi, kemisk sammansättning hos ved och massaegenskaper. Databasen är strukturerad för att tillhandahålla samhörande värden på egenskaper hos bestånd, träd, stockar, ved, kvistar, fibrer och viktiga egenskaper hos produkter från olika grenar av skogsindustrin. Den har utnyttjats för omfattande arbete med modellering av tillväxt och olika ved- och fiberegenskaper. Dessutom har en plattform utvecklats for att förenkla användandet av modellerna for simulering och åskådliggörande av träds tillväxt och egenskaper samt hur dessa påverkas av olika växtbetingelser. Data, modeller och plattformen har använts i tre fallstudier, som handlat om tillverkning av både massa och produkter av trä, med målet att optimera utnyttjandet av veden med avseende på produkt men även själva tillverkningsprocessen. Den förbättrade kunskapsgrunden och de nya verktygen fungerade väl. De utgör nu grunden för ytterligare forskning och utvecklingsarbete hos projektdeltagarna.

16.1 Introduction

16.1.1 Background

New innovative products from wood will provide new benefits to society based on renewable raw materials. They will also offer new opportunities for industry and forestry. For development of such products and efficient ways to produce them, there is a need for:

- new knowledge on many properties of wood
- new methods to optimise allocation of raw materials and industrial production

In the Innovood project, researchers and companies from different areas of expertise in Finland and Sweden have joined forces in order to improve the basis for this. The aim is also to favour the development and use of new products based on wood contra alternatives based on non-renewable resources. This is a contribution to sustainable development. Wood-based manufacturing is often performed in small and medium size enterprises, in many cases located close to the resources and in areas where their success or failure has a large impact on the local community. The strengthening of their competitiveness has, thus, a large importance for the society. Cornerstones of the project are databases, model-based tools and case studies.

16.1.2 Objectives

The general objective was to build up an improved knowledge-base and new tools, which will support the development and production of new innovative products with a high content of further converted wood or wood fibres.

16.2 Results and discussions

A keystone of the Innovood project has been the building of a database for Nordic softwood with related data on properties of stands, trees, logs, wood, knots, fibres, etc., including the properties

of most importance for the different sectors of the wood-based industries. The data on different properties originate from the same wood materials, allowing not only the investigation of variations but also studies of relationships, building of models and development of new and better products. Models have been developed, spanning a wide range of levels of detail relevant for the different industries: from "log scale" models, relevant for yield in sawing and kraft cooking, to "fibre scale" models, describing fibre length, width and wall thickness within growth rings.

During the first year, sampling and measurement activities were emphasized. Existing models were compiled and adapted for the project. Some of these models have later been further developed based on new property data compiled in the database. During the second year, the work on measurements and models continued. Models for other properties were developed. A framework was developed for simulation and visualization of growth and property variations within and between stems. The data, models and simulation tools were finally applied in case studies related to different products in the different sectors of the forest-based industries.

16.2.1 Sample material and database

16 stands of Norway spruce and Scots pine in Sweden and Finland were selected to represent Nordic softwood of different ages and growth conditions. Large, medium and small size trees, totally 48 trees, were samples at different heights along the stem. Radial variations were determined to describe systematic differences between parts of trees. Property variations in different scales were determined, from averages of stem/log cross-sections to within growth ring variations, depending on the features to be analysed:

- Stand and tree data were collected on sampling in the forest.
- VTT Building and Transport measured the branching structure within the stems (wood structure, size and properties of knots) with X-ray tomography, figure 1.
- STFI-Packforsk analysed radial variations in wood density, fibre width, fibre wall thickness and microfibril angle with SilviScan, figure 2, and calculated wood stiffness and other properties. The annual growth of the trees was evaluated. Averaged for annual rings and their earlywood and latewood were calculated. Pulps were produced from radial sub-samples and analysed for fibre length.
- Metla analysed fibre length variations between individual annual rings from the pith to the bark, as well as within growth rings with microscopy, figure 3.
- STFI produced pulps from wood of different origins, sheets for manufacturing of composites and analysed the sheets for paper related properties, figure 4.
- Pergo produced fibre composites from these sheets and tested them for moisture uptake and dimensional stability.
- Södra Cell FoU characterized the chemical composition of a large number of radial sub-samples (lignin, cellulose, hemi-celluloses and extractives).

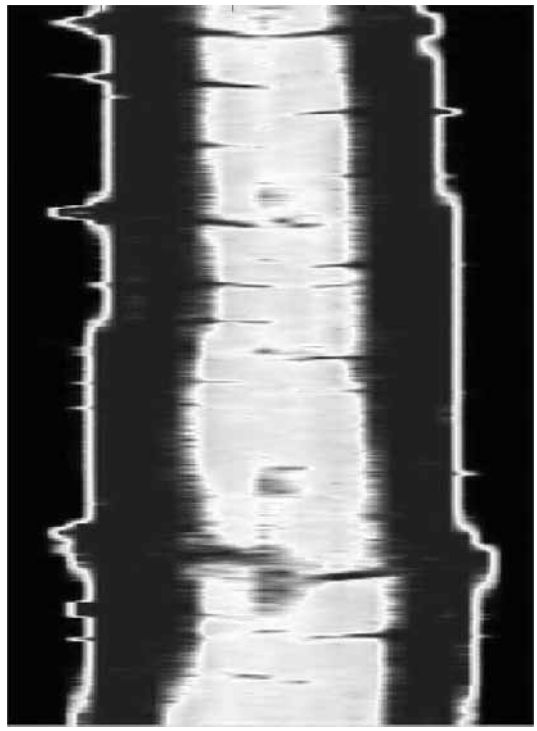


Figure 1. Knots of different characters in a spruce log measured with X-ray tomography (VTT).

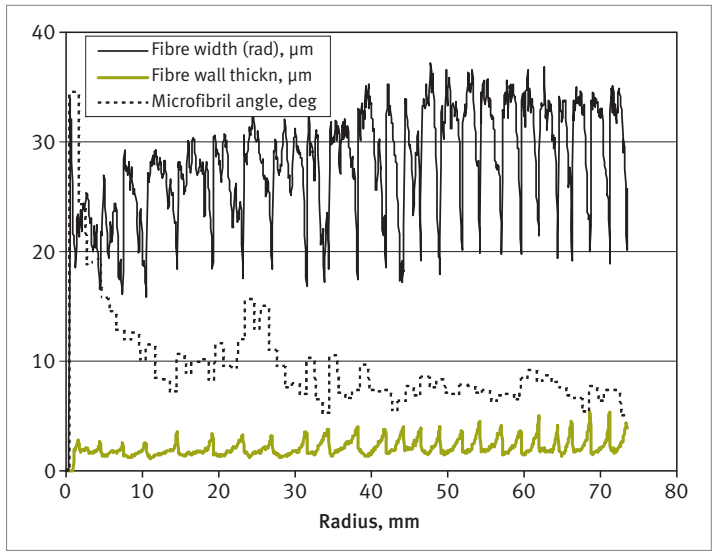


Figure 2. Radial variations in fibre width, fibre wall thickness and microfibril angle at breast heights in a spruce tree, analyzed with SilviScan (STFI-Packforsk).

STFI-Packforsk has built the database in which the data are compiled. The database is a good basis for evaluation of property variations, studies of relationships and development of models. It is a unique basis for continued research and development of applications, with a large potential in many areas. The project partners will continue to perform research based on these data, individually and in different cooperative constellations.

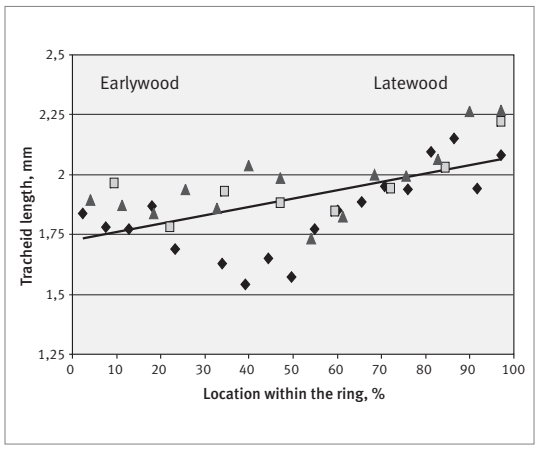


Figure 3. Radial variation of fibre length from early- to latewood of annual rings in juvenile wood of three trees of Scots pine (marked with different symbols) and a linear regression line (Metla).

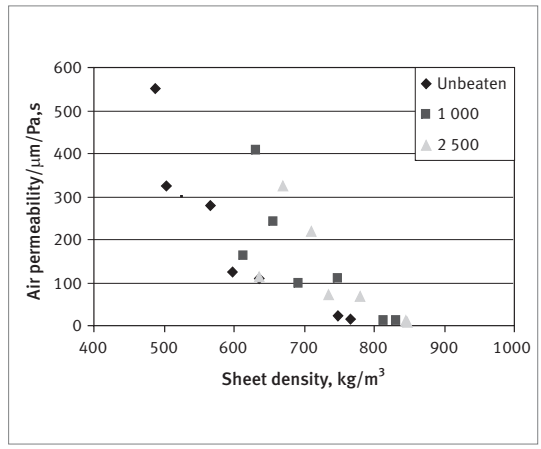


Figure 4. Properties of pulps produced from wood of various origins after different refining. Example: Air permeability versus sheet density for pine pulps produced from very fast-grown to very slow-grown wood (STFI-Packforsk).

16.2.2 Models and simulation

An integrated set of models has been developed, describing relationships between:

1. stand and site properties and tree growth
2. tree growth and wood properties, such as knots, in 3-dimensional stems
3. wood and fibre properties, such as fibre length, in 3-dimensional stems

The models also describe the property variations within and between stems and stands.

An important part of the model system is the RetroSTEM model, developed by University of Helsinki and Metla. With this model, the internal structure of trees, such as widths of growth rings and heights of internodes, may be estimated from external measurements, knowledge about previous thinning, etc.

When this structure is known, other models are used to estimate the within stem variations of important properties, such as wood density, number and sizes of knots and fibre dimensions. In figure 5, this is illustrated with the maximum diameters of knots in all whorls along a stem, showing measured data and data estimated with models from University of Helsinki and Metla. Figure 6 shows the radial variation in wood density from pith to bark (averages of growth rings) for two stem cross-sections. This is illustrated based on data measured with SilviScan and data simulated with models developed by STFI-Packforsk and Metla.

The "Innovood Tree Visualiser" has been developed to facilitate simulations with the models in conjunction with information in the database or other data, figure 7. It has been implemented by University of Helsinki as a stationary full version to be used for instance in the case studies and by STFI-Packforsk as a web-based limited version, which will become a showcase for the project and possible to use in education, etc. Growth and properties of trees grown under different conditions may be visualised and images may be generated for download and use in presentations and publications (if proper references are given).

The level of detail in these simulations of property variations within stems is averages for individual annual rings of arbitrary cross-sections. With this detail, properties of logs, sawn products and sawmill chips from different parts of the stem may be calculated.

VTT has improved its previous models for value chains, emphasising chains for products from solid wood, and developed new functionalities. The new tool is called "InnoSim". Routines are now implemented also for the bucking of stems into logs and for calculation of properties of different wood entities along the value chain: from raw materials of different origins all the way to stage of the ready wood components.

Figure 8 illustrates a virtual chain from tree to product. Stems are divided into logs, which are analysed with X-rays and evaluated for determination of location and properties of all knots in the stem. Similar information from estimation of other properties may be added. Various sawing patterns, or other alternatives, may be simulated and the results are compared, including also grading of "virtual boards", in order to find optimal solutions. Various alternatives may be simulated for optimization.

16.2.3 Case studies

Three case studies have been performed. The case study "Customer designed fibres", managed by Södra Cell, has focused on how the raw-material affects the product properties. Södra has built a large dataset of basic properties of logs, representing the supplies of pulpwood to three of its pulp mills. Södra has used data from the pulp investigations of STFI-Packforsk, see figure 4, to develop models for pulp properties, such as water retention value, sheet density, tensile index and tear index. With these and other models, the wood density, chemical composition of wood, fibre dimensions and properties of the pulp produced have been estimated for pulpwood logs.

Statistical distributions have been estimated for these properties for the three mills. This is illustrated in figure 9 with the fibre length distributions of wood delivered to the mills, calculated from estimated averages for bundles on trucks. Wide distributions for fibre length and other properties (not shown) indicate that the wood can be segregated into classes with different fibre properties for various products, if the benefits reached from such se-

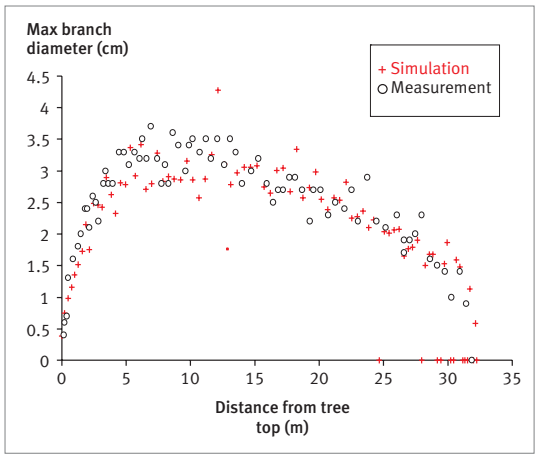


Figure 5. Measured and simulated maximum branch diameter for whorls along the stem of a tree: the smallest branches (knots) at the top of the trees and the largest at the base of the crown (University of Helsinki and Metla)

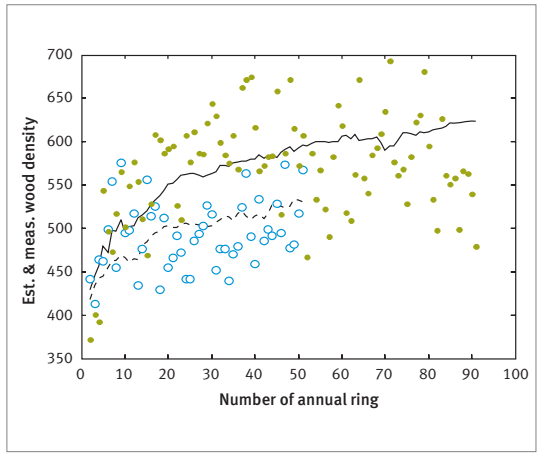


Figure 6. Measured and estimated variation in wood density (averages for annual rings) for two cross-sections sampled from Scots pine (STFI-Packforsk).

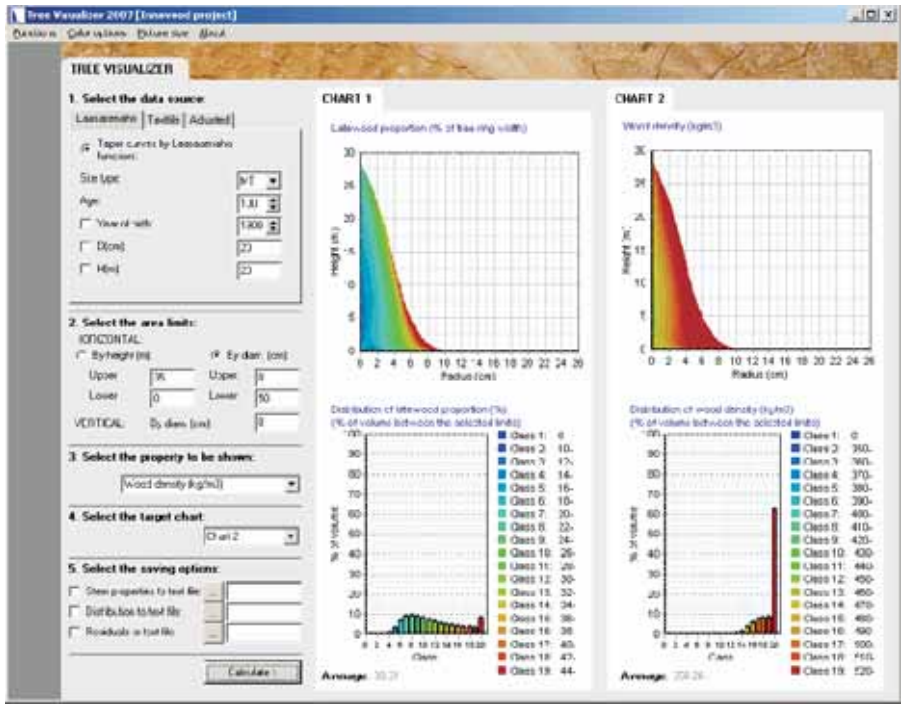


Figure 7. User interface of the "Innovood Tree Visualiser", showing growth, latewood content and wood density, simulated with Retro- STEM growth models and property models (University of Helsinki and STFI-Packforsk).

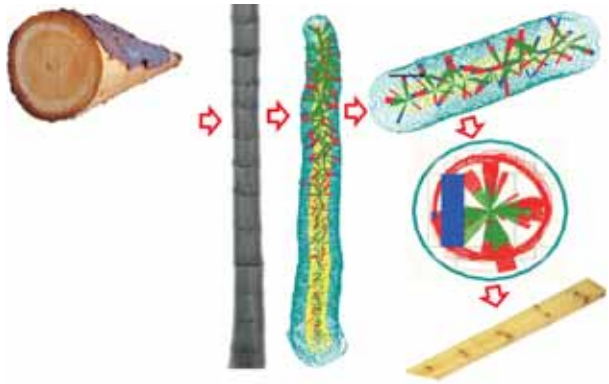


Figure 8. Example of analysis in a virtual chain from tree to product: Stems and logs are scanned and their interior properties are evaluated. Properties are estimated (not shown). Results of different sawing strategies are simulated and compared (VTT).

lection exceed the costs. The consequences of using different types of wood raw materials for production of various pulp products have been simulated, providing an improved knowledge-base for selective use of wood for improved pulp and paper products.

In the case study "Profitable secondary conversion concepts for end user mill", managed by VTT, a number of different conversion alternatives have been simulated with the value chain models mentioned above. Simulation results have been analyzed to support strategic decision made by sawmills. In figure 10, the value yields obtained with traditional cant sawing and with the more recently introduced "live sawing" are compared. For this type of logs, live sawing has clear advantages, especially for logs with smaller top diameters. The results clearly indicated that there is a big potential to increase the value yield by producing value added components instead of traditional bulk production. The sales value of the production may increase with up to 100 percent, under favourable conditions even more.

In the case study "Selection of suitable raw material for customer-specific products", performed by University of Helsinki and VTT, three different forest stands were selected for comparison. Based on stand data, virtual stems were generated using the RetroSTEM models. The InnoSim tool was used to simulate cross-cutting of the stems and conversion into final sawn timber products according to the demand on the market, as well as to analyse the results of different alternatives. It was demonstrated that there was a clear ranking of the stands regarding cost efficient production of the desired products.

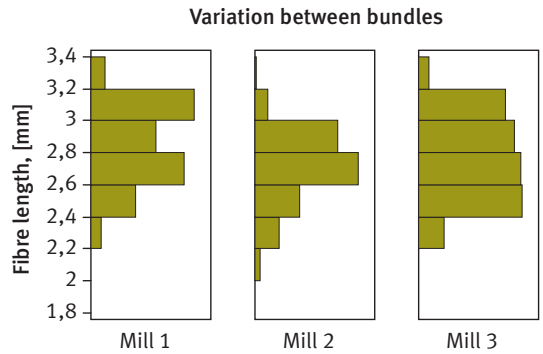


Figure 9. Fibre length distributions for wood used in three different mills of Södra Cell, estimated with models in conjunction with data on logs delivered to the mills. The distributions are calculated from estimated averages for bundles on trucks (Södra Cell).

To communicate the results of the project, a public concluding seminar was organised at STFI-Packforsk in March 2007. Some results are published and further dissemination is being prepared. The cooperation has inspired the partners to several joint applications to new research programs. Some project results are already applied in re-

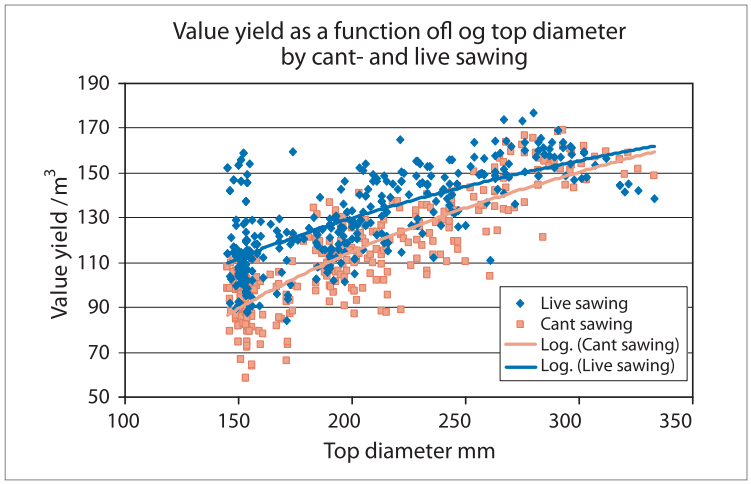


Figure 10. Comparison of value yield resulting from cant sawing and "live sawing", using the tool "InnoSim". Live sawing has clear advantages, especially for logs with small top diameter (VTT).

search and development and further applications in industry are being discussed.

16.3 Conclusions

An improved knowledge-base and new tools supporting the development and production of new wood-based products have been created. This includes the database on important properties of stands, trees, logs, wood, knots, fibres and products. It also includes new models and an integrated system for simulation of tree growth and properties, and how these are influenced by growth conditions. The data, models and tools developed in the project have proven to be useful in case studies in different sectors of the industry. They are forming a basis for further research and development of applications of the partners, individually and in cooperation.

By merging the competences and resources of the research partners, it has been possible to address difficult tasks involving different sectors of the forest-based industries as well as forestry. The cooperation has also strengthened the Nordic innovation system through the formation of a denser network among partners complementing each other, which has inspired the partners to take initiatives to new joint projects.

16.4a Capabilities generated by the project

The following capabilities have been generated by the Innovood project:

- The new database with data on many properties of importance for different wood-based industries, including measurement data on properties of stands, trees, wood, knots, logs, boards, chemical composition, fibres and pulps of Norway spruce and Scots pine trees sampled in Sweden and Finland.
- New raw material data, expanding the existing stem bank databases of VTT and wood and fibre databases of STFI-Packforsk on properties of various forest resources, used to reach for more efficient allocation and processing.
- New models for wood density and fibre dimensions, describing averages of individual annual rings from pith to bark in arbitrary stem cross-sections.
- New methods for identification of the most efficient and significant independent variables and model structures.
- The new system of integrated models for estimation of differences in growth and in wood and fibre properties within and between trees and stands. The system includes the RetroSTEM

simulation system for estimating growth of wood and knots in previous years from current measurements on trees.

- The new "Innovood Tree Visualiser" tool for simulation of growth and property distributions within stems at various growth conditions and for visualisation of these property variations.
- The web-based version of the Tree Visualiser, with functions useful in education, etc. It also includes measurement data on a set of trees, useful to visualise examples of trees grown under different conditions.
- New models for chemical composition of wood and pulp properties of wood of various origins, based on a limited sub-set of the samples.
- New data on within annual ring variations from measurements with microscopy at Metla and with SilviScan at STFI-Packforsk, providing a better understanding of the full property variability on the fibre level, for instance in pulps.
- The InnoSIM model system and software developed at VTT for simulation of whole conversion chain – from the forest to the sawn timber products and components. It is a new research tool especially for designing future manufacturing processes, new type of products and new harvesting strategies. This is possible due to very accurate description of wood raw materials, processes and products. The InnoSIM software is integrated with the RetroSTEM growth simulation system, which provides virtual stems as additional input data.
- Results from use of InnoSIM, providing a basis for optimization of future conversion processes based on product specifications and for valuation of potential forest stands to be harvested.
- Improved models and procedures to predict pulp properties resulting from use of wood raw materials of different origins, developed by Södra Cell for investigations at Södra pulp mills.
- The project has strengthened the Nordic innovation system through fruitful networking between research groups with different specialties in Sweden and Finland.

16.4b Utilisation of results

The database with related data for all parts of the forest sector: wood conversion, pulp and paper and forestry, is a unique basis for continued research and development with a large potential in several areas. The project partners will continue to perform research and development based on these data, individually and in different cooperative constellations.

Knowledge gained in the project about demands of paper products on wood raw materials is being further utilized by STFI-Packforsk in the European EFORWOOD project (Tools for Sustainability Impact Assessment of the Forestry-Wood chains).

The methods for identification of the most efficient and significant independent variables and model structures arrived at in the Innovood project have been further developed and rationalised. They are now used in other projects for modelling of other forest resources.

The vizualisation tool is used in teaching and in research by other groups. It is also incorporated in other model systems as an additional vizualisation resource.

The data and models developed in the project and tested in the cases studies have also been used to further develop existing tools by VTT (WOODCIM, InnoSIM) and by STFI-Packforsk (WoodSim, FibreSim) for optimal allocation and processing of wood and fibres for specific products and mills. There is still a potential to further improve these tools and to add new functionalities based on the data and models from the project.

The InnoSIM model system is the key tool in the big (budget 1 million euros) Finnish industrially oriented project "Adaptive and flexible manufacturing systems for wooden components". The project aims at creating new concepts to move from bulk products and production to value added products with specified product properties.

InnoSIM is now applied in several ongoing contract works for woodworking companies, within and outside Europe. The goal of the biggest project is to find optimal processing strategies for a company planning to build new mills. InnoSIM is also

used in the EFORWOOD project and several other national and international projects are in the planning stage.

Procedures developed in the project for comparison of pulps from wood of different origins are now being applied on other wood species in the STFI-Packforsk research Cluster "Tools for Optimal Fibre Utilization", funded by a group including several of the largest paper companies in Sweden and Finland.

Data and models developed within the project are now utilized in further studies of wood use and pulp properties by Södra Cell.

The project partners are now involved in a number of joint applications to the WoodWisdom – Net programme. These initiatives have benefited from the results of the project and also from the improved knowledge among the partners about available resources, methods, competences and new research possibilities through cooperation.

16.5 Publications and communication

a) Scientific publications

The most important publications are indicated with an asterisk.

1. Articles in international scientific journals with referee practice

Jaakkola, T., Mäkinen, H., Sarén, M.-P., and Saranpää, P. 2005. Does thinning intensity affect the tracheid dimensions of Norway spruce? Can. J. For. Res. 35: 2685-2697.

Jaakkola, T., Mäkinen, H. & Saranpää, P. 2006. Wood density of Norway spruce: responses to timing and intensity of first commercial thinning and fertilization. For. Ecol. Manage. 237(1): 513-521.

Jaakkola, T., Mäkinen, H. & Saranpää, P. 2006. Wood density within Norway spruce stems. Wood Sci. Technol. (submitted).

* Jaakkola, T., Mäkinen, H. & Saranpää, P. 2006. Thinning and fertilisation cause only minor changes in tracheid dimensions and lignin content of Norway spruce. Holzforschung (in press).

Kantola A. and Mäkelä A. 2006. Development of biomass proportions in Norway spruce (Picea abies [L.] Karst.). Trees 20(1):111-121.

Kantola, A., Mäkelä, A. 2004.Crown development in Norway spruce (Picea Abies [L.] Karst.) Trees 18:408-421.

* Kantola, A, Mäkinen, H. & Mäkelä, A. 2006. Stem form and branchiness of Norway spruce as a sawn timber – predicted by a process based model. For. Ecol. Manage. 241: 209-222.

* Mäkinen, H., Jaakkola, T., Saranpää, P. & Piispanen, R. 2006. Predicting wood and tracheid properties of Norway spruce. For. Ecol. Manage. 241: 175—188.

* Mäkinen, H., Jaakkola, T. & Saranpää, P. 2007. Variation of tracheid length within annual rings of Norway spruce and Scots pine. Holzforschung (submitted).

Vanninen P., Härkönen S., Enkenberg J. and Mäkelä A. 2006. PuMe – Interactive learning environment employing the PipeQual model for forest growth and wood quality. New Zealand Journal of Forestry Science 36 (in press).

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series

Lundqvist, S.-O. (editor), Innovative use of wood and fibres – Results from the Innovood project. Documentation of the concluding seminar, Stockholm, March 21, 2007 (A web-based version and a referable paper version in the format of an STFI-Packforsk Report are being prepared.)

Mäkinen, H., Ojansuu, R., Jaakkola, T., Saranpää, P., Hynynen, J. & Mäkelä, A. 2005. Predicting wood and branch properties of Norway spruce from simple stand and tree properties. In: Nepveu, G. (ed.). Proceedings of IUFRO WP S5.01-04 Fifth Workshop 'Connection between forest resources and wood quality: Modelling approaches and simulation software'. Waiheke Island Resort, Auckland, November 20-26, 2005, New Zealand. (in print)

Song, T., Pinto, I., Usenius, A. 2005. Sawing Simulation of pine heartwood products as a new WoodCIM® feature. In: Nepveu, G. (ed.). Proceedings of IUFRO WP S5.01-04 Fifth Workshop 'Connection between forest resources and wood quality: Modelling approaches and simulation software'. Waiheke Island Resort, Auckland, November 20-26, 2005, New Zealand. (in print)

Usenius, A., Song, T. 2005. Optimal Model system for optimal allocation of wood raw material throughout conversion chains. . In: Nepveu, G. (ed.). Proceedings of IUFRO WP S5.01-04 Fifth Workshop 'Connection between forest resources and wood quality: Modelling approaches and simulation software'. Waiheke Island Resort, Auckland, November 20-26, 2005, New Zealand. (in print)

Usenius, A., Song, T., Marjavaara, P., 2005, Automated Heartwood Detection and Optimization of the Manufacturing of Heartwood Components, ScanTech Conference 2005, Las Vegas, USA.

Usenius, A., Heikkilä, A. and Song, T. (2006) WoodCIM®Sistema de Software Integrado para Soporte en la Toma de Decisiones en Aserraderos – desde el Bosque hasta los Productos Finales (WoodCIM® - integrated software system supporting decision making at the sawmills - from the forest to the end products. Proceedings of Scantech 2006. Buenos Aires, Argentina 2-3.11.2006 Expo VESTAS and Wood Machining Institute, Berkeley, USA, pp. 4–30.

Under preparation

Lundqvist, S.-O. (editor), Key products of the forest-based industries and their demands on wood raw material properties. Public report within the EFORWOOD project, April 2007 (being finalized).

Lundqvist, S.-O., Mohlin, U.-B., Johansson, M., Key products of the paper industry and their demands on wood raw material properties, STFI-Packforsk Report, May 2007 (being finalized).

b) Other dissemination

Jaakkola, T., Mäkinen, H. & Saranpää, P. Influence of thinning intensity on wood properties in Norway spruce. The Forestry Woodchain - Quantification and forecasting quality from forest to end product. Heriot-Watt University, Edinburgh, 28-30 September, 2004. (poster)

Jaakkola, T., Mäkinen, H., Saren, M.-P., Saranpää, P. Effects of thinning intensity on growth rate, wood density and fibre properties in Norway spruce. International Symposium on Wood Sciences, October 24-29, 2004, Montpellier, France (poster)

Jaakkola, T., Mäkinen, H. & Saranpää, P. Harvennusvoimakkuuden vaikutus kuusen kasvunopeuteen, puuaineen tiheyteen ja kuidunpituuteen. Forest Science days, 20.10.2004, Helsinki. (poster, in Finnish)

Jaakkola, T., Mäkinen, H., Sarén, V.-M. & Saranpää, P. Vaikuttaako harvennusvoimakkuus kuusen (Picea abies) (L.) Karst) puuaineen rakenteeseen? Forest Science days, 20.10.2004, Helsinki. (poster, in Finnish)

Lundqvist, S.-O., The Innovood project. Presentation at the annual meeting of the programme "Wood Material Science and Engineering", Stockholm, 050413

Jaakkola, T., Mäkinen, H. & Saranpää, P. Tracheid properties of Norway spruce grown at long-term thinning-fertilisation regimes. Forest Science days, 18.10.2005, Helsinki. (poster, in Finnish)

Jaakkola, T., Mäkinen, H. & Saranpää, P. Ympäristötekijöiden vaikutus puun ja puukuitujen ominaisuuksiin. Paremmasta parempaa - Puuraaka-aineen määrän ja laadun optimointi metsän kasvatuksessa ja teollisuuden prosesseissa. Suomen Luonnonvarain Tutkimussäätiö, 29.3.2006, Helsinki. (presentation, in Finnish)

Jaakkola, T., Mäkinen, H. & Saranpää, P. Ympäristötekijöiden vaikutus puun ja puukuitujen ominaisuuksiin. Forest Science days, 1.11.2006, Joensuu. (poster, in Finnish)

Jaakkola, T., Mäkinen, H., Sarén, M. & Saranpää, P. Tracheid properties of Norway spruce grown in different thinning and fertilisation regimes. 6th PRWAC, December 3, 2005, Kyoto, Japan. (presentation)

Jaakkola, T., Mäkinen, H. & Saranpää, P. Tracheid properties of Norway spruce grown at long-term thinning-fertilisation regimes. 2nd meeting of the Nordic-Baltic network in wood material science & engineering, 30-31 October 2006, Stockholm, Sweden. (presentation)

Lundqvist, S.-O., Poster presentations at the annual meetings of the programme "Wood Material Science and Engineering", Helsinki 2004, Stockholm 2005 and Helsinki 2006

Lundqvist, S.-O., "Innovood – Multi-sectorial database, model system and case studies", Poster at Conference COST Action E44 "Wood as a renewable resource", 15-17 November 2004, Växjö, Sweden

Lundqvist, S.-O. (organiser), Concluding seminar: Innovative use of wood and fibres – Results from the Innovood project. Stockholm, March 21, 2007

Lundqvist, S.-O., Innovood – improved knowledge-base for innovative use of wood. Article in STFI-Packforsk Partner Update, May 2007 (in press).

Prof. Arto Usenius, VTT, is using models and materials produced by the project in his teaching at the Lappeenranta University of Technology in courses on "Information technology for wood industry" and "Woodworking and Secondary conversion of wood products".

Results on wood properties and processing have been presented by VTT in a number of national and international meetings with strong industrial involvement.

Results have continuously been presented to the pulp and paper industry by STFI-Packforsk. One channel has been the industry board of the Cluster ´Tools for Optimal Fibre Utilization (TOFU)", in which several of the largest Swedish and Finnish paper companies participate. Results have also been presented nationally and internationally to other companies, universities and institutes at meetings and within other cooperative research projects.

Similar information activities have been performed also by the other project partners, addressing also the forestry side.

c) Degrees

Räsänen, O. 2005. Effect of site fertility and thinning on wood quality of Scots pine (Pinus sylvestris L.). MSc thesis. Department of Forest Resource Management, Faculty of Agriculture and Forestry, University of Helsinki. (in Finnish with English summary)

Jaakkola, T. 2007.Environmental control of wood and tracheid properties of Norway spruce. Department of Forest Resource Management, Faculty ofArgiculture and Forestry, University of Helsinki.

d) Publications and other dissemination under preparation or planning

* Olsson, L., Grahn, T. Lundqvist, S.-O., Mäkinen, H., Models for annual ring averages of wood density, fibre dimensions and latewood content in Norway spruce and Scots pine. Conference presentation and scientific publication (Tentative title. Manuscript under preparation).

Kantola, A. Härkönen S., Mäkinen H. and Mäkelä A. Evaluating the RetroSTEM model for Norway spruce. Manuscript (Tentative title, forum to be decided).

Lundqvist, S.-O. et al, Articles about the Innovood project and its results in scientific and technical magazines and newspapers for different target groups, successively during 2007.

Lundqvist, S.-O. et al, Integrated measurement database and models for within stem variations in wood and fibre properties of Norway spruce and Scots pine on annual ring level. Examples from simulations. Conference presentation and scientific publication (Tentative title. Co-authors and forum to be decided)

Mäkelä, A., Härkönen, S. et al, Platform for simulation and visualization of growth and property variations in tree stems. (Tentative title. Under planning, suitable forum to be identified)

Hansson, Å., Lundqvist, S.-O., Härkönen, S., Simulate properties in tree stems with new tool on the web. (Tentative title. Article in scientific/technical magazine, under planning)

16.6 National and international cooperation

Advisory board of the consortium

Coordinator *Sven-Olof Lundqvist*, STFI-Packforsk
Deputy Coordinator *Arto Usenius*, VTT
Anders Pettersson, STFI-Packforsk
Arto Usenius, VTT
Pekka Saranpää, Metla
Annikki Mäkelä, University of Helsinki

From the industrial and funding parties
Dag Molteberg, Södra Cell
Håkan Wernersson, Pergo (Europe) AB
Juha Ropilo, Heinolan Sahakoneet Oy
Ismo Heinonen, VAPO Timber Oy
Olli Raunio, Raunion Saha Oy
Pekka Ulvas, Koskisen Oy
Tuomo Moilanen, Ponsse Oy
Juha Vaajoensuu, Tekes
Bengt Larsson, VINNOVA

Research partners

STFI-Packforsk (Coordinator)
Metla
Pergo (Europe) AB
Södra Cell
VTT
University of Helsinki

Funding parties

Tekes
VINNOVA
Pergo (Europe) AB
Södra Cell
Raunion Saha Oy
Koskisen Oy
VAPO Timber Oy
Ponsse Oy
Heinolan Sahakoneet Oy

Other contacts outside the project

In Sweden, experts from the Swedish Agricultural University (SLU) have been supportive in the acquisition of the sample material.

STFI-Packforsk has a long-term cooperation with CSIRO in Australia in the field of analysis of wood properties.

17 Functional Genomics of Wood Formation (FuncWood)

REPORT	
Name of the research project	**Functional Genomics of Wood Formation (FuncWood)**
Coordinator of the project	Teemu Teeri

BASIC SUB-PROJECT DATA	
Name of the sub-project	**Chemical and biochemical modification**
Project period	1.12.2005–31.12.2007
Organization in charge of research	University of Helsinki
Sub-project leader	Teemu Teeri
Contact information of the sub-project leader (institute/unit, address, telephone, fax, e-mail)	Department of Applied Biology P.O. Box 27 (Latokartanonkaari 7) 00014 University of Helsinki Tel. +358-9-191 58380 Fax +358-9-191 58727 teemu.teeri@helsinki.fi
URL of the sub-project	http://www.woodwisdom.fi/en/

FUNDING	
Total sub-project budget in EUR	1 368 000
Public funding from Wood Material Science and Engineering Programme:	Total funding granted in EUR by source:
Tekes	1 368 000
Other public funding	
University of Helsinki	Salaries for Fagerstedt, Helariutta, Kangasjärvi and Teeri Laboratory space and facilities
FGSPB	Part of salary for Warinowski
VGSB	Salary of Kaisa Marjamaa

RESEARCH TEAM

Name, degree, job title	Sex (M/F)	Organization, graduate school	For a visitor: organization & country of origin	Funder	Person-month
Kurt Fagerstedt, Professor	M	University of Helsinki		Univ. Helsinki	8
Yrjö Helariutta, Professor	M	University of Helsinki		Univ. Helsinki	8
Jaakko Kangasjärvi, Professor	M	University of Helsinki		Univ. Helsinki	8
Teemu Teeri, Professor	M	University of Helsinki		Univ. Helsinki	8
Anna Kärkönen, Ph.D.	F	University of Helsinki		Tekes	13
Arja Santanen, Ph.D.	F	University of Helsinki		Tekes	11
Sanna Koutaniemi, M.Sc.	F	University of Helsinki		Tekes	25
Tino Warinowski, M.Sc.	M	FGSBP		FGSPB/Tekes	25
Maaret Mustonen, technician	F	University of Helsinki		Tekes	25
Jorma Vahala, Ph.D.	M	University of Helsinki		Tekes	25
Hannele Tuominen, Ph.D.	F	University of Helsinki		Tekes	6
Tuula Puhakainen, Ph.D.	F	University of Helsinki		Tekes	7
Airi Lamminmäki, M.Sc.	F	University of Helsinki		Tekes	11
Tuomas Puukko	M	University of Helsinki		Tekes	13
Jarkko Salojärvi, Ph.D.	M	University of Helsinki		Tekes	8
Melanie Decourteix, Ph.D.	F	University of Helsinki		Tekes	24
Juha Immanen, M.Sc.	M	University of Helsinki		Tekes	24
Mikko Herpola	M	University of Helsinki		Tekes	24
Eija Kukkola, Ph.D.	F	University of Helsinki		Tekes/KCL	25
Kaisa Marjamaa, M.Sc.	F	University of Helsinki		VGSB	25
Total person-months of work conducted by the research team person-month = full-time work for at least 36 h/week, paid holidays included					323

DEGREES

Degrees earned or to be earned within this project.

Year	Degree	Sex (M/F)	Name, year of birth and year of earning M.Sc., D.Sc., ect. Degree	University	Supervisor of thesis, supervisor's organization
Autumn 2007	Ph.D.	F	Kaisa Marjamaa, 1973, M.Sc. 1998	University of Helsinki	Kurt Fagerstedt, University of Helsinki
Autumn 2007	Ph.D.	F	Sanna Koutaniemi	University of Helsinki	Teemu Teeri, University of Helsinki

Abstract

Wood is a major source of renewable raw materials extensively used in the pulp, paper, and timber industries. Intensive research efforts are ongoing in order to further the use of wood for green materials and green chemicals. This development has called for an increasing research interest in understanding basic molecular and physiological mechanism underlying wood variation and wood properties, with a final goal to grow trees with designed wood and fibre properties.

This project combines efforts in Finland and Sweden to obtain, using newly developed genomic tools, detailed knowledge of the molecular determinats of cambial activity and wood development.

Tiivistelmä

Puu on globaalisti merkittävä uusiutuvan raaka-aineen lähde, jota käytetään sellu- ja paperiteollisuudessa sekä rakennusmateriaalina. Puun käyttö luontoystäväällisenä materiaalina ja kemikalien raaka-aineena on myös intensiivisen tutkimuksen kohteena. Puunmuodostumisen sekä puun ominaisuuksien luonnollisen vaihtelun ymmärtäminen fysiologisella ja molekyylitasolla mahdollistaa tulevaisuudessa raaka-aineen täsmällisten ominaisuuksien jalostamisen.

Tässä hankekessa on yhdistetty suomalaisten ja ruotsalaisten tutkimusryhmien osaaminen ja uusin genomiikkaan perustuva lähetymistapa puunmuodostumisprosessin ymmärtämiseksi ja soveltamiseksi molekyylitasolla.

Sammanfattning

Ved utgör ett förnybart råmaterial, som i stor utsträckning används i såväl massa- och pappersindustrin som i byggnadsindustrin. Intensiv forskning pågår för att ytterligare öka användningen av ved som råvara för miljövänliga material och kemikalier. Denna utveckling har inneburit ett ökat intresse för forskning rörande de molekylära och fysiologiska variationerna i veden och dess egenskaper, varvid det slutliga målet är att möjliggöra odling av ved med önskade specifika egenskaper.

I detta projekt har forskargrupper från Finland och Sverige samarbetat för att genom utnyttjande av nytt genomiskt kunnande ta fram detaljerad kunskap på molekylnivå rörande vedens och dess egenskapers utveckling.

17.1 Introduction

17.1.1 Background

Domestication of woody species is needed for future supply of renewable raw material to the benefit of the environment. A major bottleneck in the application of molecular tools in tree breeding is inadequate knowledge of the genes underlying important traits. Current advance in gene sequencing and analysis of gene function in forest trees is opening the perspective of rapid progress in this field. High-throughput efforts, such as cDNA microarray analysis and proteomics, are expected to result in identification of key regulatory genes in wood formation. Such knowledge can then be implemented in tree breeding, either by the use of molecular markers or by transgene technology.

Pioneering work on wood and fibre modification using gene technology has already been successfully accomplished in the modification of lignin. Trees with either decreased lignin content or modified lignin chemistry have successfully been produced. Some of these genes have been tested in field trails, and despite the modified lignin the trees performed well under natural conditions and was shown to have improved pulping properties in large scale industrial tests. Genes regulating other cell wall components or wood and fibre properties are currently being discovered using molecular tools, and opportunities for modifying properties other than lignin content are thereby emerging.

It is believed both in wind sway and leaning that the wood forming tissues respond to signals generated by stem movement and the associated shift in gravity. The signalling system mediating this response is unknown. However, exogenous application of the plant hormone ethylene, a simple two carbon gas molecule, stimulates cambial growth. Ethylene has therefore been hypothesized to have a

role in mediating gravitational sensing into at least some aspect of reaction wood characteristics. The approach taken in this sub-project is to understand and dissect ethylene signalling resulting in increased cambial growth and modified fibers. This will provide novel knowledge in wood development and molecular tools to be used in future breeding. Our research is based on the solid foundation of ethylene perception/signalling research in the model plant *Arabidopsis*, the genome programs on poplar (*Populus tremula* × *P. tremuloides*, hybrid aspen) as well as birch (Betula pendula) initiated in Sweden and Finland, platforms for fiber characterisation developed at UPSC and the expertise on plant hormones and wood biology among the partners.

A major challenge for the implementation of forest biotechnology is to understand the molecular mechanisms underlying important traits in forest trees. In particular, information is required on the identity and function of genes and enzymes that regulate traits of interest. The poplar genome program initiated by the Swedish partners of this program, Umeå Plant Science Centre (UPSC) and The Royal Institute of Technology (KTH), has made Sweden a world leader in exploring the genome of forest trees, with the focus on modifying wood and fibres. This project is a part of the networks that are currently being established to co-ordinate and optimise efforts in forest biotechnology between Finnish and Swedish researchers.

17.1.2 Objectives

Hormonal control of wood formation

Cytokinin and wood. After annotation of the cytokinin related Populus genes, their expression will be analyzed across the cambial zone under standard conditions, under increased cytokinin concentration and in trees engineered to contain less cytokinins in the cambial zone. In addition to the spatial analysis using cryosections, we will investigate their expression during seasonal activation of the cambium.

Great emphasis will be given to the transgenic approach for understanding the role of cytokinin signaling during cambial development. To suppress cytokinin signaling, we will use two approaches. Firstly, we are going to express tissue specifically cytokinin oxidase in the cambial zone. Cytokinin oxidase genes have recently been effective in suppressing cytokinin responses. Secondly, together with Tatsuo Kakimoto we have recently obtained evidence that the mutant protein, mWOL, corresponding to the Arabidopsis wol mutation is in fact a phosphatase that acts negatively on the downstream pathway. Following this observation, we are going to introduce mWOL tissue specifically in the cambial zone.

To enhance cytokinin signaling, we will overexpress certain type B response regulatory genes (active in birch cambium) that have been suggested to positively regulate cytokinin signaling.

Substantial amount of this work will be conducted in UPSC where he will be co-supervised by Prof. Rishi Bhalerao.

Ethylene and wood. The main objectives are to understand the function of endogenous ethylene in wood formation and to identify the molecular components in the ethylene-dependent signalling pathway that are responsible in stimulating cambial growth and fiber modification. To accomplish this task, information is needed on candidate genes that regulate xylogenesis in an ethylene-dependent manner. In the later stage of the project, transgenic trees will be made to analyze the function of the ethylene-dependent genes in intact trees but also to modify the cell death specifically in xylem fibers and eventually also wood properties in forest trees. We will identify the ethylene-response factors (ERFs) that are active in wood-forming tissues, and responsible for the regulation of tension wood and ethylene-stimulated xylogenesis by real-time quantitative PCR (qPCR). These transcription factors will be expressed under constitutive and/or xylem-specific promoters. Analyses of wood formation and fiber quality in these lines will elucidate the exact role of ethylene in the processes. Decision of the genes to be expressed will be done based on experiments performed by both Finnish and Swedish partners (Björn Sundberg, UPSC and Hannele Tuominen, Umeå University). All the gene constructs needed for the transgenic trees will be constructed by the Finnish partner and transformations will be done in the UPSC high throughput

transformation facility as a paid service. The wood quality analyses of these lines will be performed by the Swedish partner. The results are expected to provide molecular tools for forest biotechnology applications and molecular markers for forest tree breeding.

Lignin biosynthesis

Genetic components of lignin formation in wood. Biosynthesis of monolignols in both softwood (spruce) and hardwood (birch and Populus) tree species is well understood, providing a characterized set of genes that mark this developmental process. Using analysis of samples collected during different biological processes of lignin biosynthesis (development of normal and reaction wood, wound responses, lignin formation in the cell culture, needles/leaves etc.), microarrays and bioinformatics are used to deduce through expression correlations which of the less well characterized genes are involved in the same process. The long sought after 'lignin forming' peroxidases and laccases are expected to stand out in this analysis.

Genomic and bioinformatics analysis will provide hypotheses (weaker and stronger) of genes that are involved in lignin formation or wood development in general. Verification will be done using detailed expression analysis in situ in developing wood, and finally using transgenic trees, where expression of the tested genes is altered. For birch, this is routine and will be done in our own laboratories. For spruce, the task is more difficult and is done in collaboration with Dr. David Clapham in the Swedish Agricultural University (SLU, Uppsala).

Design of breeding strategies for altered lignin composition in birch wood. Birch is an important hard wood model used extensively in our research. Identification of new lignin related genes will be tested in transgenic birch lines, but their analysis will fall beyond the scope of this proposal. Relating to reduced *CCoAOMT* expression and wood development, transgenic lines showing altered wood structure are already available. We seek to complete the molecular and chemical analysis of these trees. The latter is done in collaboration with Kristiina Poppius-Levlin/KCL (analysis of lignin content with the pyrolysis method developed in KCL). If successful, we will also assess the applied value of this wood as a raw material and iIf the transgenic experiments are promising, the next step along these lines would be to screen for a naturally occurring mutations to be used in selection breeding (without GMOs).

17.2 Results and discussion

A major technical task has been the design of oligonucleotide microarrays for birch, poplar and spruce. The birch array is part of a collaborative project, and an existing set of 8000 oligos were expanded by 4000. For spruce, the available technology had developed since the beginning of the project and instead of self-made oligonucleotide arrays, custom printed arrays were purchased. Both birch and spruce microarrays are based on EST collections developed earlier within the consortium (and its collaborators). The poplar genome sequence was finished during the project (with involvement of some of us, see Tuscan et al., 2006), and a microarray much larger than planned in this project will be available in the near future.

The spruce genes and gene family members for enzymes involved in monolignol biosynthesis and polymerisation were analysed using gene specific real-time reverse-transcription PCR. In the gene families encoding monolignol biosynthesis genes, by rule, a particular member of the gene family was most highly expressed. For genes encoding the putative polymerizing enzymes (peroxidases and laccases), the opposite was true and each tissue or condition had a specific set of genes active (Koutaniemi et al., 2007).

One of the tasks of this project has been the determination of cellular localisation and substrate specificities of the Norway spruce peroxidases cloned earlier.. This goal has been reached with a GFP-protoplast transformation technique, where the effect of signal peptide sequences of peroxidases in protein transport has been tested. Combined with experiments on substrate specificities, bioinformatics and a RT-PCR expression study, it has been concluded that PX1 and PX2 are expressed in normal xylem where they most probably take part in monolignol polymerisation. PX2 is induced in

compression wood xylem and and PX2 and PX3 are preferentially expressed in phloem after fungal infection Marjamaa et al., 2006; Koutaniemei et al., 2007). To further investigate the enzymatic properties of individual spruce peroxidases, a transient Agrobacterium leaf infiltration based expression method was optimised. We reached high expression levels (6% of soluble protein) with the test gene (luciferase), but the expression levels for peroxidases have not yet been determined.

Peroxidase genes (Pa-PX1, Pa-PX2 and Pa-PX3) are ready for the transformation of Norway spruce in collaboration with Assoc. Prof. David Clapham in Uppsala.

The distribution of specific lignin substructures such as dibenzodioxocin have been clarified in normal as well as in compression wood with antibody labelling under confocal fluorescence microscopy in collaboration with French researchers (Prof. Katia Ruel of CERMAV in Grenoble). Interestingly, in Norway spruce compression wood the condensed dibenzodioxocin (DB) structure in found in the inner part of the cell walls both in normal and compression wood, but in Scots pine compression wood DB is not found in this region. Hence, the two conifers differ significantly in their cell wall lignin structure in compression wood (Kukkola et al., 2007).

In collaboration with Karel Dolezalin (Umeå Plant Science Center), we have verified reduced cytokinin levels in the pBHK4::CKX2 *Populus* lines generated earlier. This opens possibilities for further studies on the role of cytokinins in the control of cambial activity of woody plants. We have further designed gene specific oligonucletotides for all *Populus trichocarpa* genes involved in cytokinin signalling to be used in real-time PCR analysis later.

A system for specific and continuous ethylene exposure to stem tissues was established by fixing sealed flow through chambers to stems of ca 2 m tall greenhouse grown tree. Treatment with a gas mixture of synthetic air, carbon dioxide (350 ppm) and ethylene (2 ppm) increased both xylem and phloem growth, and reduced vessel diameter and frequency. This experimental system was also required to produce large amount of ethylene treated material for subsequent analysis and tissue localisation of ethylene response factors.

By utilizing the existing knowledge about the Arabidopsis, rice and tomato ethylene pathway genes, we mined accordingly altogether 217 poplar genes involved in ethylene biosynthesis and action from the recently sequenced genome of *Populus trichocarpa*. As a result, the number of especially ethylene-response-factor (ERF) proteins (174 genes) that directly regulate ethylene-targeted genes was found to be significantly higher in poplar than in Arabidopsis and rice genomes. This may directly reflect the need for more complex involvement of ethylene pathway and ethylene-dependent gene induction for perennial growth habit and xylogenesis. To explore the involvement of ethylene on xylogensis, we designed gene specific primers for poplar ERF family according to *P. trichocarpa* gene models. When available, supporting information in the Populus EST database (http://www.populus.db.umu.se/) of hybrid aspen was always exploited for a gene model derived from *P. trichocarpa* genome to achieve accurate primersfor qPCR.

We are interested, which ERF genes are induced in active cambium and thus involved in the control and stimulation of xylogenesis. We screened with highly sensitive real-time qPCR the whole poplar ERF gene family using gene specific primers and whole-stem material, which was treated with 2 ppm of ethylene for two weeks. When compared to controls, we identified 53 genes at least with 2-fold up-regulation, 43 genes at least with 2-fold down-regulation, and 78 genes with no induction. Approximately 30% of poplar ERF genes are present in Umeå EST collections and thus in microarray gene chips. Microarray experiment (in UPSC) was conducted with *in vitro* -grown hybrid aspen material treated with ACC for 2, 5 and 10 hours. From this experiment, we identified seven ERF genes with a clear up-regulation. Two of these ERF genes showed similar result in response to both ethylene (with big trees) and ACC treatments (with *in vitro* trees). This indicates that some of the ERF genes might have considerably transient expression status, which we still must further investigate. Additionally, because of the surprisingly high number of up-regulated ERF genes by ethylene treatment,

we need to explore the ERF family in more detail. Especially to gain and confirm more knowledge about the cambium-specific ERF genes, a separately isolated cambial material treated with ethylene is currently under investigation. As well, ACC-treated *in vitro* -material is currently also under screening for the whole poplar ERF family, and experiments of short ethylene treatments of stem (1, 2 and 5 days) have been conducted to detect the ERF genes with transient inductions.

17.3 Conclusions

We have identified all genes involved in ethylene biosynthesis and signaling in poplar, and have tentatively identified candidate ethylene signaling genes (ERFs) responsible for wood modification.

17.4a Capabilities generated by the project

The final outcome of the project has not realized yet as our work is still continuing through the year 2007.

17.4b Utilisation of results

Knowledge generated during this project wil be used to strengthen both the basic understanding of wood development as well as capabilities in utilizing molecular knowledge in tree breeding (transgenic and non-transgenic). Many of the activities will reach to the future, e.g. the anaysis of peroxidase modified spruce trees. Similarly, we are currently in the way to generate transgenic poplar trees in order to stimulate wood formation by over-expressing chosen ERF-genes. These transgenic poplars could be utilized for example to provide high-output source of material for bioenergy production and paper industry.Results concerning Norway spruce peroxidases have been used in a collaborative project with Keskuslaboratorio, KCL, on the practical application of peroxidases in the pulping process.

17.5 Publications and communication

a) Scientific publications

1. Articles in international scientific journals with referee practice,

2. Articles in international scientific compilation works and international scientific conference proceedings with referee practice,

3. Articles in Finnish and Swedish journals with referee practice,

4. Articles in Finnish and Swedish scientific compilation works and Finnish and Swedish scientific conference proceedings with referee practice,

5. Scientific monographs, and

6. Other scientific publications, such as articles in scientific non-refereed journals and publications in university and institute series.

Marjamaa, K., Hildén, K., Kukkola, E., Lehtonen, M., Holkeri, H., Haapaniemi, P., Koutaniemi, S., Teeri, T.H., Fagerstedt, K. and Lundell, T. 2006: Cloning, characterization and localization of three novel class III peroxidases in lignifying xylem of Norway spruce (Picea abies). Plant Mol. Biol., 61:719-732.

Kukkola, E., Saranpää, P. and Fagerstedt, K.V. 2007: Juvenile and compression wood cell wall layers differ in lignin structure in Norway spruce and Scots pine. Provisionally accepted for publication in IAWA Journal.

Koutaniemi, S., Warinowski, T., Kärkönen, A., Alatalo, E., Immanen, J., Fossdal, C.G., Fagerstedt, KV, Simola, L.K., Rudd, S., Paulin, L. and Teeri, T.H. 2006: Delineation of the lignin biosynthetic pathway in Norway spruce. Submitted.

Tuskan, G. A., S. DiFazio, S. Jansson, J. Bohlmann, I. Grigoriev, U. Hellsten, N. Putnam, S. Ralph, S. Rombauts, A. Salamov, J. Schein, L. Sterck, A. Aerts, R. R. Bhalerao, R. P. Bhalerao, D. Blaudez, W. Boerjan, A. Brun, A. Brunner, V. Busov, M. Campbell, J. Carlson, M. Chalot, J. Chapman, G.-L. Chen, D. Cooper, P. M. Coutinho, J. Couturier, S. Covert, Q. Cronk, R. Cunningham, J. Da-

vis, S. Degroeve, A. Déjardin, C. dePamphilis, J. Detter, B. Dirks, I. Dubchak, S. Duplessis, J. Ehlting, B. Ellis, K. Gendler, D. Goodstein, M. Gribskov, J. Grimwood, A. Groover, L. Gunter, B. Hamberger, B. Heinze, Y. Helariutta, B. Henrissat, D. Holligan, R. Holt, W. Huang, N. Islam-Faridi, S. Jones, M. Jones-Rhoades, R. Jorgensen, C. Joshi, J. Kangasjärvi, J. Karlsson, C. Kelleher, R. Kirkpatrick, M. Kirst, A. Kohler, U. Kalluri, F. Larimer, J. Leebens-Mack, J.-C. Leplé, P. Locascio, Y. Lou, S. Lucas, F. Martin, B. Montanini, C. Napoli, D. R. Nelson, C. Nelson, K. Nieminen, O. Nilsson, V. Pereda, G. Peter, R. Philippe, G. Pilate, A. Poliakov, J. Razumovskaya, P. Richardson, C. Rinaldi, K. Ritland, P. Rouzé, D. Ryaboy, J. Schmutz, J. Schrader, B. Segerman, H. Shin, A. Siddiqui, F. Sterky, A. Terry, C.-J. Tsai, E. Uberbacher, P. Unneberg, J. Vahala, K. Wall, S. Wessler, G. Yang, T. Yin, C. Douglas, M. Marra, G. Sandberg, Y. Van de Peer, D. Rokhsar (2006) The Genome of Black Cottonwood, Populus trichocarpa (Torr. & Gray). Science 313:1596-1604.

b) Other dissemination

Vahala, J., Love, J., Björklund, S., Tuominen, H., Sundberg, B. and J. Kangasjärvi, J. Discovering the ethylene-dependent wood formation. COST-E28 WG1 Meeting, October 13-14 2005, Orleans, France (oral presentation).

17.6 National and international cooperation

The advisory board of the consortium is as follows:

Lars Gädda, M-Real

Markku Halinen , UPM-Kymmene

Christine Hagström-Näsi, Tekes

Hanna Rantala, Tekes

Mikko Ylhäisi, Tekes

Leena Paavilainen, Metla

Kristiina.Poppius-Levlin, KCL and Wood Wisdom

Liisa Viikari, VTT

Tapio Palva, University of Helsinki

Teemu Teeri, University of Helsinki

Members of the consortium are also part of the Finnish Center of Excellence in plant signal research (2000-2011; before 2006 under the name Plant Molecular Biology and Forest Biotechnology Research Unit) that consists of eight research groups. We have had both within this Center of Excellence and outside of it an extensive network of collaborations both nationally and internationally with both Universities and research institutes.

Appendix 1. **Number of human resources and Degrees earned within the programme**

Sub-programme 1 for basic research projects

	Total	Female/Male	Fin/Swe
Project coordinators	8	1/7	6/2
Sub-project leaders	24	5/19	12/12
Researchers	101	28/73	62/39
Degrees, total	26	11/15	20/6
MSc./M.Sc. (Eng.)	9	5/4	7/2
Lic.	2	-/2	1/1
Ph.D., D.Agr. & For., D.Sc., D.Tech.	15	6/9	12/3

Sub-programme 2 for innovation targeted research and development projects

	Total	Female/Male	Fin/Swe
Project coordinators	9	4/5	4/5
Sub-project leaders	36	15/21	18/18
Researchers	216	92/124	112/104
Degrees, total	26	15/11	10/16
M.Sc./M.Sc. (Eng.)	12	6/6	5/7
Lic.	9	5/4	1/8
Ph.D., D.Agr. & For.	5	4/1	4/1

Appendix 2. **Seminars and Meetings**

2003

Programme seminars (0)

Project Seminars (0)

Meetings between the Funding Organizations (1)

- October 8, Tekes, Helsinki

Programme Board Meetings (0)

Projects´ Advisory Board Meetings (3)

SPWT

- May 26, Metla, Helsinki
- September 10, Metla, Vantaa

STDUT

- December 15, SP-Trätek, Stockholm

2004

Programme Seminars (1)

- Opening Seminar and the poster exhibition on April 6, Marina Congress Center, Helsinki, Finland, 170 participants

Project Seminars (0)

Meetings between the Funding Organizations (4)

- March 2, VINNOVA, Stockholm
- August 30, Tekes, Helsinki
- November 30, VINNOVA, Stockholm
- October 25, Academy of Finland, Helsinki

Programme Board Meetings (1)

- September 10, Helsinki

Projects´ Advisory Board Meetings (31)

VACHA

- February 9, Metla, Helsinki
- November, 29, Metla, Helsinki

IMWO

- March 22, Metla, Helsinki
- August 23-24, Flakaliden, Umeå and Vindeln

SPWT

- May 5, SLU, Uppsala
- September 15, Wood Focus, Helsinki
- November 24, Metla, Vantaa

STDUT

- June 17, VTT, Espoo
- November 4, VTT, Espoo
- December 2, LUT, Skellefteå

BUNDLE

- May 12, KCL, Espoo
- November 10, STFI-Packforsk, Stockholm

WoodBiocon

- May 10, VTT, Espoo
- May 28, VTT, Espoo
- November 2, STFI-Packforsk, Stockholm

NewCell

- May 18, TUT, Tampere
- September 15, KTH, Stockholm
- October 12, ÅAU, Turku

NanoCell

- April 7, TKK, Espoo
- September 20, Kemira, Helsinki
- October 25, STFI-Packforsk, Stockholm
- December 8, Loparex, Lohja

ECOMBO

- April 8, Kick-off meeting, VTT, Espoo
- November 15, SP-Trätek, Stockholm

EcoMod

- November 22, VTT, Espoo

InnoFireWood

- April 6-7, VTT, Espoo
- October 4, SP-Trätek, Stockholm

InnoLongSpan

- August 26, VINNOVA, Stockholm
- December 9, VTT, Espoo

Innovood

- May 25, STFI-Packforsk, Stockholm
- August 23, Raunion Saha, Koski TL

2005

Programme Seminars (1)

- Annual Seminar, April 13, Citykonferensen, Stockholm, 120 participants

Projects´ Seminars (1)

- Metsäpuiden biotekniikka workshop 4.2. Helsinki Congress Paasitorni (mainly in Finnish). 70 participants

Meetings between the Funding Organizations (2)

- August 23, AoF, Finland
- December 9, Formas, Stockholm

Programme Board Meetings (2)

- February 3, VINNOVA, Stockholm
- September 28, AoF, Helsinki

Projects´ Advisory Board Meetings (33)

VACHA

- December 20, Metla, Helsinki

IMWO

- April 12, SLU, Southern Swedish Forest Research Center, Alnarp

SPWT

- May 25, Setra Group Ltd., Stockholm
- June 9, Wood Focus, Helsinki
- June 13, Setra Group Ltd., Stockholm

STDUT

- June 10, SP-Trätek, Skellefteå

BUNDLE

- April 21, KCL, Espoo
- November 22, KCL, Espoo

WoodBiocon

- May 3, VTT, Espoo
- November 10, ÅAU, Turku

NewCell

- January 25, Domsjö AB, Örnsköldsvik
- March 22, VTT, Espoo
- April 12, KTH, Stockholm
- August 24-25, TUT, Tampere
- December 14-15, Akzo Nobel, Stenungssund

NanoCell

- April 7, M-real, Espoo
- April 12, STFI-Packforsk, Stockholm
- August 17, HUT, Espoo
- October 10, STFI-Packforsk, Stockholm
- November 22, UPM-Kymmene, Tampere

ECOMBO

- March 22, VTT, Espoo
- April 14, KTH, Stockholm
- June 21, VTT, Espoo
- December 20, Conenor, Vantaa

EcoMod

- April 14, SP-Trätek, Stockholm
- October 25, VTT, Espoo

InnoFireWood

- April 4, Wood Focus, Helsinki
- October 18, SP-Trätek, Stockholm

InnoLongSpan

- May 26, Lund University, Lund
- November 17, VTT, Espoo

Innovood

- February 10, Koskisen Oy, Järvelä
- April 14, STFI-Packforsk, Stockholm
- September 7, Pergo, Trelleborg

2006

Programme Seminars (1)

- Annual Seminar, April 5, Marina Congress Center, Helsinki, 140 participants

Project Seminars (4)

- Forest Biotechnology Seminar, February 1, Helsinki Congress Paasitorni, 70 participants
- Nanostructured cellulose and new cellulose derivatives – a Joint Seminar, NanoCell and NewCell projects November 14, STFI-Packforsk, Stockholm, 33 participants
- Puun ominaisuudet ja käyttömahdollisuudet tulevaisuudessa -seminaari, VACHA–IMWO-projektit. 30.11. Porthania, Helsinki. Seminar language Finnish, 70 participants
- Specific wood and timber properties, competitive ability and advance conversion of Nordic pine in mechanical wood processing – SPWT-project, the final expert group and professional workshop, December 7, Uppsala, 20 participants

Meetings between the Funding Organizations (1)

- October 27, AoF, Helsinki

Programme Board Meetings (3)

- January 30, VINNOVA, Stockholm
- September 8, Tekes, Helsinki
- November 29, VINNOVA, Stockholm

Projects´ Advisory Board Meetings (22)

IMWO

- April 12, Helsinki

SPWT

- February 24,Metla, Helsinki
- December 7, Uppsala

STDUT

- March 13, Arlanda

WoodBiocon

- June 7, STFI-Packforsk, Stockholm

NewCell

- March 7
- April 4, Helsinki
- October 2, KTH, Stockholm

NanoCell

- January 16, UPM-Kymmene, Tampere
- May 22, HUT, Espoo
- November 6, HUT, Espoo
- November 13, STFI-Packfors, Stockholm

ECOMBO

- April 4, VTT, Espoo
- October 3, SP-Trätek, Borås

EcoMod

- May 30, SP-Trätek, Borås
- October 10

InnoFireWood

- March 7, Finnforest, Espoo
- June 9, SP-Trätek, Stockholm

InnoLongSpan

- May 11, Skogsindustrierna, Stockholm

Innovood

- February 7, VTT, Espoo
- April 4, VTT, Espoo
- September 21, STFI-Packforsk, Stockholm

2007

Programme Seminars (1)

- Final Seminar, May 22, Wenner-Gren Center, Stockholm

Project Seminars (2)

- Innovate Wood – Innovative use of wood and fibres. Results from the Innovood project, March 21, STFI-Packforsk, Stockholm, 20 participants
- Specific wood and timber properties, competitive ability and advance conversion of Nordic pine in mechanical wood processing – SPWT-project. Public Seminar, May 3, at Sibelius -talo, Lahti, Finland

Meetings between the Funding Organizations (1)

- March 20, Tekes, Helsinki

Programme Board Meetings (2)

- February 14, Telephone meeting
- April 20, Tekes, Helsinki

Project Advisory Board Meetings (5)

WoodBiocon

- March 30, VINNOVA, Stockholm

NewCell

- March 14-15, ÅAU, Turku

ECOMBO

- February 15, VTT, Espoo

EcoMod

- March 13, Restaurant Ylajali, Oslo

InnoLongSpan

- February 8, Metla, Helsinki

ABBREVIATIONS

AoF	Academy of Finland
TKK	Helsinki University of Technology
KCL	Oy Keskuslaboratorio – Centrallaboratorium Ab
KTH	KTH – Royal Institute of Technology – Kungliga Tekniska högskolan
LUT	Luleå University of Technology
Metla	Finnish Forest Research Institute
SLU	Swedish University of Agricultural Sciences
SP-Trätek	SP-Technical Research Institute of Sweden
STFI	STFI-Packforsk AB
Tekes	The Finnish Funding Agency for Technology and Innovation
TUT	Tampere University of Technology
UH	University of Helsinki
VINNOVA	Swedish Governmental Agency for Innovation Systems
VTT	VTT Technical Research Centre of Finland
ÅAU	Åbo Academy University

Appendix 3. **Publications**

Number of publications in the sub-programme for basic research projects (1-8)

	No of publications
a) Scientific publications	101
Articles in international scientific journals with referee practice	
Articles in international scientific compilation works conference proceedings with referee practice	33
Articles in Finnish and Swedish journals with referee practice	4
Articles in Finnish and Swedish scientific compilation works with referee practice	-
Scientific monographs	9
Other scientific publications (such as articles in scientific non-refereed journals and publications in university and institute series)	43
b) Other dissemination	168
(such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results).	
Patents (applications exist, numbers not available)	-
Manuscripts/plans	12
TOTAL	**370**

Number of publications in the sub-programme for innovation targeted research and development projects (9-17)

	No of publications
a) Scientific publications	26
Articles in international scientific journals with referee practice	
Articles in international scientific compilation works conference proceedings with referee practice	28
Articles in Finnish and Swedish journals with referee practice	-
Articles in Finnish and Swedish scientific compilation works with referee practice	-
Scientific monographs	3
Other scientific publications (such as articles in scientific non-refereed journals and publications in university and institute series)	21
b) Other dissemination	38
(such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results).	
Patents (applications exist, numbers not available)	
Manuscripts/plans	11
TOTAL	**127**

Number of Publications in the Finnish-Swedish Wood Material Science and Enginering Programme 2003-2007

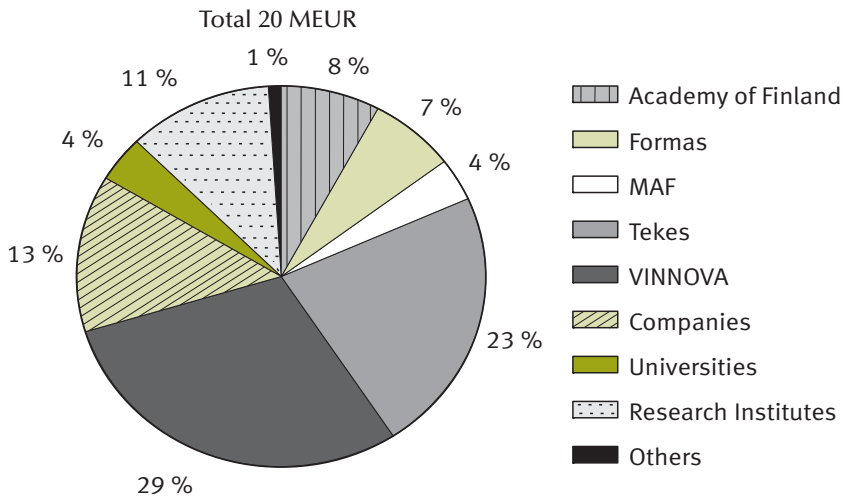
a) Scientific publications	127
Articles in international scientific journals with referee practice	
Articles in international scientific compilation works conference proceedings with referee practice	61
Articles in Finnish and Swedish journals with referee practice	4
Articles in Finnish and Swedish scientific compilation works with referee practice	-
Scientific monographs	12
Other scientific publications (such as articles in scientific non-refereed journals and publications in university and institute series)	64
b) Other dissemination	206
(such as text books, newspaper articles, TV and radio programmes, meetings and contacts for users and results).	
Patents (applications exist, numbers not available)	-
Plans/under preparation	23
TOTAL	**497**

Appendix 4. **Companies involved in the Wood Material Science and Engineering Research Programme**

Total number 48, of which 33 big companies and 15 SME´s *

* SME´s are small and medium companies with employees under 250 persons and annual turnover max 50 MEUR

Appendix 5. **Funding**



Wood Material Science
Research Programme

Wood Material Science and Engineering Research Programme

The Finnish-Swedish Wood Material Science and Engineering Research Programme (2003-2007) was ended in March 2007. The programme was designed to improve the competitiveness of the forestry sector and forest-based industry and to promote the sustainable use of renewable natural resources. The programme was funded by the Academy of Finland, the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning (Formas), the Ministry of Agriculture and Forestry of Finland, the Finnish Funding Agency for Technology and Innovation (Tekes), VINNOVA (Swedish Governmental Agency for Innovation Systems) and the forest cluster companies.

The programme created multidisciplinary core competences enabling the development of innovative and eco-efficient forest-based products and processes. In addition, it has strengthened the innovation system in the field of wood material science and engineering and intensified the transfer of knowledge and technology between producers and users. The programme consisted of two sub-programmes, one focusing on basic research and the other on innovation-targeted research and development.

The Wood Material Science and Engineering Research Programme, with a budget of 20 million euros, was unique even at European level, building a wood material research platform between five funding organizations, a research community spanning 30 institutes and universities, and more than 50 forest cluster enterprises. The programme had jointly planned goals and research areas and it was jointly implemented and managed. The projects involved Finnish and Swedish research partners and industrial companies.

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The Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning

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