WURC

Stage 1

1 July 1996 - 31 December 1998

Final Report

WURC (Wood Ultrastructure Research Centre) is a NUTEK Competence Centre based at SLU. Partners in the Centre are NUTEK, SLU, six pulp and paper companies and one chemical company. Research is performed in co-operation between SLU, CTH, KTH, STFI and the companies

WURC
Wood Ultrastructure Research Centre
Final Report - Phase 1

Sammanfattning

Etapp 1 har omfattat 30 månader, 1 juli 1996 - 31 december 1998. Deltagare i WURC har varit sex skogsindustriföretag och ett kemiföretag samt SLU, som också varit centrumets hemvist. SLU har inom WURCs ram samverkat med CTH, KTH och STFI. Verksamheten har varit inriktad mot grundläggande industrirelevant forskning om vedens och vedfiberns morfologiska och kemiska ultrastruktur samt fysikaliska egenskaper med fokus på ved och fibrer av gran. Forskningen har gradvis byggts upp under etappen och omfattar vid etappens slut 8 projekt där 11 högskole/institut-forskare och 8 doktorander medverkar. Industrideltagarna har aktivt medverkat i projektarbetet med att ta fram ved och specialtillverkade fibrer till alla projekt och utföra olika analyser. Forskningsresultat har presenterats vid internationella och inhemska konferenser och möten. Den vetenskapliga publiceringen har kommit igång under 1998. Centrumet har också anordnat två internationella seminarier med inbjudna framstående forskare, haft en gästforskare från Japan och initierat en COST-action (EU).

Den ekonomiska omslutningen under etappen har uppgått till 18,4 MSEK varav 10,2 MSEK utgjorts av kontanta bidrag från NUTEK och medlemsföretagen och 8,2 MSEK av naturabidrag från SLU (inkl. CTH, KTH och STFI) och medlemsföretagen.

Summary

Phase 1 comprised 30 months, 1st July 1996 - 31st December 1998. Partners in WURC have been six forest industry companies, one chemical company and SLU, the centre of WURC. SLU has, within the framework of WURC, co-operated with CTH, KTH and STFI. WURC is focused on basic and industrially relevant research on morphological and chemical ultrastructure as well as physical properties of wood fibres, which in Phase 1 was centred upon wood and fibre from spruce. Research has been built up gradually and comprises at the end of the first phase 8 projects in which 11 university/institute scientists and 8 PhD-students are contributing. The industrial members have actively taken part in the project work by producing specially designed pulps for all the projects and carrying out analyses. Research results have been presented at international and domestic conferences, seminars and meetings. Publication in scientific journals started during 1998. The centre has also organised two international seminars with prominent international scientists invited as

speakers, engaged a guest researcher from Japan and initiated a COST-action (EU).

The economic turnover in Phase 1 has been 18,4 MSEK of which the contribution in cash from NUTEK and the industry members amounted to 10,2 MSEK and the support from SLU (CTH, KTH and STFI included) and the industry members amounted to 8,2 MSEK.

1. Accomplishment of goals

WURC can be said to have two main goals: i) one aimed at developing an efficient organisation and creative research environment, with improved interaction, communication and networks and establishment of a recognised platform in science etc.; and ii) a second aim involving the production of novel research results.

Although WURC is still in a period of strong development, goals and visions set up at the start have already been partly achieved.

- It has been possible, based on the sparse and scattered competence that existed at the start of WURC, to organise and gradually strengthen the activity at the core organisation of WURC, i.e. at SLU/Uppsala.

- A clear competence profile and a research direction have been defined which gives WURC a clear identity within the Swedish research infrastructure.

- WURC has become internationally well known, i.e. through activities such as the annual international WURC seminar and the initiative and work for a European COST action on wood cell wall structure as well as through several presentations of WURC research at international conferences.

- WURC has been able to intensify the interdisciplinary university and institute research in its field of interest and to activate participation of the forest industry in WURC's research, although much more remains to be done in these respects in the future.

- The planned number of PhD-students for Phase 1 in WURC has been achieved

WURC's research has gradually been built up and results of real scientific value corresponding to the original aims began to appear during the second half of Phase 1. Research results from Phase 1 are reported briefly in chapter 4 and presented more in detail in Appendix 2. In addition to specific results, the following more general comments can be made:

- The results achieved show that projects initiated during Phase 1 are well qualified to continue in Phase 2.

- Interesting results have been obtained regarding various properties of different types of pulp which can constitute the basis and material for further research to improve the understanding of interactions between the morphological and chemical ultrastructure on the one hand and the physical properties of pulp fibres on the other.

The original long term goals and visions for WURC are still valid. WURC is focused on basic research of industrial relevance. The major objective is to enhance the industrial utilisation of wood fibre by significantly improving the understanding of its morphological and chemical ultrastructure including the interaction between constituting wood polymers. In addition, it was agreed to concentrate research on spruce in Phase 1 and also to a great extent in Phase 2. A few minor changes in planned activities on the project level have also taken place. One project was for instance transferred from STFI to SLU (resignation of the original project leader) and one project planned to start in Phase 2 was begun already towards the end of Phase 1 (process-initiated damages and distorted zones was identified as an important problem).

2. Additional value of co-operation through a competence centre

Concentrated organisation for wood ultrastructure research

Before the creation of WURC, a few geographically separated and small research groups in Sweden carried out most of the research on wood ultrastructure as a subsidiary part of their research activities. The group at SLU was one of those. WURC as a competence centre required a considerable concentration of the research to SLU. As a result of the establishment of WURC, the morphological and microscopical resources and knowledge at SLU, earlier mainly directed towards wood as a solid material and its biodegradation, was redirected for studies of wood and fibres for paper production. The specific competence and resources at KTH, CTH and STFI were made available for WURC by separate agreements with SLU. Thereby it has been possible to organise, in a proper way, the wood and fibre ultrastructure research in Sweden, which was earlier at a low level and dispersed.

Research concentration

In this competence centre the best university and institute resources in wood fibre research in the country have been brought together, their research efforts concentrated, initially on spruce wood and spruce fibres, and a technol-scientific approach adopted for tackling the research tasks. This strategy we believe has improved the chances for successful research and scientific breakthroughs of the centre.

Improved interdisciplinary collaboration

An advantage with an organisation like WURC, which is crossing over the traditional university structure, is that inter-disciplinary and inter-organisational collaboration is stimulated. New channels have been opened and many more people now know each other than before. This is e.g. evident from the more integrated structure of the new projects planned for Phase 2.

Intensified industry-university contacts

Another clear advantage with a competence centre is that contacts between university and the industry in question have become easier and more frequent. This is especially true for SLU, where contacts with companies have traditionally been more developed in forestry. The improved contacts have been achieved by i.e. regular meetings, seminars, informal meetings and study visits. The fact that the Centre Director, during phase 1 has come from industry has also contributed much to the improved contacts between industry and university.

Improved understanding of industry and academia

The form of work in a competence centre is based on co-operation and confidence in a new way between industrial researchers and university scientists, leading to a more profound understanding of the different ways of work and working environments, which will be beneficial for both parts. It should also facilitate for university scientists to work in industry and thereby make reciprocity possible. In addition research questions of importance for industry can be studied in a scientific-technical way with clear aims and pronounced focus and with the involvement of several disciplines including targeted basic as well as applied research.

Research co-operation between industry and university

The way WURC is organised promotes an integrated research co-operation between industry and university, which is difficult to obtain in traditional types of co-operation. The industrial partners in WURC have been directly involved in WURC's research in several ways. Company representatives in the Industrial Advisory Group have taken active part in the planning of, and given their approval to all WURC-projects in operation. Furthermore, industrial laboratories have produced, analysed and characterised the wood and fibre raw material used in all projects during Phase 1. Special wood raw material and pulps used for specific purposes in certain projects have also been produced in industry laboratories. Co-operative research between industry and university concerning electron microscopy and ultrastructural fibre models has been initiated. Finally, the project groups include researchers from industry which allows industry close contact with WURC's research.

Industrial networks and recruitment of PhD:s

Since industry is closely involved in WURC's activities, WURC's PhD-students will obtain a thorough understanding of the industry and its research needs and they have an extraordinary possibility to build up their own industrial networks. This will, in all, facilitate the recruitment of qualified, knowledgeable and well-informed people to the Swedish forest industrial sector.

Visiting scientists

WURC, being a national competence centre in the field of wood ultrastructure research, is the natural base for bringing visiting scientists with interesting competence to Sweden.

3. Economy

The economic turnover in Phase 1 was 18 392 196 SEK of which the contribution in cash from NUTEK and the industrial members amounted to 10 160 041 SEK and the services from SLU (CTH, KTH and STFI included) amounted to 8 232 128 SEK. It should be remembered that Phase 1 was extended to 30 months instead of 24 months, which was the original budget period.

The income in cash exceeded the budget by 275 041 SEK mainly as a result of income from interest and cash from KTH. In addition the income was 354 370 SEK higher than the cash expenditure. Furthermore, 445 270 SEK, which constitutes overdrawn rent costs charged by SLU, is expected to be repaid. The result, 799 640 SEK (354 370 + 445 270) has been reserved for planned purchase of a new LV-SEM (Low Vacuum Scanning Electron Microscope). Services rendered from the companies as well as from SLU and partners altogether exceed the budget by 397 128 SEK.

Financial structure

	Cash		Services rendered	
Income	Results	Budget	Results	Budget
Source				
NUTEK	5 240 000	5 240 000	0	0
Assi-Domän AB	900 000	900 000	72 140	100 000
Eka Nobel AB	100 000	100 000	14 800	50 000
Korsnäs AB	650 000	650 000	122 186	125 000
Mo och Domsjö AB	840 000	840 000	99 460	100 000
SCA AB	630 000	630 000	243 425	100 000
STORA	1 050 000	1 050 000	813 547	660 000
Södra	475 000	475 000	54 400	100 000
SLU	0	0	4 695 480	3 800 000
KTH	100 000	0	524 000	700 000
CTH	0	0	857 130	700 000
STFI	0	0	735 560	1 400 000
Other sources	41 221	0	0	0
Interest	133 820	0	0	0
Total	10 160 041			

Expenditure of cash

Item	Cost	Responsible
Centre administration	405 073	SLU
Visiting scientist	166 000	SLU
Scientific conferences	259 985	SLU
Science activities at centre	1 451 064	SLU
Project 1	812 113	SLU
Project 2	2 018 095	SLU
Project 3	399 063	SLU
Project 4	1 000 000	STFI
Project 5	1 100 000	KTH
Project 6	1 000 000	CTH
Rent	1 194 278	SLU
Total	9 805 671	
Result	354 370	SLU

Note: WURC has appealed to SLU to transfer 445 270 SEK back to WURC. This amount corresponds to a too high deduction for rent.

4. Research projects

Research during Phase 1 has been conducted within 8 projects. These are listed below and some examples of interesting achievements are given. More detailed information about the projects is found in Appendix 2.

Project 1. Fibre models

Aim: to generate morphological fibre models for tracheids of spruce including data on chemical and physical properties.
Achievements: New modified models of the cell wall ultrastructure and a 3D-reconstruction of a tracheid have been accomplished.

Project 2. The ultrastructure of wood fibre surfaces

Aim: to characterise the surface ultrastructure of wood fibres and to investigate how this changes after various mechanical, chemical and enzymatic treatments.
Achievements: An unexpected "woven" structure with great voids in the fibre cell wall has been revealed by the use of the FE-cryo-SEM-technique. Furthermore, the original fibre ultrastructure in wood has been shown to change much more than expected during the pulping process.

Project 3. Dislocations or nodes in wood fibres

Aim: to study dislocations or weak points in the fibre wall and the ultrastructural background to their occurrence and impact on properties of paper fibre. The project started 1998-08-10.

Project 4. Fibre chemistry: structure of cellulose and hemicellulose

Aim: to elucidate important relations between structural characteristics of wood cellulose and hemicellulose and the reactivities and properties of the fibre substrate.

Achievements: Measurements using solid state ^{13}C-NMR have shown the crystallinity of cellulose to increases drastically during the sulphate pulping process. The technique used allows for example determination of cellulose chains on crystallite surfaces, and the amount of amorphous cellulose. The technique appears promising for further studies on wood and pulp fibres coupled to process studies.

Project 5. Fibre strength of pulp fibres

Aim: to increase the knowledge on how changes in molecular composition, polymer structure and ultrastructure influence the strength properties of pulp fibres, and how this depends on pulping conditions. This knowledge can then be used to design chemical pulping processes that will give a better fibre.

Achievements: differences in certain paper properties made from different pulps were found to be proportional to the cellulose/hemicellulose content, leading to the conclusion that a decrease in cellulose/hemicellulose ratio gives a stiffer, more brittle fibre.

Project 6. Ultrastructural modification of wood with respect to metal ions

Aim: to determine the occurrence, localisation and extractability of different metal ions in wood.

Achievements: Distribution of different metals in spruce wood before and after chelation, acid treatment or ion exchange has been demonstrated on a micro-level i.e. by using Synchrotron Radiation Microbeam X-ray Fluorescence Analysis.

Project 7. Lignin and hemicellulose structures in wood fibres (financial support by SJFR)

Aim: to clarify the molecular structures of lignin and hemicellulose in spruce wood using advanced mass spectrometry.

Achievements: Lignin molecules up to 1500 Da have been ionised and analysed using positive ionisation. Lignin model compounds subjected to thioacidolysis have been identified using tandem mass spectrometry.

Project 8. Mechanical interactions between wood polymers and their orientation in wood structure (financial support from SJFR)

Aim: to obtain more knowledge on how the various wood polymers, cellulose, lignin and hemicellulose interact under mechanical loading and thereby facilitate conclusions of how different polymers affect the physical properties of fibres.

Achievements: Spectra of pure polymers have been recorded in order to assign their specific wave numbers. Some dynamic spectra have also been recorded.

5. Participants working in the Centre

Research groups

WURC's main activities have been at the Department of Wood Science (former Dept. of Forest Products), SLU, where the ultrastructural research has expanded very much in recent years as a consequence of the establishment of WURC. CTH, KTH and STFI are partners to SLU in WURC and provide necessary complementary competence and equipment.

The research group at SLU is directed towards studies on the morphological ultrastructure of wood, wood attacking microorganisms and wood preservation. The department has powerful microscopic and other equipment for research on wood biology/microbiology and a culture collection of wood degrading microorganisms. Engaged in WURC activities at the end of Phase 1 were: 4 senior scientists, 1 post doctoral scientist, 3 PhD-students, 2 technical staff members and one administrative person from the rectors office dealing with WURC's economy. The involvement in WURC has been successively upgraded during Phase 1 and a further increase is expected during Phase 2.

From KTH (Royal Institute of Technology), the Department of Pulp & Paper Technology conducts research within WURC. This department specialises in research on pulp and bleaching technology/chemistry, mechanical pulping, chemicals from wood and analysis of biomass compounds. It is well equipped for this type of research. At the end of Phase 1 two senior scientists and 2 PhD-students (one financially supported by SJFR) were engaged in WURC-projects.

STFI (the Swedish Pulp and Paper Research Institute), a joint resource for the Swedish pulp and paper industry and its allied industries has approximately 225 employees, of which about 130 are university graduates. Regarding research of relevance to WURC, STFI has excellent competence on natural polymers, wood polymer physics and chemistry, surface chemistry, wood mechanics, measurement of fibre properties, pulping chemistry and functional demands on fibres. The equipment for chemical analysis, including NMR, is particularly relevant. At the end of Phase 1, three senior scientists and two PhD-students (one financially supported by SJFR) were engaged in WURC projects.

At CTH (Chalmers University of Technology) the Department of Forest Products and Chemical Engineering conducts research within WURC. Research of particular interest to WURC at this department deal with pulping and bleaching and with chemical wood and fibre modification. Equipment of interest is a pilot plant for chemical modification of wood and instrumentation for chemical analysis of wood and pulp. At the end of Phase 1 two senior scientists and one PhD-student were engaged in WURC's research. The group co-operates with the Department of Physics at CTH regarding analysis of metal ions, which has made possible the use of the European Synchrotron Radiation Facility in Grenoble.

Industrial partners

During Phase 1 WURC has had 7 industrial partners, *viz.*: the forest industry companies AssiDomän, Korsnäs, Mo och Domsjö, SCA, StoraEnso (former STORA) and Södra Cell and the chemical company EKA Chemicals.

The industrial partners have contributed significantly to the development of WURC during Phase 1. WURC's Director comes from one of the companies. Five out of eight Board members are also from industry. Research personnel from the industrial companies have been active as members of the Industrial Advisory Group, various Project Groups and as Industrial Contact Persons for the PhD-students. Study visits for the PhD-students have been arranged by SCA and StoraEnso. The companies have also taken part in WURC's research in several ways. Examples of interaction include:

- a representative sample of spruce wood has been produced for experimental purposes in the different projects
- different types of sulphate pulps have been produced by the industrial laboratories to be used in all WURC projects
- specific pulps have been produced for project no 5 aimed at the improved utilisation of the inherent strength of fibre materials
- pulps have been analysed and their properties determined
- pulps from wood chips made in chippers with knives of different quality have been produced and analysed for further studies in project no 3.
- ultrastructural fibre studies using LV-SEM (Low Vacuum Scanning Electron Microscopy) have been conducted in co-operation with researchers in project no 1.
- occurrence and distribution of metals in spruce sulphate pulps have been studied in co-operation with researchers in project no 6.
- results from studies made in industrial laboratories and mills on different subjects of wood storage, chipping, pulping, bleaching and pulp strength have been presented at a WURC seminar on pulp quality.

During these activities responsible researchers as well as technical staff in all industrial companies have participated.

Persons working in the Centre

A. From SLU and university/institute partners

Persons working in the centre and their activity in man years at the end of Phase 1* are listed below.

Category/Name	*Activity, man years*	
Professors		
Geoffrey Daniel, SLU	0.90	
Göran Gellerstedt, KTH	0.10	
Björn Henningsson, SLU	0.50	
Tommy Iversen, STFI/KTH	0.35	
Thomas Nilsson, SLU	0.70	
Rune Simonson, CTH	0.10	
Ants Teder, KTH	0.10	2.75
Senior scientists		
(univ.lektorer, forskare)		
Paul Ander, SLU	0.90	
Tomas Larsson, STFI	0.10	
Lennart Salmén, STFI	0.10	1.10
Post doctoral scientists		
(forskarassistenter)		
Stig Bardage, SLU	0.90	
Harald Brelid, CTH	0.10	1.00
PhD-students		
Annica Berglund, CTH	1.00	
Jonas Brändström, SLU	1.00	
Isabelle Duchesne, SLU	1.00	
Eva-Lena Hult, STFI/KTH	1.00	
Ulrika Molin, KTH	1.00	
Karolina Nyholm, SLU	1,00	
Margaretha Vickes, STFI/KTH	1.00**	
Hans Önnerud, KTH	1.00**	8.00
Administrative personnel		
Gabriella Danielsson, SLU	0.15	
Per Jennishe, SLU	0.10	
Brita Swan, WURC StoraEnso (STORA)	0.20	0.45
Technicians		
Ann-Sofie Hansén, SLU	1.00	
Eva Nilsson, SLU	0.50	1.50

* In 1997 civ.eng. E. Brännvall, KTH, was engaged for research in project no 5
** Financial support by SJFR

B. From industrial partners

Persons and research groups actively involved in WURC's research during Phase 1

Bo Ahlqvist, MoDo
Sture Backlund and co-workers (technicians), SCA
Anders Brolin and co-workers (foresters, engineers and technicians), StoraEnso
Ivan Dalin, EKA Chemicals
Monica Edsborg, AssiDomän
Ulrika Ekholm and co-workers (technicians) Korsnäs
Stefan Högman, Korsnäs
Ulla Jansson and co-workers (technicians) Södra Cell
Carina Johansson, EKA Chemicals
Ann Marklund and co-workers (technicians) MoDo
Frank Peng, StoraEnso
Ove Rehnberg, AssiDomän
Brita Swan, StoraEnso

6. Management, organisation and activities during Phase 1

WURC has been regulated by the following agreements:

- Avtal mellan Närings- och teknikutvecklingsverket, Sveriges Lantbruksuniversitet samt AssiDomän AB, Eka Chemicals AB, Korsnäs AB, Mo och Domsjö AB, SCA AB, STORA AB och Södra Cell AB om inrättande av ett Kompetenscentrum vid SLU

- Avtal mellan Sveriges lantbruksuniversitet och Chalmers Tekniska Högskola om samarbete inom Kompetenscentrumet WURC vid SLU

- Avtal mellan Sveriges lantbruksuniversitet och Kungliga Tekniska Högskolan om samarbete inom Kompetenscentrumet WURC vid SLU

- Avtal mellan Sveriges Lantbruksuniversitet och Skogsindustriens Tekniska Forskningsinstitut om samarbete inom Kompetenscentrumet WURC vid SLU

- Forskaravtal mellan WURC och forskare verksamma inom centrumet

WURC is located at the Department of Wood Science at SLU. WURC has a status which closely corresponds to that of an ordinary Department. WURC is responsible for its own economy. All cash money from the companies and NUTEK has been paid to WURC's account at SLU and then redistributed to the various consumers within WURC.

An organisation structure (see Appendix 1), rules for procedures and responsibilities within WURC were established by the *Board* in 1996. The Board consists of representatives from the industrial companies, SLU and other research partners and has been chaired by Lennart Eriksson, STFI. The *Board* has had 7 meetings during this phase. The chairman of the *Board* has had frequent meetings and other contacts with the Director and Managing group.

The daily management of WURC has been carried out by the *Managing group*. The group has been lead by the Director, Brita Swan, and has consisted of five persons. The group has met frequently (at least monthly) and has had frequent communication with the Chairman of the Board, the Industrial Advisory Group and with researchers and PhD-students within WURC. The group has been responsible for following up the economy and the research projects, working out and proposing research and activity plans, organising meetings, recruiting researchers and PhD-students etc.

The detailed planning of each research project has been carried out by the respective *Project leaders*, who have also been responsible for carrying out the research and for the economy of the project. Part of the research has been carried out by PhD-students. At the end of Phase 1 eight students were engaged in WURC. It should be stressed, however that *Senior scientists* have also carried out a substantial part of WURC's research and represent a relatively high number on the list of persons working in the centre at the end of Phase 1 (See chapter 5).

Research in the area of WURC is carried out by scientifically recognised groups in Canada, Finland, France, Japan and USA. Researchers from several of these groups have been invited speakers at the two *International Seminars* that WURC has so far organised (April 1997 and 1998) in Uppsala with good attendance. Internationally the WURC seminars have proved to be appreciated and unique in this field of research. It is planned to give such international seminars once a year.

The first in a series of "internal" WURC-seminars was held in Borgvik in February 1998. The aims were i) to disseminate knowledge and experiences among industrial and university/institute researchers about the properties of kraft pulp and how these properties are influenced by the wood raw material and its processing, and ii) to ensure that research in WURC, in addition to high scientific quality, also has industrial relevance. It is planned to arrange such seminars yearly.

An important task for WURC is to keep the industrial partners informed about research progress. To that end an informative seminar reporting on activities and results obtained during Phase 1 in the various WURC-projects was held at STFI in November 1998. The seminar was designed to be a more comprehensive follow-up of informative meetings and poster exhibitions at the companies. Furthermore, a special meeting concerning image analysis of wood and fibre structures was arranged in October 1998 with participation from both industry and academia.

7. International co-operation

International networks and contacts are essential for WURC's successful development. One reason for arranging the International Seminars outlined above was to develop and strengthen the international network. In addition to these seminars a series of other steps have been taken to increase the international scientific input into WURC.

In autumn 1997, WURC took the initiative to propose a so called COST-action: "Wood Fibre Cell Wall Structure". A European working group chaired by WURC (P. Ander) developed a proposal which has been processed within COST. Fifteen to twenty European institutions have expressed their interest to participate. An important aspect here, besides creating a European network on wood ultrastructure, is to link expertise from research on non-wood cell walls. (Final approval of this Action was taken within COST in February 1999.)

It is intended to regularly invite visiting scientists to WURC to strengthen or expand competence in certain areas of research. Professor N. Terashima from Japan spent autumn 1997 in Sweden as a visiting scientist (partly financed by WURC) to work on lignin chemistry. From August to December 1998 professor Keiji Takabe (Department of Wood Science and Technology, Kyoto University, Japan) joined WURC at SLU. His stay in Uppsala was particularly beneficial for project no 2. Professor Takabe also brought two of his PhD-students to work together with WURC researchers and PhD-students for a couple of weeks. The co-operative research established during professor Takabe's visit will continue and a second visit to Uppsala is planned during spring 1999.

New international channels have also been opened by project 6 as a result of the regular utilisation of the European Synchrotron Radiation Facility in Grenoble for studies on the ultrastructural distribution of metal ions in wood.

Through the activities mentioned and with respect to the international networks that WURC´s senior scientists already have, good conditions for future international co-operation should be ensured.

8. Results and effects at the company level

From the companies point of view the following has been expressed by the Industrial Advisory Group:

- As a whole WURC's research has been in progress for too short a time to bring about any great effect within the participating companies

- Through co-operation between the various participants in the Centre at several levels and on several fronts, and through an improved exchange between different universities/institute in the Centre etc., an improved general impression and awareness has been created among the companies compared to what can be achieved by "traditional co-operation" in single or a few projects carried out at individual research institutions. Co-operation within a Competence Centre also offers improved chances to make the research program more industrially relevant.

- Strong concentration to one specific area of research, wood fibre ultrastructure, will hopefully generate much new knowledge in a relatively short time.

- Improved contacts have been established between industrial researchers and university/institute scientists, i.e. through participation in the Advisor/Mentor Groups set up in the different projects. These improved contacts will also facilitate possible future employment of PhD-students.

- Methods and techniques used by university scientists in WURC have been adopted and used by industry researchers and vice versa.

- Contacts established in the Centre - in Adviser Groups, at seminars etc. - have increased the contact surfaces in all directions and the competence in WURC's field of research has been clearly improved in a wide sphere of industry people.

- The improved competence in industry provides a good base for increased exchange of knowledge and ideas between industry and university researchers and allows a more rapid implementation of research results obtained in the Centre.

9. Scientific production

At the start of WURC, only a very limited number of scientists, senior as well as post doctoral, were available. Therefore, much of the research started by engaging PhD-students which greatly influenced the time schedules for the projects during Phase 1. As a consequence, none of the projects were planned to be completed during the first phase and therefore no final results are available. However, WURC-activities in progress have been presented in several ways. Research results have for instance been reported at various conferences and meetings, including Tappi Biotechnology Conference, 1997 and the 7th International Conference on Biotechnology in the Pulp and Paper Industry, 1998. A limited number of written research papers have also been produced and certain manuscripts submitted to scientific journals.

Publications in scientific journals

Berglund, A., H. Brelid and R. Simonson (1999) Spatial distribution of metal ions in spruce wood by Synchrotron Radiation Microbeam X-ray Fluorescence Analysis. Accepted for publication in Holzforschung.

Brändström, J. (1998) Micro- and ultrastructural aspects of Norway spruce (*Picea abies* (L) Karst.) tracheids: A review. Submitted to IAWA Journal.

Daniel, G. and I. Duchesne (1998). FE-SEM observations on the surface ultrastructure of spruce kraft pulp fibres - Effects of preparatory techniques. Submitted to Holzforschung.

Duchesne, I. and G. Daniel (1999). The ultrastructure of wood fibre surfaces as shown by a variety of microscopical methods - A review. Accepted for publication in Nordic Pulp and Paper Research Journal.

Hult, E-L., P.T. Larsson and T. Iversen (1998) A comparative CP/MAS^{13}C-NMR study of the cellulose structure in spruce wood and kraft pulp. Submitted to Carbohydrate Research.

Conference contributions (international)

Ander, P. et al. (1998) The in vitro ^{14}C-labelled kraft pulp assay as used for evaluation of laccases and mediators intended for pulp bleaching. 7th Intern. Conf. on Biotechnology in the Pulp and Paper Industry, Vancouver BC, June 1998, Vol. A pp A 111 - A 115.

Ander, P. (1998) Enzymes - tools for ultrastructural research on wood fibres. WURC International Seminar, April 1998.

Awano, T., K. Takabe, M. Fujita, and G. Daniel (1998). Immuno-FE-SEM reveals distribution of xylan in beech. To be presented at 49th Annual meeting of the Japanese Wood Research Society, April 1998.

Berglund, A., H. Brelid and R. Simonson (1998): Metal ions - wood ultrastructure. WURC International Seminar April 1998.

Daniel, G. (1997) Use of HR-Cryo-FE-SEM for studies on wood and pulp fibre ultrastructure. International Cryo-Electron Microscopical Meeting, York, UK, June 1997.

Daniel, G. (1997). Studies on wood and pulp fibre ultrastructure using HR-CRYO-FE-SEM.1997 Tappi Biological Sciences Symposium, San Francisco, USA, Abstract p.447.

Daniel, G and I. Duchesne (1998) Revealing the surface ultrastructure of spruce fibres using Field Emission SEM. 7th Intern. Conf. on Biotechnology in the Pulp and Paper Industry, Vancouver BC, June 1998. pp 81-84. (Supplemented and further submission to Holzforschung).

Nilsson, T and G. Daniel (1997) The "true" structure of the wood fibre. WURC Wood Ultrastructure Seminar, April 1997.

Teder, A. (1998) Future chemical pulping processes. WURC International Seminar, April 1998.

Terashima, N., J. Hafrén, U. Westermark, Y. Xie, K. Fukushima, C. Lapierre and D. L. VanderHart (1998) Proposed 3D structural model for soft wood lignin. Prepared for the International Symposium on Wood and Pulping Chemistry, Japan , June 1999.

Domestic conferences and meeting contributions

Duchesne, I. (1998) Ultrastructure of pulp fibre surfaces. Presentation at SCA Research in Sundsvall Oct. 1998.

Daniel, G. (1998) Use of Field emission-SEM and HR-Cryo-Field-SEM in WURC for studying wood fibre structure. Fibre Morphology Club Meeting in Högsjö, April 1998.

Hult, E-L., K. Wickholm and P. T. Larsson (1998) A comparative study of the cellulose structure in spruce wood and kraft pulp. Presented at STFIs basic research program information day, Sept. 1998.

Molin, U. (1998) Fibre strength of pulp fibres. Presentation at SCA Research in Sundsvall Oct. 1998.

Other publications

Anon. (1998) Skogsforskningen har trätt in i en ny era. Teknik i Tiden, 3:1998, s.15.

Swan, B. (1997) WURC - Wood Ultrastructure Research Centre, centrum för forskning om vedfiberns ultrastruktur. Svensk Papperstidning 4:1997, p.17.

Swan, B. (1997) Mycket duktiga forskare knyts till WURC. Svensk Papperstidning nr 5:1997, p.12.

Swan, B. (1998) Stort intresse för årets WURC-seminarium. Svensk Papperstidning/Nordisk Cellulosa nr 6:1998.

10. Results and effects at the university level

From SLU's point of view, being the core of WURC, a number of examples can be given where additional positive effects have been achieved as a result of the competence centre compared to previous ways of research collaboration and thinking. The following effects are mainly a result of WURC:

- development of new and wider networks between university scientists, other research organisations and company researchers
- intensified co-operation with researchers in the companies
- a better understanding among university scientists as regards research needs in industry
- improved knowledge in general among university people about industry (companies), its organisation, problems and thinking
- PhD-students in WURC can easily establish their own industrial networks through industry people engaged in WURC
- the industry's capacity and resources within applied research, analyses and testing can be utilised
- employment of PhD:s in industry is enhanced by the close contacts
- the establishment and development of WURC resulted, on a faculty level, in allocation of more resources to WURC's field of research
- at the departmental level the involvement in WURC lead to a clear change in research focus
- WURC has contributed to the development of a more industrially oriented thinking in research within the Faculty of Forestry.

APPENDIX 1

Organisation and participants during Phase 1

Member companies

AssiDomän AB	Mo och Domsjö AB	Södra Cell AB
EKA Chemicals AB	SCA AB	
Korsnäs AB	StoraEnso AB (STORA AB)	

Organisation

Board

Lennart Eriksson, vice president, STFI, Chairman
Inger Eriksson, R&D Manager, SCA Graphic Sundsvall AB
Björn Henningsson, Professor, SLU
Håkan Jöves, Dir., Korsnäs AB
Steve Moldenius, Tech.Dir., Södra Cell AB
Sune Wännström, Dir., MoDo Paper AB
Ola Sallnäs, Professor, SLU
Yngve Stade, Man.dir. StoraEnso Research
Lars Ödberg, Professor, AssiDomän AB

Board members exchanged during the period:
Bengt Hylander, Dir., AssiDomän, 1996-97
Anders Nordstrand, Dir. MoDo AB, 1997
Tom Lindström, Professor, MoDo AB 1996

Industrial Advisory Group
Inger Eriksson, SCA Graphic, Sundsvall AB, Chairman
Anders Brolin, STORA AB (StoraEnso AB), 1996-97
Monica Ek, STORA AB (StoraEnso Research) from 1998
Ivan Dalin, EKA Chemicals AB
Stefan Högman, Korsnäs AB
Ulla Jansson, Södra Cell AB
Mikael Lindström, STFI from 1998
Ann Marklund, MoDo AB
Ove Rehnberg, AssiDomän AB
Lars Ödberg, STFI, 1996-97

Managing Director
Brita Swan, WURC (StoraEnso Research)

Managing Group
Brita Swan, WURC
Gabriella Danielsson, SLU
Björn Henningsson, SLU
Per Jennische, SLU
Thomas Nilsson, SLU

Researchers Group
Thomas Nilsson, SLU, Chairman
Paul Ander, SLU
Stig Bardage, SLU
Harald Brelid, CTH
Geoffrey Daniel, SLU
Göran Gellerstedt, KTH
Tommy Iversen, STFI
Tomas Larsson, STFI
Lennart Salmén, STFI
Rune Simonsson, CTH
Björn Sundberg, SLU
Brita Swan, WURC
Ants Teder, KTH
Tuula Teeri, KTH
Ulla Westermark, LUTH (STFI)

APPENDIX 2

Project reports

Project 1. Fibre models

Aim
The aim of this study is to generate morphological fibre models for tracheids of Norway spruce (*Picea abies* L. Karst.) including data on chemical and physical properties.

Background
Earlier generated models are out of date and do not consider new research. Therefore they are partly inaccurate and at high resolution the structure of the cell is often shown as a small indefinable area.

Project description
Literature studies and results generated within WURC by this and other projects should make possible the generation of integrated fibre models of spruce tracheids.

Initially there will be a literature survey on wood anatomy with special interest on the tracheids of spruce xylem. After that, data for macro- , micro- and ultrastructure will be collected, e.g. cell wall layers, microfibrillar orientation etc. In cases where information is lacking in the literature, LM- TEM- and SEM- studies will be carried out. Data for ultrastructure may come from studies on other conifer species than Norway spruce. Later there will be a survey to find software dealing with structure modelling. Since the other projects within WURC will contribute with their results it should be possible to generate models of high resolution.

Project report:
The project started in April 1997. A literature survey has been done concerning the micro- and ultrastructure of Norway spruce tracheids. It was found that the variation in tracheid morphology is considerable and thus the use of generalised fibre models should be avoided.

Regarding modelling, we have found that we have to utilise many techniques to optimise our object. A preliminary 3D-reconstruction of a spruce tracheid has been accomplished together with researchers at Göteborg University. A more detailed study has thereafter been initiated with the intention to accomplish a 3D-reconstruction of a whole tracheid. We have also used various CAD (Computer Aided Design) - software in order to create models. These types of software give a very generalised model but they enable powerful simulations of structures. The microfibril angle (MFA) in the S_2- layer has a profound effect on the properties of tracheids. Thus a study comparing different techniques revealing the MFA has been started in order that modelling of the MFA together with the whole tracheid may be accomplished. These techniques include: X-ray diffraction, confocal microscopy and a biological method for measuring MFA.

Project group
PhD-student: Jonas Brändström, Department of Wood Science, SLU.
Thomas Nilsson (Head supervisor), Geoffrey Daniel (Assistant supervisor), Stig Bardage, SLU. Lennart Salmén , STFI. Inger Eriksson, SCA Gunilla Söderstam, StoraEnso.

Project 2. The ultrastructure of wood fibre surfaces

Aim
The project's overall aim is to characterise the surface ultrastructure of wood fibres and investigate how this changes after various enzymatic, mechanical and chemical treatments.

Background
The surface ultrastructure of wood fibres has been insufficiently characterised previously, partly because of the unavailability of good analytical techniques and partly because of a lack of information on the native ultrastructure of wood fibres.

Project description
In the present project the surface characteristics are being studied using a variety of complementary microscopical methods including: high resolution scanning electron microscopy (FE-SEM), high resolution SEM in conjunction with cryo-techniques, transmission electron microscopy (TEM), environmental scanning electron microscopy (ESEM), and atomic force microscopy (AFM). Emphasis is placed on spruce fibres which are studied to develop a model representing a reference surface structure of softwood fibres. This reference will be then compared with standard fibres subjected to prior enzymatic, mechanical or chemical treatments.

Project report
1) During Stage 1 studies on the surface ultrastructure of WURC pulp fibres have concentrated on determining the best preparative methodology for visualising the pulp fibres. A range of conventional and rapid freezing techniques have been applied and of the methods used, rapid freezing in LN_2 followed by freeze drying (FD) or by deep etching appear to be the most suitable. Aspects from this work have been presented at various conferences and currently the work is been synthesised for journal submission. More recently a further rapid freezing technique has been utilised namely "high pressure freezing using LN_2" followed by thin replica formation at Kyoto University in connection with the stay of Prof. K.Takabe (WURC guest researcher). This material was then examined at SLU using TEM and FE-SEM. Using this technique it has been possible to verify that the *ca* 20 nm "macrofibrils" observed by us on the surface of pulp fibres using HR-cryo-FE-SEM are indeed comprised of sub-cellulose microfibrils of *ca* 3.5-5.0 nm

dimensions, results indicative for a possible reassociation of the cellulose microfibrils during the manufacture of WURC conventional and polysulphide kraft pulps. Current studies are aimed at further characterising these macrofibril elements and determine initially whether xylan is distributed on their surfaces. This work is being conducted using immuno-cytochemical labelling methods and via the use of monoclonal antibodies raised against xylan. Visualisation of the xylans on the pulp surface is being achieved by TEM and FE-SEM.

2) Studies are being conducted to try and increase our current knowledge on the morphological changes in fibre surface morphology at different stages during a conventional kraft cook (samples prepared together at KTH with Ulrika Molin). A preliminary title for the study is "Surface ultrastructure of *Picea abies* (L.) Karst pulp fibres at different stages of kraft delignification using Field-Emission-SEM". The material studied is comprised of both washed and unwashed spruce chips/pulp material. Two microscope preparation techniques have been adopted: namely rapid freezing in LN_2 slush followed by freeze drying (FD), and FE-SEM and rapid freezing followed by observations after deep etching using HR-cryo-FE-SEM. Much of the FD observations have already been conducted and some of the results were presented at the WURC meeting at STFI in November 98. The remaining studies will be completed by the end of March 1999. Currently, studies are being carried out on results of this work using image analysis (SIS) to determine whether it is possible to produce quantitative data on the surface ultrastructure of the progressively delignified spruce fibres during kraft cooking and to determine macrofibril widths and/or the total surface areas constituted by "pores" and "macrofibril" elements.

3) A review of the current literature on the surface ultrastructure of wood fibres with emphasis on spruce fibres has been carried out and accepted for publication in the Nordic Pulp and Paper Research J. The work emphasises the poor knowledge currently available on the surface morphological ultrastructure of wood fibres and reviews electron microscope techniques, past and present that are being and have been applied. During this work help was given by StoraEnso with AFM analyses of fibres.

Project group

The project group consists of Ph.D student Isabelle Duchesne, Geoffrey Daniel (Head supervisor) and Thomas Nilsson (Assistant supervisor), SLU. Bert Pettersson, STFI. Inger Eriksson, SCA and Ingrid Rokahr, StoraEnso.

Presentations/publications during WURC Stage 1

1) Daniel, G. (1997). Use of HR-Cryo-FE-SEM for studies on wood and pulp fibre ultrastructure. Presented at the International Cryo-Electron Microscopical Meeting,York, U.K, June 97.
2) Daniel, G. (1997). Studies on wood and pulp fibre ultrastructure using HR-CRYO-FE-SEM. Presented at the 1997 TAPPI Biological Sciences Symposium, San Francisco, USA, Abstract p. 447.
3) Daniel, G. and I. Duchesne (1998). Revealing the Surface Ultrastructure of Spruce Pulp Fibres using Field Emission SEM. 7th Int. Conference on

Biotechnology in the Pulp and Paper Industry, Vancouver, June, 1998 pp. 81-84.
4) Duchesne I. and G. Daniel (1999). The ultrastructure of wood fibre surfaces as shown by a variety of microscopical methods – A review. Nordic Pulp and Paper Research Journal (In press)
5) Duchesne, I. (1998). Presentation made at SCA Research in Sundsvall Oct 9th on the "Ultrastructure of pulp fibre surfaces".
6) Daniel, G. (1998). Fibre Morphology Club Meeting, Hogsjo, April 98. "Use of Field Emission-SEM and HR-Cryo-Field-SEM in WURC for studying wood fibre structure"

Project 3. Dislocations or nodes in wood fibres

Aim
The aim of this project is to study dislocations or weak points in the fibre wall and the ultrastructural background to their occurrence and impact on properties of paper fibre.

Background
It is known that greater fibre strength is obtained during pulp manufacture if the fibres are not damaged or subjected to deformation forces. After various treatments of fibres, dislocations (nodes) have been observed. They can be developed by mild swelling in a cellulose solvent (LiCl/DMAC) and occur regularly at a distance of 0.1-0.4 mm (Forgacs 1961, Wallin 1992) in many fibres. It has been observed (Ander, Daniel et al. 1996) that cellulases can locate these nodes and also preferentially attack pulp fibres at the node areas. The origin of nodes is not clear. They may have a biological origin possibly connected with ray cells. Pulp fibres tend to swell, bend and rupture at the site of the nodes and this will probably reduce paper strength.

Project description
We want to investigate the origin and composition of nodes and dislocations and how they are affected by mechanical, chemical and enzymatic treatments. It is also of interest to study how cellulolytic enzymes swell or cleave fibres and how this affects paper properties. It is important to find out how formation of strength-decreasing nodes can be avoided. The studies will be performed using light and polarized light microscopy and electron microscopy. Paper properties will be tested using conventional paper testing methods.

Project report
The project started 1998-08-10 with literature studies. It appeared soon that modern literature was scant indicating that our research will fill a great need. During the autumn we learned several techniques and gathered equipment and cellulolytic enzymes from the project group mentioned below. The enzymes obtained were endoglucanase 1 and 2 (EG 1 & 2), cellobiohydrolase 1 (CBH 1) and cellobiose dehydrogenase (CDH). K. Nyholm participated in purification of

CBH 1. Methods for measurement of enzyme activity has been studied and the so called BCA-method was found to be more sensitive than other known methods to measure microgram quantities of sugar released from pulp fibres.

EG 1 and CBH 1 have been shown to have a strong synergistic activity on the release of sugars from spruce fibres. We are presently working on a method to detect nodes in fibres using cellulase binding in combination with a staining technique. Together with MoDo differently cut spruce wood chips were prepared and cooked to kraft pulp in order to see how cutting affects frequency and structure of nodes. We are also trying to develop a method to study node cleavage with the light microscope during enzyme action.

Project group

Paul Ander, Geoffrey Daniel and Thomas Nilsson, Dept of Wood Science, SLU,
Bert Pettersson, STFI, Jerry Ståhlberg, SLU/BMC, Gunnar Henriksson, KTH
Industrial mentors: Ann Marklund, MoDo and Frank Peng, StoraEnso
Ph.D student: Karolina Nyholm, Dept of Wood Science, SLU.

Research objective and preliminary title of thesis:

Dislocations in wood fibres – their formation, characteristics and effect on paper properties.

Publications

Ander, P. et al. (1998) The in vitro ^{14}C-labelled kraft pulp assay as used for evaluation of laccases and mediators intended for pulp bleaching. 7th Intern. Conf. Biotechnology in the Pulp Paper Industry, Vancouver BC, June 16-19, 1998, Vol A, pp. A111-A115.

Conference

Ander, P (1998) Enzymes as tools for ultrastructural research on wood fibres. WURC International Seminar, Sveriges Lantbruksuniversitet, Uppsala, April 22, 1998

Project 4. Fibre chemistry: structure of cellulose and hemicellulose

Aim

The aim of the project is to elucidate important relations between structural characteristics of wood cellulose and hemicellulose and the reactivities and properties of fibre substrates.

Background

Our knowledge of cellulose structure in native wood and how it is modified during various types of industrial processing is limited. Any perspective on the factors influencing the functional properties of wood based products such as paper pulps, requires an in-depth knowledge on the structural characteristics of cellulose fibrils in the native as well as processed wood.

Project description
CP/MAS ^{13}C-NMR spectroscopy (Cross Polarisation Magic Angle Spinning Carbon-13 Nuclear Magnetic Resonance) and spectral fitting is a method well suited for investigation of the different solid state structures in cellulose substrates. The most informative region in the NMR spectra of cellulose I samples is a signal cluster with distribution between 80-92 ppm. This region contains fairly sharp signals (86-92 ppm) corresponding to C-4 carbons situated in crystalline Iα and Iβ domains together with para-crystalline cellulose. The signals from C-4 carbons in more disordered regions are distributed in a broad band ranging from 80 to 86 ppm. These signals have been assigned to cellulose at (solvent) accessible surfaces, cellulose at inaccessible fibril surfaces (interior or exterior) and to C-4 chain fragments of hemicellulose if present in the sample.

Project report
During the first part of the WURC-project a comparative study on the state of order of celluloses found in wood composed of different cell types and kraft pulp fibres prepared from Norway spruce (*Picea abies*) was carried out using CP/MAS ^{13}C-NMR spectroscopy in combination with spectral fitting.

The kraft pulp sample has a substantially higher crystallinity (24 %) compared with the pulpwood sample (9 %). The low crystallinity found in sorted normal wood was also found in wood containing different cell types i.e. juvenile wood, compression wood, early- and latewood. The difference in crystallinity between wood and pulp cellulose is explained by a conversion from inaccessible fibril surfaces in wood to Iβ and/or para-crystalline cellulose during kraft pulping.

There are substantial differences in the region typical of non-crystalline cellulose (80-86 ppm) for pulpwood and kraft pulp samples. Two additional lines are visible in the kraft pulp spectrum which are absent in the pulpwood spectrum. These two lines are attributed to hemicellulose chain units. The difference in spectral behaviour is probably related to process induced changes in the molecular structure of hemicellulose (e.g. deacetylation and removal of branch units in hemicellulose).

The increase in crystallinity due to molecular rearrangement during kraft cooking was confirmed in a study of samples taken at different time and temperatures during kraft cooking.

Project group
Tommy Iversen (project leader), Thomas Larsson (scientific advisor), Eva-Lena Hult (PhD-student) STFI Box 5604 114 86 Stockholm. Torsten Nilsson Korsnäs AB 802 55 Gävle, Monica Ek StoraEnso AB, 118 66 Stockholm, Ulla Jansson Södra Cell AB, 375 86 Mörrum

Presentations/publications etc.
1) E-L. Hult, P.T. Larsson, and T. Iversen. A comparative CP/MAS ^{13}C-NMR study of the cellulose structure in spruce wood and kraft pulp (submitted to Carbohydrate Research)

2) E-L. Hult, K. Wickholm, and P.T. Larsson. A comparative study of the cellulose structure in spruce wood and kraft pulp. Presented at STFIs basic research program information day, 22 September 1998.
3) E-L. Hult, K. Wickholm, and P.T. Larsson. A comparative study of the cellulose structure in spruce wood and kraft pulp. Presented at NUTEK's Competence Centre Day in Lund, October 20, 1998.
4) E-L. Hult. Fibre chemistry – cellulose and hemicellulose structure. Presented at the WURC seminar in Uppsala 1998
5) E-L. Hult and T. Iversen. Participation in the fifth International Scientific Workshop on Biodegradable Plastics and Polymers, June 9-13 1998, Stockholm (Sweden).
6) E-L. Hult and T. Iversen. Participation in the fifth European workshop on lignocellulosics and pulp. August 30 – September 2 1998, Aveiro (Portugal).

Project 5. Fibre strength of pulp fibres

Aim

To increase the knowledge on how changes in molecular composition, polymer structure and ultrastructure influence the strength properties of pulp fibres, and how this depends on pulping conditions. This knowledge can then be used to design chemical pulping processes that will give a better fibre.

Background

A very important aspect of the pulping process is to achieve a satisfactory level of strength properties. Much work has been done in this area, but there are still large gaps of knowledge on how changes in the fibre ultrastructure, both in the fibre wall and on the fibre surface, influence the strength properties of fibres.

Project description

One can obtain fibres with different ultrastructure from the same raw material by using different pulping methods. Fibre properties vary to a large extent with the raw material, so this variable must be constant. Variations in ultrastructure can for example be caused by chemical composition and molecular weight.

These pulps are then examined to evaluate the differences in mechanical properties and try to correlate the changes in ultrastructure. Ways to examine changes in ultrastructure are to examine changes in chemical composition, molecular weight, levelling of DP, morphology (with microscopy), crystallinity, charged groups and pore structure.

Project report

This project started in September 1997. A literature survey was carried out to determine what had been done previously in this area. It was found that in depth knowledge was missing in many areas. In the first part of the project we have examined the influence of hemicellulose and cellulose on the strength properties of fibres and paper. Hand chipped wood was used to eliminate any mechanical defects in the fibres. The cellulose/hemicellulose ratio varied between 3 to 9 through different pulping methods, while the lignin content was held constant at about 3 %. This gave a large difference in the ultrastructure of the fibre wall.

But these large differences in ultrastructure and cellulose content did not influence the fibre tensile strength, measured as zerospan. One explanation for this may be that glucomannan also contributes to the fibre tensile strength. But although the fibre tensile strength did not change, there were large differences in paper properties. These changes were proportional to the cellulose/hemicellulose content at a constant bonding degree (measured as Z-directional strength, Scott Bond and density). Large differences could be seen in tear index and tensile index. One possible explanation for these differences is that a decrease in the cellulose/hemicellulose ratio gives a stiffer, more brittle fibre.

Project group

The group consists of Ulrika Molin (PhD-student), Ants Teder (Project leader), Göran Gellerstedt, KTH. Tommy Iversen STFI. Thomas Nilsson and Geoffrey Daniel, SLU. Stefan Högman, Korsnäs, Ann Marklund, MoDo, Sture Backlund, SCA, Frank Peng, Stora Enso, and Martin Waubert-de- Puiseau, Södra.

Publications

An oral presentation at the Ekman Days 1999 "Cellulose/hemicellulose and strength" was given.

Project 6. Ultrastructural modification of wood with respect to metal ions

Aim

The aim of this project is to determine the occurrence, localisation and extractability of different metal ions in wood.

Background

The content of inorganic materials in wood is relatively low. Nevertheless, the inorganic constituents may be of great significance in many respects when wood is used for pulp production. The increasing degree of system closure in kraft pulp production induces problems caused by accumulation of non-process elements (NPE's). High levels of NPE's, e.g. Ca, Ba, K and Si, increases the scaling and the formation of deposits in the system. The presence of transition metals, such as Mn, Fe and Cu, in high-yield pulps as well as in chemical pulps, can lead to a severe decomposition of hydrogen peroxide during bleaching. Although the mode of attachment and the morphological distribution of metal ions in wood may certainly have an impact on the behaviour of different metal ions during pulp production, they have not yet been studied to any greater degree.

Project description

This project involves analyses of the metal ion distribution throughout the manifold morphological regions in different wood samples. By using metal-removing techniques such as chelation, ion exchange and acid washing, it may be possible to gain information on the extractability and mode of chemical attachment for different metal ions.

Project report

At the beginning of this project (April -97), an extensive literature study on the content, distribution and chemical bonds of metal ions in the different morphological regions of wood was performed. The literature study showed that very little has been done on the subject of extractability and chemical modification earlier. In short it can be concluded that different metals are bound with varying strength and hence may not always be extracted with the same solutions. The torus, pit membrane, middle lamella and ray parenchyma cell wall hold the largest amounts of inorganic materials.

During spring 1997, our first spruce wood samples were prepared at Chalmers and analysed at the European Synchrotron Radiation Facility (ESRF) in Grenoble. Some of the samples were pre-treated using either chelation with EDTA, acid treatment with diluted sulphuric acid or ion exchange using solutions of $MgSO_4$, $ZnSO_4$ or $CaCl_2$. This preliminary study on the possibility of using the X-ray fluorescence analysis was carried out in co-operation with Anders Rindby, Microscopy and Microanalysis, Chalmers. Wood meal was also treated with the corresponding solutions to give data on the bulk content comparative to the more specific analyses made in Grenoble.

During autumn –97, data from the first analyses were evaluated. The metals studied were Mn, Fe, Cu, Ca and Zn, and also to some extent Ba, Ni and As. The advantage of using μ-X-ray fluorescence compared to other techniques is that it is a non-destructive method, which permits analysis of the same specimen before and after chemical treatment. So far, however, resolution is limited to about 1 μm.

An article with the working-title *"Spatial Distribution of Metal Ions in Spruce Wood by Synchrotron Radiation Microbeam X-ray Fluorescence Analysis"* was submitted to Holzforschung in June 1998. It was concluded that μ-XRF is a powerful technique, suitable for further analysis on treated wood. The studies on untreated wood showed a very heterogeneous distribution of some metals (Fe, Cu, Ba, Ni, Zn), whereas others (Ca, Mn) had a more smooth distribution. After comments from the referees, the article was revised, to some extent, and re-submitted in December –98.

Further μ-XRF analyses were conducted during the summer/autumn 1998. In July, more untreated areas were studied to generate a larger statistical basis of the material. The changes following two different chemical treatments were also investigated: after an acid washing and after EDTA-treatment. In September, the potential of using XANES (X-ray Absorption Near Edge Structure) to study possible differences in the localisation of Mn of different oxidation numbers, was investigated. Certain difficulties to obtain a sufficient resolution at such low primary energy levels were detected. The data from these two analyses are currently being evaluated.

Project 7. Lignin and hemicellulose structures in wood fibres

Aim

The aim of this study is to clarify the molecular structures of lignin and hemicellulose in spruce wood (*Picea abies*) using advanced mass spectrometry.

Background

Many areas within the field of wood chemistry are dependent on access to analytical methods, which make the analysis of complex and heterogeneous systems possible. The continuous development of such methods is of great importance for obtaining more detailed chemical information of wood, pulp fibres and single wood components.

The traditional way of analysing biopolymers such as lignin has been performed using different types of wet chemical analysis. Our present knowledge about the molecular structure of lignin is mainly based on identification of monomers and dimers. These fragments have been obtained by degrading isolated lignins, e.g. milled wood lignin, using different forms of controlled and well-defined chemical degradation.

Most of the knowledge about the molecular structure of lignin as well as polysaccharides is based on work done several decades ago. The most important analytical principle for polysaccharides was hydrolysis followed by separation and identification of sugar monomers.

Project description

The intention of this project is to investigate different morphological regions in wood and to study the molecular structures of lignin and hemicelluloses in detail. Mass spectrometry will be utilised as the main tool in this work. A better knowledge of the molecular structure of these two compounds would help in understanding biosynthesis, fibrewall composition, morphology and the interaction between the different polymeric materials in the fibre wall.

Project report

This project is at present focused on the analysis of lignin. Thioacidolysis is used for degrading wood, isolated lignin samples and lignin model compounds. Preparative gel permeation chromatography is mainly used for fractionating lignin. Structure analysis is done using FT-ICR-MS, Fourier Transform-Ion Cyclotrone Resonance-Masspectrometry. This instrument uses a high magnetic field, 9.4 T, which provides a very high resolution (i.e. 10^6). Electrospray is used as the ionisation technique, which makes possible the ionisation of macromolecules. Lignin molecules with a molecular weight of up to *ca* 1500 Da have been ionised and analysed using positive ionisation. Lignin model compounds subjected to thioacidolysis have been identified using tandem mass spectrometry. The project is now focused towards gaining more knowledge of fragmentation patterns of thioacidolysed lignin, tandem masspectrometry of lignin and on improving sample preparation.

Project group

PhD-student: Hans Önnerud, M.Sc. Project leader: Göran Gellerstedt, Prof. Associate project leader: Tord Eriksson, Ph.D. Royal Institute of Technology, Pulp & Paper Chem. & Techn., Div. of Wood Chemistry

Project 8. Mechanical interactions between wood polymers and their orientation in wood structure

Aim

To obtain more knowledge on how the different wood polymers, cellulose, lignin and hemicellulose interact under mechanical loading and thereby be able to draw conclusions of how the different polymers effect the physical properties of fibres.

Background

The physical properties of wood fibres like stiffness, flexibility and swelling ability are all determined by how the different wood polymers mechanically interact in the structure. The question is the importance of each polymer to the overall properties of the fibres. There is also some uncertainty about how hemicellulose and lignin are arranged in relation to the cellulose in the cell wall. Such knowledge may lead to better control and utilisation of the properties of the fibre in the pulping process.

Project description

A new method, two-dimensional correlation spectroscopy or dynamic IR spectroscopy, will be used to study the mechanical interactions between wood polymers. In this method the sample is exposed to dynamic stress at the same time as IR spectra are recorded. The dynamic spectra give information about how different molecular groups are oriented as a result of the perturbation and how they move relative to other groups. By recording the changes in interactions between the polymers as the chemical composition of the fibre changes an understanding of the important mechanisms for the strength development in papers may be reached.

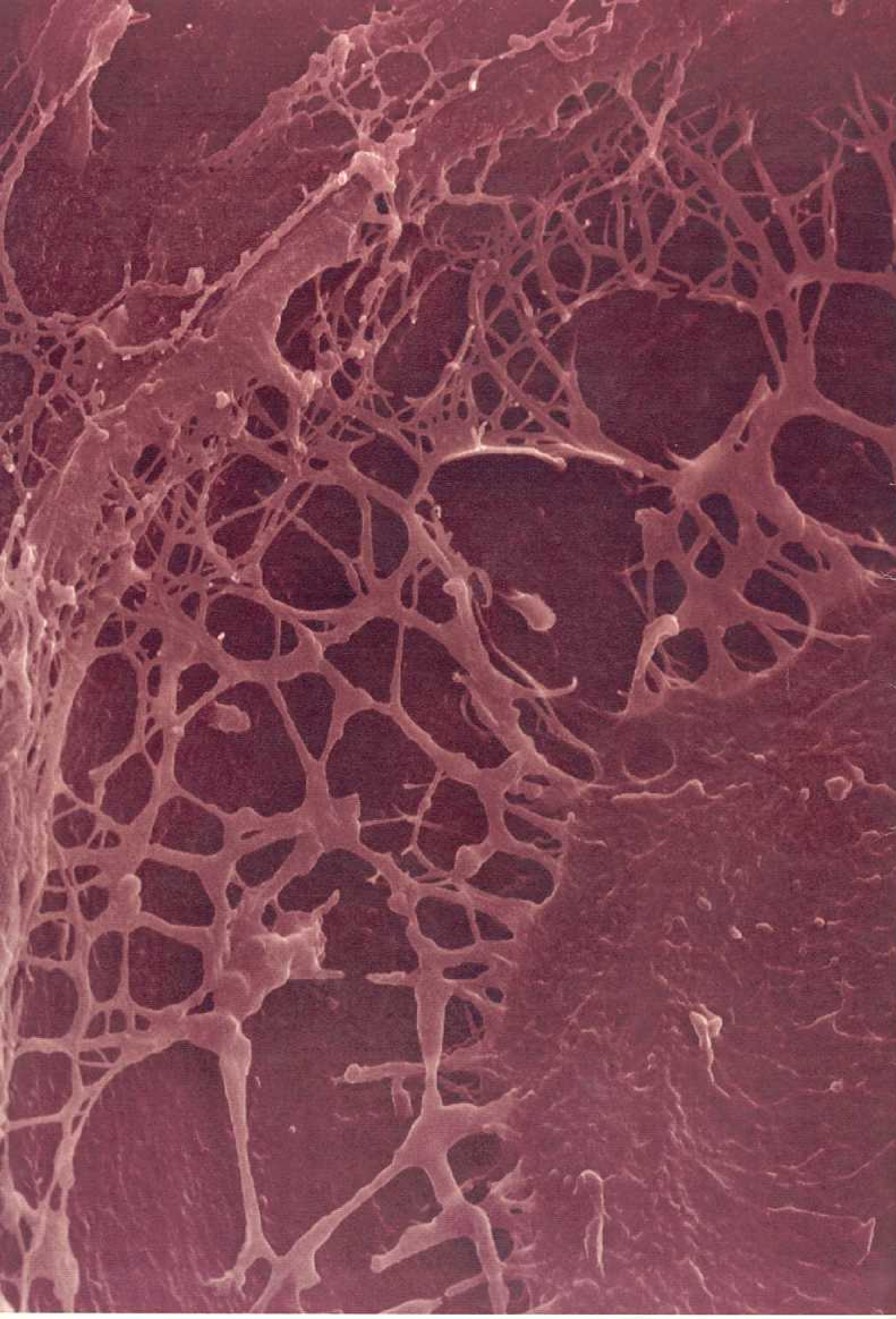
Project report

A multivariate study was performed and spectra of pure polymers were recorded in order to assign specific wave numbers for the different polymers. A series of pulps with different hemicellulose content was made for this study by chlorite delignification and alkali extractions. Xylan has specific bands corresponding to the vibrations in the acid group and glucomannan has some specific ring stretching vibration frequencies because of the equatorial hydrogens in mannose. For cellulose there are no specific groups but there are bands in the 1450 – 1300 cm^{-1} region (CH and CH_2 vibrations) which are specific for cellulose because of its crystal structure.

For dynamic measurements, a new polymer stretcher was installed in December 1998 with which it is possible to measure the stress on samples during measurement. Some dynamic spectra have been recorded but the evaluation is not yet ready.

Project group

The group consists of Lennart Salmén (project leader) and Margaretha Vickes (PhD-student), STFI, Box 5604, 114 86 Stockholm and Göran Gellerstedt, KTH, 100 44 Stockholm.



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