Workshop: Application of NIR spectroscopy for wood science and technology research

NIR & WOOD – SOUNDS GOOD!

in memoriam of

Dr Manfred Schwanninger (1963-2013)

Book of abstracts

April 15, 2014

CNR-IVALSA, Via Biasi 75, 38010 San Michele all' Adige, Italy

edited by:

Jakub and Anna Sandak

co-organized by:

Italian Society of NIR Spectroscopy (SISNIR)

National Research Council, Trees and Timber Institute (CNR-IVALSA)

COST Action FP1006 „Bringing new functions to wood through surface modification“

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Workshop: Application of NIR spectroscopy for wood science and
technology research - NIR & WOOD – SOUNDS GOOD!
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Workshop program

	Session 1 Introduction and keynote 1
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9:05	obituary: Manfred Schwanninger 1963-2013
9:10	**Keynote 1: NIR spectroscopy and the forestry value chain, *Roger Meder***
9:40	**questions**
9:45	COFFE-BREAK + equipment presentation
	Session 2 NIR & forest
10:10	NIR phenotyping in tree improvement programs, *Roger Meder*
10:25	Utilization of NIR for proper biomass conversion, *Magdalena Zborowska, Boguslawa Waliszewska, Anna Sandak, Jakub Sandak*
10:40	Biomass properties and sustainable exploitation, *Bogdan Marian Tofanica*
10:55	Relationship between NIR spectra and the geographical provenance of timber, *Anna Sandak, Jakub Sandak, Martino Negri, Bartosz Palubicki*
11:10	How near infrared spectroscopy can help estimating properties from forest to finished products, *Denilson da Silva Perez, Audrey Guillemain*
11:25	Project T4F: Designing trees for the future, *Sven-Olof Lundqvist*
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16:35	NIR assessment of chemical pattern of wood after thermal modification, *Ottaviano Allegretti, Anna Sandak, Jakub Sandak, Ales Straže, Ignazia Cuccui*
16:50	Hyperspectral imaging: a new tool for detecting and quantify mould growth on wood surfaces, *Ingunn Burud, Lone Ross Gobakken, Andreas Flø, Thomas K: Thiis*
17:05	NIR Spectral Imaging for the In-line Detection of Preservatives in Recovered Wood, *Dirk Mauruschat, Jochen Aderhold, Burkhard Plinke, Peter Meinlschmidt*
17:20	Project SWORFISH: Superb Wood Surface Finishing, *Jakub Sandak*
17:25	**round table discussions COST Action FP1006**
17:50	COFFE-BREAK + equipment presentation
18:10	discussions and closing remarks, possibilities for collaborations (Horizon 2020)
18:30	practical presentation at laboratories of IVALSA/CNR (optional)
19:00	DINNER

Preface

Italy hosted in Verona in 1999 the IX International Conference on Near Infrared Spectroscopy. The Conference is still in the memory of the attendees, not just for the magnificent frame of the location but also for the scientific contents that opened the door to, at that time, undiscovered fields.

The Organizing Committee of the Conference, chaired by Dr. Roberto Giangiacomo, decided to give voice to young or relatively young early stage scientists promoting unusual applications of Near Infrared Spectroscopy. Among them Laurie Schimleck was invited to give a talk about the application of NIR to wood, a topic which was really new for most of the NIR scientists.

Since then a great amount of papers highlighted the potential of NIR in the characterization of wood in a wide sense, up to the point that the Journal of Near Infrared Spectroscopy published two special issues in 2010 and 2011.

It's on the basis of this background that the Italian Society of NIR Infrared Spectroscopy, taking advantage of the experience gained by Jakub and Anna Sandak and their network of scientists in the field, promoted this Workshop.

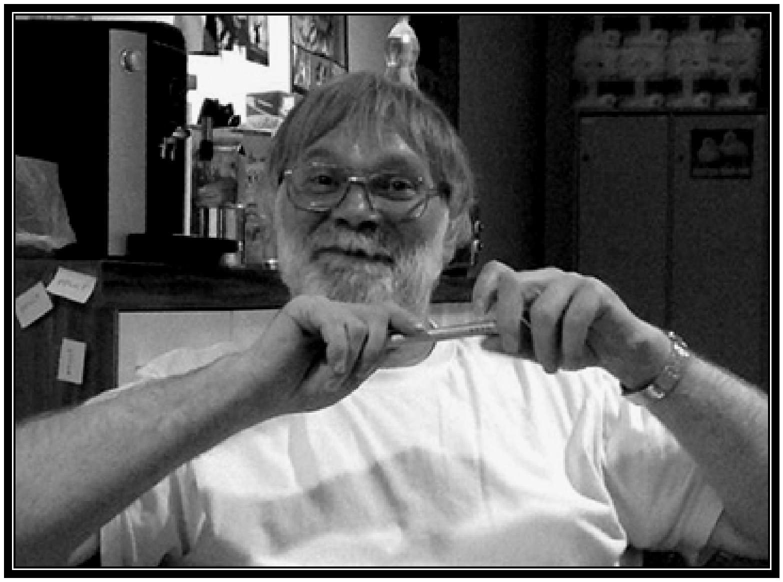
Our distinguished guest Roger Mader together with and Laurie Schimleck in the Guest editorial of the 2nd special issue remind “In the last Special Issue we raised the question as to why, when NIR of wood has been an active field of research for 20 or more years, is it that is still struggling to gain acceptance in the mainstream of wood characterization”.

It seems that the ongoing COST Action FP1006, the content of the keynotes and all the papers presented here overcome the question and NIR spectroscopy for wood science and technology research is today a reality with a scientifically recognized bright future.

SISNIR is very grateful to both keynote speakers Roger Meder and Satoru Tsuchikawa, to Jakub and Anna Sandak of CNR-IVALSA, the COST Action FP1006, and the Bruker Optics for having made possible this valuable event.

Tiziana M.P. Cattaneo

President of the Italian Society for Near Infrared Spectroscopy – SISNIR



Manfred Schwanninger (4 IX 1963–25 XII 2013)

It is with great sadness that the wood science and the NIR communities mourn the passing of Manfred Schwanninger on Christmas Day 2013 after a short illness.

Manfred was born to an innkeeper family in Upper Austria. He studied biotechnology at BOKU (Universität für Bodenkultur Wien / the University of Natural Resources and Life Sciences) in Vienna, Austria, and completed both his Master's and Doctoral theses at the Institute of Chemistry. Manfred was an exceptional student, always digging very deep into the subject matter. His doctoral thesis signified the start of his work in the field of infrared spectroscopy of wood. Since graduating he has worked as a Senior Lecturer at BOKU. Although he was employed on the teaching staff, he had a strong interest and talent for research - which he conducted after completing his teaching duties; often till late in the night and at the weekend. Very early on, he recognised the power of spectroscopy in characterising the complex heterogeneous nature of wood and other biomaterials and deepened his understanding and use of infrared techniques in particular. He became a well-known specialist in the field of infrared spectroscopy and its application to biomaterial characterization.

Many of us have had our precious manuscripts reviewed by Manfred and, as a result, our publications were always so much better. He made us think about our spectroscopy and asked us to be accurate in our observations and descriptions, just as he always was. He made us better scientists through his questioning and advice. To that end, he was always approachable: he had time for everyone from colleagues to students struggling to comprehend the nuances of spectroscopy.

He was also a prolific publisher in his own right as a result of his late-night efforts and a multitude of collaborations with scientists world-wide. He published more than 60 papers and numerous book chapters since 2001, many of them rapidly becoming highly cited. All of these papers are a must-read for anyone beginning in NIR spectroscopy of wood and even for those of us who think we know it all. We are still able to learn from Manfred.

Manfred was working up until the end. In fact he left work in such a rush one afternoon that everyone thought he was late for a meeting somewhere. Only later, they found out that he had checked himself into hospital due to a heart attack. He was placed in an induced coma and sadly did not recover, passing away on 25 December.

He will be missed by many. May he rest in peace...

We would like to dedicate the Workshop “NIR and wood: sounds good” for the memory of unforgettable Manfred Schwanninger.

His friends, including

Roger Meder, Satoru Tsuchikawa, Jakub and Anna Sandak, José Carlos Rodrigues, Barbara Hinterstoisser, Ena Smidt, Barbara Stefke, Notburga (Burgi) Gierlinger, Gilles Chaix, among others

Source

http://www.impublications.com/content/manfred-schwanninger-4-september-1963%E2%80%9325-december-2013

Acknowledgment to COST

COST is an intergovernmental framework for European Cooperation in Science and Technology, allowing the coordination of nationally-funded research on a European level. COST has a very specific mission and goal. It contributes to reducing the fragmentation in European research investments and opening the European Research Area to cooperation worldwide. As a precursor of advanced multidisciplinary research, COST plays a very important role in building a European Research Area (ERA). It anticipates and complements the activities of the EU Framework Programmes, constituting a “bridge” towards the scientific communities of emerging countries. It also increases the mobility of researchers across Europe and fosters the establishment of scientific excellence in nine key domains, with Forestry, their Products and Services (FPS) being one of these. At any given time, there are between 20 and 30 COST Actions running within the FPS Domain, each one running for 3-4 years.

The Workshop “NIR and wood: sounds good” has been fortunate to be linked with the COST Action FP1006 „Bringing new functions to wood through surface modification“. The Action has officially started its activities with the kick-off meeting on 13th of April 2011 in Brussels. The main objective of this Action is to provide the scientific-based framework and knowledge for enhanced surface modification of wood and wood products towards higher functionalization and towards fulfillment of higher technical, economic and environmental standards. Such improvement is essential for a wider and more innovative usage of wood and wood based products, also in the scope of tackling the climate changes, by lowering of usage of non-wood materials.

As part of the interaction, the following presenters and attendances have been provided with financial assistance toward travel and subsistence for their involvement at this Workshop:

- Rohumaa Anti (Finland)
- Agnès Burgers (France)
- Ingunn Burud (Norway)
- Noël Marion (Switzerland)
- Jorge Manuel Martins (Portugal)
- Dirk Mauruschat (Germany)
- Roger Meder (Australia)
- Denilson da Silva Perez (France)
- Morwenna Jane Spear (UK)
- Antikainen Toni (Finland)
- Satoru Tsuchikawa (Japan)
- Magdalena Zborowska (Poland)

Welcome from CNR-IVALSA

Our adventure on working with near infrared spectroscopy started eight years ago when a new “strange” instrument appeared in our laboratory. Being very skeptic at the beginning of using NIR we discovered that it surprisingly sees something that our eyes can never see. Indeed, infrared measures light which is “more red, than red”. After all this years we become more convinced that there is a great potential for application of NIR spectroscopy in wood science and technology. But in the same time we are fully aware for the limitations, problems and uncertainties of the state-of-the-art NIR technology when applied on wood. This is why we are more than happy to host in our institute distinguished group of researchers, engineers and scientists interesting in pushing forward NIR in to new areas and applications. It is our hope that the Workshop could improve our common understanding and inspire each other for new solutions overtaking the present obstacles. We also believe that the meeting in San Michele will be a starting point for new collaborations and common research projects.

We would like to dedicate the Workshop to the memory of Manfred Schwanninger. He has inspired us to look at our research from different perspective. The date of this meeting was proposed by Manfred; it was the only “free” time in his busy schedule. We are sure that his spirit will be with us.

We would like to thank all the speakers and participants for their support to this workshop. Their time in preparing materials for the book of abstracts and their presence at the workshop is greatly appreciated. As a result we were able to collect 17 scientific contributions covering all aspects of the forest-wood-product chain. In addition 3 presentations of the ongoing research projects where NIR is used for studying wood are integrated to the Workshop program. Very important deliverable of the workshop is a list of problems/challenges of NIR&wood as pointed in each contribution.

We would like to thank:

- SISNIR, especially Tiziana M.P. Cattaneo and Roberto Giangiacomo for the initiative to organize the NIR workshop on wood and for trust in us
- our sponsor BRUKER, especially Francesca Bertolani, Paolo Belloni and Diego Sali for financial support
- those who helped per-review and edit this book of abstracts, particularly Sven-Olof Lundqvist and Roger Meder
- COST Action FP1006, especially Stefanie Wieland and Ingrid Seidl for inspiration, support in organization, encouragement and providing travel grants
- our colleagues from CNR-IVALSA, who helped us to organize this event, especially Maria Litrenta, Alessandra Zecca, Marco Fellin and Maximiliano Natale
- Provincia Autonoma di Trento for continuous support and books for our keynote speakers

We are particularly grateful to both keynote speakers, Roger Meder and Satoru Tsuchikawa for their kind acceptance of our invitation, understanding and taking place of Manfred Schwanninger. It is our honor to host you and to learn from you.

We are wishing you a very inspiring, intellectual and fruitful Workshop. Please enjoy CNR-IVALSA and welcome you to visit us again.

Jakub and Anna Sandak

Local organizers

List of contents

Session 1

Introduction and keynotes

Keynote 1: NIR spectroscopy and the forestry value chain

Roger Meder

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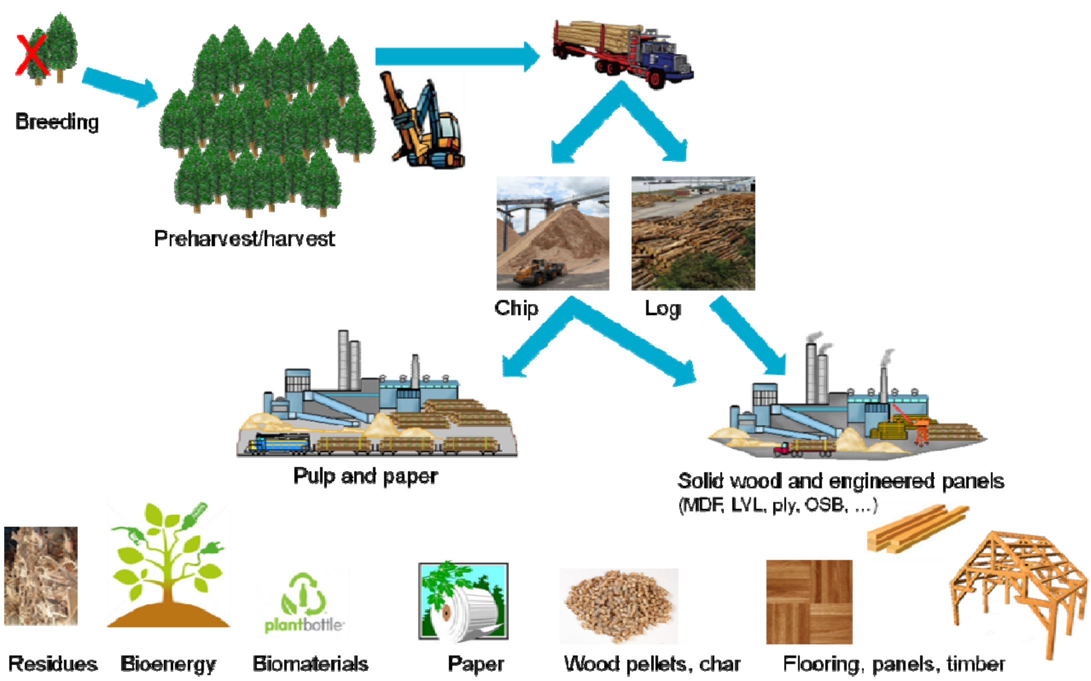
In the 25 years since Birkett and Gambino [1] first used NIR spectroscopy to determine pulp Kappa number, there has been a steady growth in the range of application of NIR throughout the forestry value chain. Much of the early effort focused on pulping applications, primarily due to the sampling requirement of the day requiring the ground wood or ground pulp samples being packed into sample cups. The advent of fibre optic probes changed that: solid wood and on-line applications became viable although it has taken time for even those applications to emerge. Today though, portable and handheld systems mean that the NIR spectrometer can now be taken to the sample – a standing tree out in the forest! This means that the range of applications extends from assessment of seedlings in the glasshouse to performance measurement of final product – solid or engineered products or pulp and paper and now the quality control of bioproducts.

This paper will highlight application of NIR at a number of intervention points along the value chain, including some applications that don't work. It will also highlight the technology transfer gap between incubation of applications and the uptake of those applications by industry. Vince Hurley, CEO of Australian Sustainable Hardwoods said that "It is up to industry to make the most of research findings", but there is also a role of researchers to seek out the innovative industry partners that are looking for the next technological breakthrough in optimizing their manufacturing process – from feedstock control to quality control of final product.



Questions remain:

- Where is the best value to be gained in using NIR?
- Is there value in preharvest assessment?
- How do we as researchers promote our technology to industry partners?

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Keynote 2: Application of NIR spectroscopy to wood science and technology - recent topics due to on-line and at-line technique

<u>Satoru Tsuchikawa</u>

Graduate School of Bioagricultural Sciences, Nagoya University, Japan

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Near-infrared (NIR) technology, in terms of transmitted or reflected spectra of electromagnetic waves ranging from 800 to 2500 nm (12,500 to 4000 cm^{-1}), has mainly been used for the nondestructive measurement of organic materials such as agricultural products or foods. However, it shows great potential in all facets of material assessment (for example, polymers, textiles, pharmaceuticals, petrochemicals, etc.). It has been applied in the pulp and paper industry to monitor the moisture content or basic weight under on-line conditions. In the case of wood or forest products, NIR spectroscopy is widely used in a state where not only the cellular structure but also its bulky shape is retained. It is a promising technique for analyzing the physical state of such materials as well as the chemical composition. In this presentation, some applications of NIR spectroscopy due to the nondestructive assessment of wood products are introduced.

Our research group newly designed NIR spectrophotometer with linear image sensor for high speed acquisition of the NIR spectra in fast moving wood scenarios. The feasibility of NIR spectra to predict moisture content (MC) and modulus of elasticity (MOE) was also investigated.

We have designed a novel densitometer that consists of a continuous wave NIR laser source and an avalanche photodiode module as the detector, which can rapidly and non-destructively measure the density of wood. By conducting a validity evaluation with statistical coefficients, it was shown that the constructed system is as accurate as a conventional x-ray densitometer.

Session 2

NIR & forest

NIR phenotyping in tree improvement programs

Roger Meder

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The advent of portable NIR systems has changed the perception of industry in Australia to the use of NIR in tree breeding. But has it changed for the good?

Much of the development of NIR applications in Australia and New Zealand over the last 20 years has focused on the prediction of Kraft pulp yield of either pines or eucalypts [1,2] to the point where today a single multi-species, multi-site global calibration exists for eucalypts, acacias and corymbias throughout the Southern hemisphere [3].

How can NIR be used in a tree breeding program?

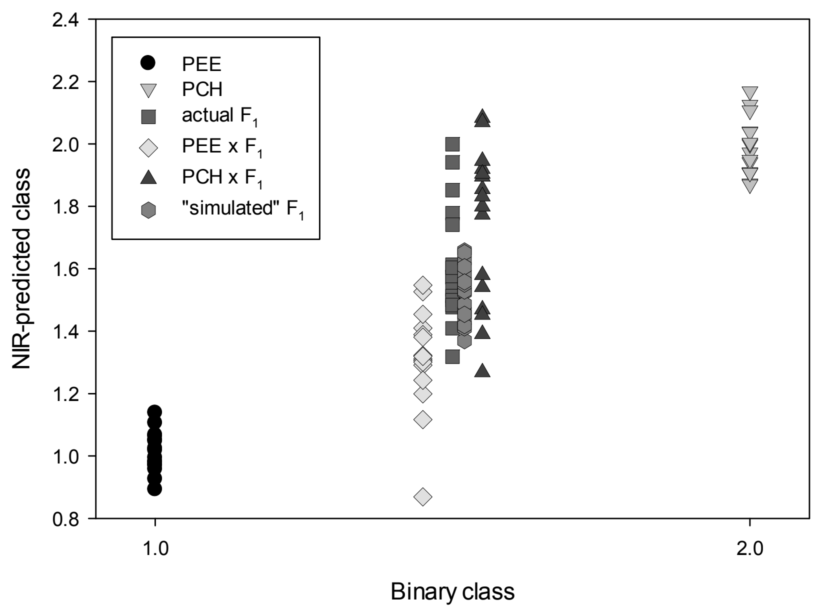

Figure 1. Plot of PLS-DA prediction of pure PEE and PCH along with simulated mixed F1, actual hybrid F1 samples and backcrosses of the F1 hybrid with either parent.

Confirmation of hybridization [4]

Foliage of Pinus elliottii var elliottii (PEE), Pinus caribaea var hondurensis (PCH), their PEExPCH F1 hybrid and the F1 backcrosses [PEEx(PEExPCH) or (PEExPCH)xPCH] was collected, dried, ground and NIR spectra acquired. In addition to the natural F1 hybrid, simulated hybrids of known parents were prepared by mixing equal parts of the two parent foliage samples. The trial from which the foliage samples were collected was established in Toolara, Queensland (26° 5’ S, 152° 50’ E) in 1987 [5,6]. Partial least squares discriminant analysis (PLS-DA) of the NIR spectra, shown in Figure 1, showed clear segregation between the PEE (1) and PCH (2) parents with the natural hybrid and simulated hybrid placed between the two parents, although considerably more spread can be seen for the actual hybrid due to genetic variance introduced in the hybridization process – something that cannot be simulated by simple mixing. The PEExF1 backcross shows a stronger additive genetic effect than the PCHxF1 backcross.

Rapid Phenotyping for Selection

The strength of NIR is its non-destructive and high-throughput capability. This is particularly useful for screening of breeding populations. Classical pulping of wood chip samples taken from trees is a costly (~US$500 – US$800/sample) and slow process (typical throughput is 6 – 10 samples per day). In addition it requires the destructive felling of the individual tree in order to obtain the 10 kg (fresh weight) of chips required for laboratory pulping. The consequence of this is that KPY is not routinely measured as a part of breeding selection program. Instead density is used as a surrogate for pulp yield.

Breeding trials often involve multiple families/provenances/progeny and multiple replicates of each. This means that a breeding trial with 100 families, 4 trees per family per replicate and 5 replicates at each site for 4 different sites will have 8,000 individual trees. The task of measuring and phenotyping this number of individuals is considerable, and for reasons given above it would be impossible to determine pulp yield traits for the individuals. However, the non-destructive prediction of KPY in standing trees using NIR can be performed by removing half or full diameter increment cores from candidate trees in the forest and returning the samples to the laboratory for analysis using laboratory-based, bench-top systems [7-9]. By taking breast height increment cores it is now possible to non-destructively predict pulp yield for each tree using NIR spectra obtained from the ground core. The power of this is shown in Figure 2. This shows that for an assumed trial design of 40 progeny assessed for DBH, a pulp yield calibration model with an R^2 of 0.81 (i.e. correlation coefficient, r = 0.90) indicates that NIR spectra from a minimum of two trees are needed to obtain a greater estimate of genetic gain than that which would be achieved with one laboratory-pulped tree.

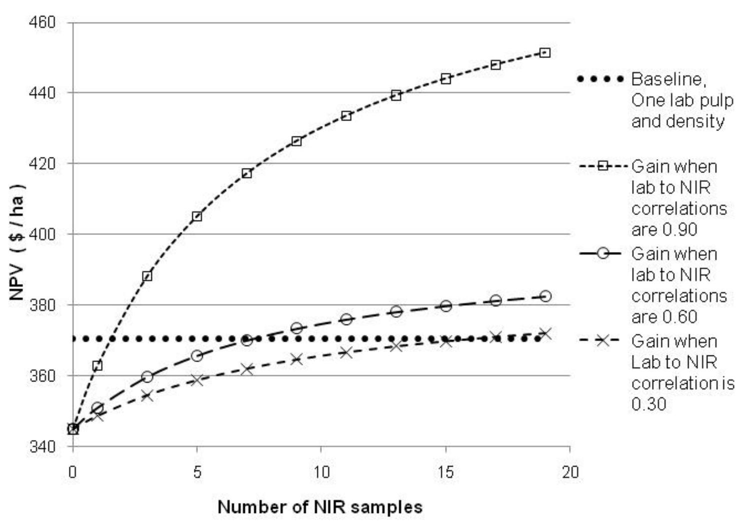

Figure 2. Economic value of genetic gain from increasing NIR sampling with phenotypic and genetic correlations between laboratory estimates and NIR predictions varying from *r* = 0.30 to 0.90.

Problems and Issues

The greatest problem associated with using NIR spectroscopy to predict properties of trees (or logs or boards) is the inherent heterogeneity of wood. Within a tree there is large radial, longitudinal and circumferential variation in all properties, including pulp yield [10]. In addition to this there is also seasonal variation (essentially radial variation). Figure 3 shows pulp yield calibrations for trees sampled in spring and in autumn (fall) using a handheld device to acquire spectra directly under the bark at breast height.

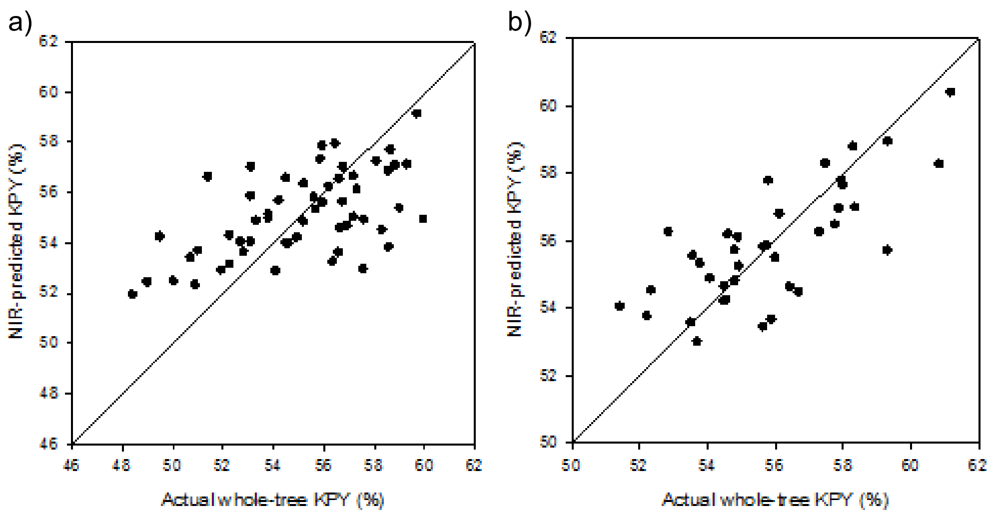

Figure 3. Predicted vs measured plots of pulp yield for 40 trees sampled using a handheld device directly under the bark in (a) spring, R^2 = 0.37 and (b) autumn (fall), R^2 = 0.56.

But do we need to instantly know Kraft pulp yield for an individual tree in the forest?

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Utilization of NIR for proper biomass conversion

Magdalena Zborowska[1], Boguslawa Waliszewska[1], Anna Sandak[2], Jakub Sandak[2]

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Biomass, typically obtained from fast growing plants or over-stocked agricultural products, has been widely recognized as a replacement for traditional energy sources. Several rapidly growing tree species are considered suitable for short rotation forestry with willow (*Salix* sp.) being one of the most promising [1-3].

The goal of this research was to explore the potential of the near infrared spectroscopy (NIR) to evaluate the chemical composition of several willow clones. Partial Least Squares (PLS) regression models for quantitative prediction of structural wood components, extractives, and High Heating Value (HHV) were developed. Figure 1 presents the prediction capability of the HHV as estimated on the base of NIR spectra measured on willow chips. The reference values were obtained from the twin samples characterized in bomb calorimeter. It was possible to discriminate different willow clones by using NIR spectroscopy, and to assign these into groups/clusters by means of Principal Components Analysis (PCA). Moreover, detailed analysis of principal components based on NIR spectra provided some explanation for the differentiation among clones. It was shown that NIR spectroscopy can be an alternative technique to standard analytical methods supporting research and development of biomass production technologies [1].

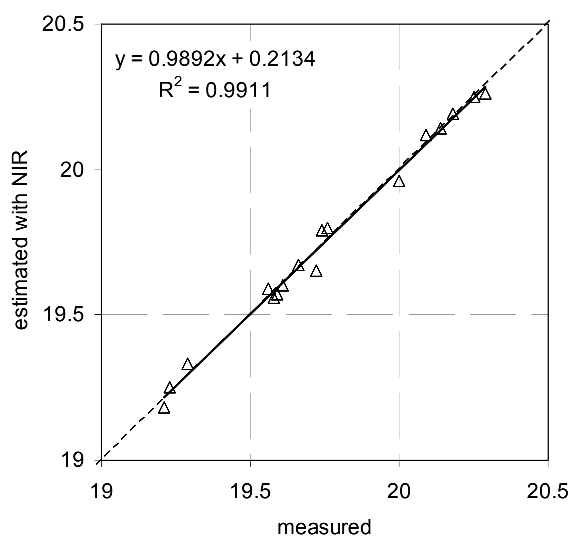

Figure 1. PLS model for prediction of HHV (R^2=0.99, *RMSECV*=0.0313, *RPD*=10.8, rank 5, preprocessing: 2nd derivative, range 9400-7500cm^{-1})

Problems:

- Specific adaptation of willow clones to environmental sites affect chemical composition of biomass and in consequence previously developed PLS models might be not accurate
- The typical sample of biomass is composed of mixture of wood, bark and other tree parts
- High number of measurements (both NIR spectra and reference values) is necessary to provide reliable prediction models

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Biomass properties and sustainable exploitation

Bogdan Marian Tofanica

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Biomass resources include a wide variety of raw materials diverse in morphological, anatomical, structural, chemical and physical properties that vary significantly within and between the different raw materials. Understanding these properties is essential and depending on their application, these variations may be critical for their final performance.

To increase the share of biomass in a biobased economy, a greater importance on local biomass resources may come from wood waste streams, agricultural wastes, municipal solid wastes and manufacturing wastes.

For this, two fundamental aspects related to biomass use are: (1) to enlarge and improve the basic knowledge on biomass availability, composition and properties; (2) to apply this knowledge for the most advanced and environmentally safe utilization.

For exploiting its whole potential in a bio-based economy, biomass can provide food, materials, chemicals and/or energy, using various transformation processes such as combustion, gasification, fermentation, liquefaction, pyrolysis. These conversion methods use chemical, thermal and/or biological processes.

The identification of (1) anatomical characteristics; (2) chemical composition (cellulose, lignin, hemicelluloses, extractives and inorganic content); (3) chemical characteristics (carbon, hydrogen, oxygen, nitrogen, sulfur, and other elements content); (4) physical parameters (density, moisture); (5) proximate analysis data (fixed carbon, volatile compounds and ash content); and (6) energetic parameters (higher heat value, lower heat value) of biomass is the initial and most important step during the investigation and application as a raw material.

Each biomass resource has different physical and chemical characteristics in terms of calorific value, moisture and ash content, etc. that requires appropriate conversion technologies for efficient utilization. The route to products depends on the biomass characteristics. Therefore, an important step in the conversion systems is the evaluation of the biomass availability and its energetic, physical and chemical characteristics.

These may have potential industrial implications in the field of technologies and processes for electrical and thermal energy generation, for future advanced and sustainable processing of biomass to biofuels and chemical feedstock, including collection, transportation, storage, upgrading, digestion, combustion, gasification, pyrolysis and liquefaction.

These properties determine biomass quality and quantity, potential applications and environmental problems related to its use for particular purposes, but also can be used for characterization and future advanced sustainable exploitation of biomass as a large-scale resource in a bio-based economy.

In order to understand the biomass potential it is of vital importance to gain better knowledge of biomass properties, which may provide decisive factors not only for its applicability, but also for the economic practicability and environmental security of many processes involving biomass.

Relationship between NIR spectra and the geographical provenance of timber

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Several wood characteristics are related to environmental and/or genetic factors. Even though, it has always been considered a difficult task to identify the origin of a timber and no effective tools are presently available for this purpose. Currently used Programme for the Endorsement of Forest Certification (PEFC) system to track the wood flow from the forest to the mill is based only on written documentation. Presented research is a trail to develop fast, non-destructive and reliable verification procedure allowing tracking of wooden biomass origin [1-4].

Norway spruce is an important wood species growing in large areas of forests all around Europe. It is primarily raw resource for the wood and paper industries. Wood samples of Norway spruce (Picea abies Karst.), originated from 75 different locations of 14 countries in Europe, were collected in order to verify, if it is possible to categorize NIR spectra of woods coming from noticeably different origins of various geographical locations, climates, altitudes and soils. 2163 samples were collected and analyzed during the project.

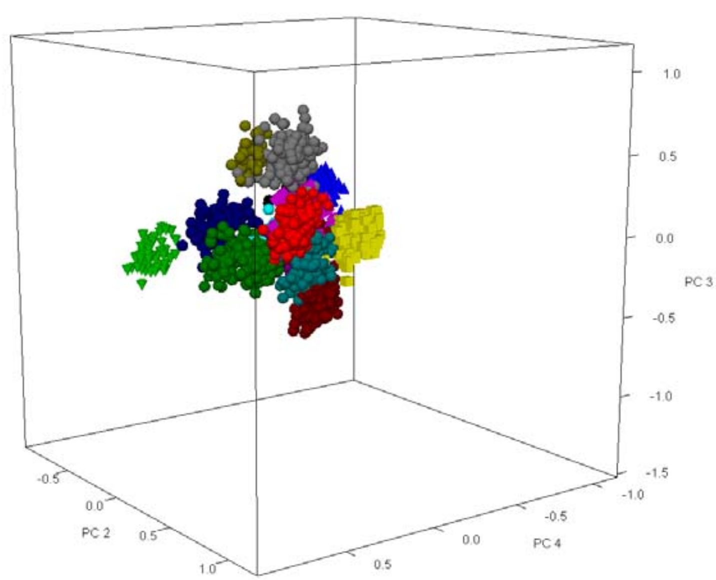

Figure 1. PCA of fresh wood obtained from samples of different European provenance

● Austria ▲ Bulgaria ▼ Czech Republic ■ Croatia ■ Estonia ● Finland
● France ● Germany ● Hungary ● Italy ● Norway ● Poland ● Romania
● Sweden

Figure 1 present principle component analysis (PCA) performed on 1822 spectra as measured on spruce samples from 14 different locations. Only one location from each country was selected in order to simplify visualization and highlight differentiations. Grouped clouds of points (each color represents one location) are presented on the chart. Some locations are clearly separated from others, such as Hungary, Bulgaria and Austria. Other clouds contain spectra which might be confused with other groups or identified not uniquely. More than 60% of spectra can be uniquely identified by means of PCA, according to the validation protocol.

It was concluded that trees growing in various locations have some differences in the chemical composition and NIR spectroscopy is sensitive enough to detect such differences. However several factors such as sample preparation, measurement condition or age of samples have and effect of obtained results [1].

Problems influencing reliability of the method:

- proper sampling procedure
- proper scanning procedure
- dealing with multivariate data with very high number of measurements
- effect of time: storage, aging, oxidation
- quantification/differentiation of the contribution(s) of natural wood variability, growth conditions, genetics and provenance

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How near infrared spectroscopy can help estimating properties from forest to finished products

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Introduction

The interest in near infrared spectroscopy as a tool for rapid characterisation of raw material and products as well as for process control systems in the forest products industry has considerably increased in the last years. The main advantage of this analytical tool is the possibility of determining both chemical and physical properties of a large number of samples by a fast, non-destructive and precise spectroscopic technique.

In view of pulp and paper production, trees should have wood containing low lignin and extractives content and high polysaccharide (and especially high cellulose) content, high density, long and coarse fibres with high proportion of earlywood fibres, and should give rise to pulps with good burst, tear, and tensile strength [1].

Different chemical (Kappa number, viscosity, chemical composition, etc), physical (bulk, density, strength properties, etc), optical (brightness, colour components, and morphological (average fibres length and width, coarseness, fines content, etc) properties are measured in different steps of the wood-to-pulp transformation chain.

The traditional analytical methods employed for wood, fibre, and pulp evaluation are usually time-consuming and expensive. Rapid and non-destructive analysis methods, such as near infrared (NIR) or Raman spectroscopy, provide an opportunity to shorten analysis time, elevate the throughput, and lower the costs. Schimleck and Workman [2] and Tsuchikawa [3]) demonstrated the analytical power of NIRS for the rapid quality assessment of wood, fibres, pulp, and paper.

In this article, the use of NIR spectroscopy for the rapid prediction of wood quality, including pulp characteristics, from the wood spectra is presented.

Materials and methods

NIR spectra were recorded using a Bruker Tensor 37 spectrometer for most of the calibrations, except for the work concerning the prediction of pulp properties from wood spectra combined with microdensimetry profile. In this later, a BIORAD Excalibur 3500 spectrometer was used. The principle of property predictions is based on a calibration correlating spectroscopic data to the laboratory measurements using PLS-based methods implemented in OPUS software furnished by Bruker with the NIR spectrometer. Details of NIS spectra acquisition and pre-treatments, PLS calibrations generation and validations are presented in previous work [4,5]).

Results and discussions

One example of using NIRS to predict elite trees for breeding programs based on pulp and paper traits was developed in the framework of European GEMINI1. In this work, NIRS calibrations for the prediction of the chemical components of maritime pine were produced. Maritime pine extractives ask for a combination of non-polar and polar solvents for a complete extraction. A sequence of solvents dichloromethane–ethanol–water (DEW) seemed well suited to this purpose. The total extractives obtained by DEW extraction were calibrated by NIR in combination with PLS models. In addition, Klason lignin, cellulose, hemicelluloses, the content of individual monosaccharides and lignin composition concerning the ratio of the basic units 4-hydroxy-phenylpropane/guaiacylpropane (H/G ratio) were also predicted by NIRS calibrations.

Calibration data of Table 1 show that all components can be predicted by NIRS with high accuracy and precision. Good results have been obtained for most wood components as indicated by statistical data (R^2 and root-mean-square error, *RMSECV*).

Table 1. Cross validation performances of the NIRS calibrations for prediction of chemical composition of maritime pine wood. Range data are reported to the non-extracted wood o.d.w. basis, except the calibration for the prediction of H/G lignin ratio.

Properties	Number of samples	Range		Rank	Calibration		Validation	
		Min.	Max.		R^2	*RMSEC*	R^2	*RMSECV*
Extractives content								
Dichloromethane	79	0.4%	3.0%	5	0.96	0.15	0.95	0.17
Ethanol	80	1.2%	2.4%	8	0.79	0.13	0.73	0.15
Water	80	1.4%	2.6%	5	0.88	0.11	0.84	0.12
Total	77	2.3%	7.1%	7	0.95	0.28	0.92	0.34
Lignin content	67	25.8%	32.7%	3	0.97	0.36	0.96	0.40
Cellulose content	67	39.9%	51.1%	2	0.93	0.77	0.92	0.80
Hemicelluloses content	68	23.3%	28.4%	4	0.77	0.54	0.71	0.59
Monosaccharide contents								
Mannose	62	14.5%	20.0%	1	0.89	0.53	0.87	0.55
Galactose	64	2.0%	10.9%	7	0.98	0.51	0.94	0.72
Xylose	67	9.1%	13.9%	5	0.77	0.41	0.63	0.50
Lignin quality								
Ratio of H/G units	62	0.041	0.111	2	0.90	0.005	0.89	0.005

The second approach aimed at investigating the possibility of predicting the strength properties of Kraft pulp (upon fixed conditions of cooking and pulp refining) from the wood NIR spectra coupled with microdensitometry (MDM) profiles [6]. Different possibilities of combining MDM and NIRS were tested to improve the quality of properties prediction. The best way of connecting them was to use the V500 parameter (% of profile below or above 500 kg/m²) as a first descriptor of wood quality to split the samples and then to use specific calibrations for each range to predict the paper properties. NIRS calibrations for the prediction of pulp physical properties (except for bulk that can be directly predicted from V500) were established for the whole set of samples and for the separated classes, in order to compare the potential of the association (MDM-NIRS). The calibrations statistical data are compiled in Table 2.

Sample extraction was a crucial aspect for rapid assessment of pulp properties. The quality of prediction obtained from non-extracted samples is obviously lower if compared to the extracted ones. However, the combination of NIRS and MDM gave a prediction of pulp physical properties from non-extracted samples of an acceptable quality. Coupling MDM and

NIRS for the measurements on non-extracted samples revealed to be the best way to improve the prediction of wood properties quality while saving measurement time.

Table 2. Summary of NIRS calibrations obtained for the prediction of pulp physical properties from wood spectra, as function of extraction and V500 parameter.

Property	Extraction	Samples	Range		Calibrations results		
			Min	Max	R^2	*RSD*	Error of prediction
Breaking length	***Extracted***	Whole set	7.9	10.7	0.822	0.296	0.510
		V500 <32	8.2	10.2	0.966	0.132	0.397
		V500 > 32	8.2	10.7	0.930	0.218	0.366
	Non-extracted	Whole set	7.9	10.7	0.711	0.356	0.490
		V500 <32	8.2	10.2	0.916	0.242	0.361
		V500 > 32	8.2	10.7	0.969	0.126	0.452
Burst index	***Extracted***	Whole set	5.4	7.7	0.858	0.223	0.378
		V500 <32	5.4	7.0	0.988	0.065	0.135
		V500 > 32	5.4	7.7	0.951	0.132	0.306
	Non-extracted	Whole set	5.4	7.7	0.706	0.264	0.341
		V500 <32	5.4	7.0	0.981	0.083	0.149
		V500 > 32	5.4	7.7	0.926	0.158	0.247
Tear index	***Extracted***	Whole set	5.9	9.9	0.924	0.342	0.460
		V500 <32	No calibration obtained				
		V500 > 32	No calibration obtained				
	Non-extracted	Whole set	5.9	9.9	0.873	0.497	0.539
		V500 <32	6.9	9.9	0.941	0.283	0.404
		V500 > 32	5.9	9.5	0.955	0.238	0.568

Conclusions

NIR spectroscopy is a powerful tool for predicting properties from forest to finished products. Good calibrations (high R^2 and low error or prediction) were observed for the content of different classes of extractives, lignin, cellulose, and hemicelluloses.

Microdensitometry and NIRS can be used together to improve the quality of the prediction of pulp physical properties from wood measurements. NIRS alone can be used for the estimation of pulp strength parameters from wood spectra, but a step of extraction of sawdust is compulsory. By introducing the V500 (portion of MDM profile higher than 500 kg/m3) for the particular case of maritime pine as a pre-screener and using separate NIRS calibrations for each V500class, a considerable improvement of estimation quality was obtained, even for non-extracted samples.

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Project T4F: Designing trees for the future

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Aim of Trees4Future

Trees4Future is an Integrative European Research Infrastructure project. The overall aim is to help the forest-based sector to respond to the increasing demands for wood products and services in a context of genetic adaptation and changing climatic conditions, sustainability and competition. The project will strengthen the European research infrastructure and it will increase our knowledge about the adaptation of forests to climate change and tree characteristics suited for tailor-made wood supply, thus optimizing the short and long-term exploitation of forest resources.

For this purpose, 28 European research organizations with major research infrastructures in forest genetics and forestry research have joined forces. The infrastructures are integrated and improved. Experiences are shared. New functionalities and tools are developed.

A core part of the project is its Transnational Access program. Within this, also the wider research community and companies can gain access to a large number of these infrastructures, with funding from the EU. The project is now up and running since early 2012.

Wide range of expertise

The project partners represent a wide range of expertise: from genetic markers to measurement of product related properties, from the tree/population scale to the forestry landscape scale. This is reflected also in the wide spectrum of research infrastructures involved; see the map in Figure 1, which also indicates the nature of the facilities.

The further improvement of these infrastructures involves the development of:

- A user-friendly analytical platform for statistical and genetic data analysis
- A platform for molecular analysis, with genetic markers and standardized laboratory protocols
- A cross-European tool to identify what trees to plant to match future environmental conditions
- Compilation of data from national and EU environmental and genetic databases, plots and resources
- Integrated compatible modeling tools for prediction of forest wood resources and services

- Efficient methods for characterization of wood, emphasizing tree adaptation and wood properties

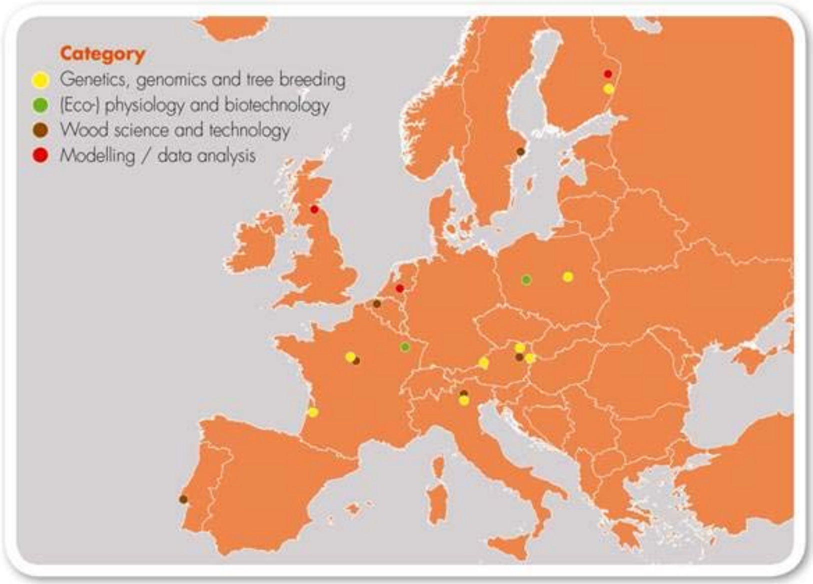

Figure 1. Locations of the infrastructures given access to: Genetic databanks, biobanks, laboratories, models and decision-support systems, useful in a wide spectrum of research fields.

Free access to research facilities

As part of the Transnational Access program, both the forest research community and industries related to the forest-wood chain based in Europe can gain access to a wide range of specialized forest research infrastructures, made available by the project partners. 28 state-of-the-art facilities are available within genetics and genomics, tree breeding, wood technology and modeling/data analysis. They include genetic databanks, biobanks, laboratories, models and decision-support systems. Researchers and other experts may apply for access according to the offers of the partners. The selected research groups will get their access costs covered by the EU.

Trees4Future in brief:

- Theme: Research infrastructures for forestry research
- Duration: 4 years
- Budget: 9M€
- Funder: EU 7th Framework Program (FP7)
- Partners: 28 organizations

References & more info

http://www.trees4future.eu/

Session 3

NIR & timber

Imaging NIR spectroscopy for investigation of wood and applications on wood materials

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Radiation of different wavelengths in the near infrared (NIR) range interacts with different chemical bonds. Therefore, NIR absorption spectra may provide information about the chemical composition of materials and related features. To estimate the contents of chemical compounds or other properties from the NIR spectra, models are needed. The models are often developed using multivariate statistical methods applied on sets of spectra and data from analyses with reference methods from the same samples. Spectra can be exploited also without reference data, for instance with modelling methods for classification of different types of objects and through exploration of spectral variations. NIR spectroscopy is used for characterization of materials and objects in a multitude of applications on various types of materials: On wood and materials from wood, in the food, medicine and chemical industries, in agriculture and mining, etc.

Most common, a NIR spectrum is recorded from a single area, small or large, providing information about average properties within the area at the time. Using hyperspectral cameras, many spectra may be collected simultaneously along a line. By moving the sample, its surface may be scanned line by line to provide an image. With imaging NIR spectroscopy, property variations over 2-dimensional surfaces may be explored, which is now used for research and applications on various materials.

For determination of chemical concentrations, prediction models are needed. With imaging spectroscopy it is however also possible to perform many types of tasks even without such models. Images of principal components offer an overview of the patterns of the main spectral variation. Occurrence of variations and their character may be established, providing a basis for how to proceed in research and development. The principal component variation can be compared with the physical samples. This way, physical features which are otherwise hard to detect may be related to spectral features, providing good results even without prediction models, for instance for classification of species, and identification of features in wood.

At Innventia, research and development is performed on many types of materials, many of them originating from wood; research projects with universities as well as development projects for forestry and industry. New measurement methods and efficient material characterization are fundaments in our work on wood, its utilization and processing. After tests on wood, paper and other materials, a new flexible research instrument for imaging NIR spectroscopy has been specified and produced. It now serves as a research tool and as a development platform for measurement methods and applications. The spatial resolution may be varied from pixels of 1.3 mm x 1.3 mm over a width of 40 cm to 30 μm x 30 μm over a width of 10 mm over infinite length.

Measurements on wood in different scales are illustrated in Figure 1 and Figure 2. The sample in figure 1 is 130 mm long and 10 mm broad. It was scanned in 42 seconds, with settings to provide a spatial resolution (pixel size) of 31 μm x 31 μm, resulting in 1.34 million spectra from the sample.

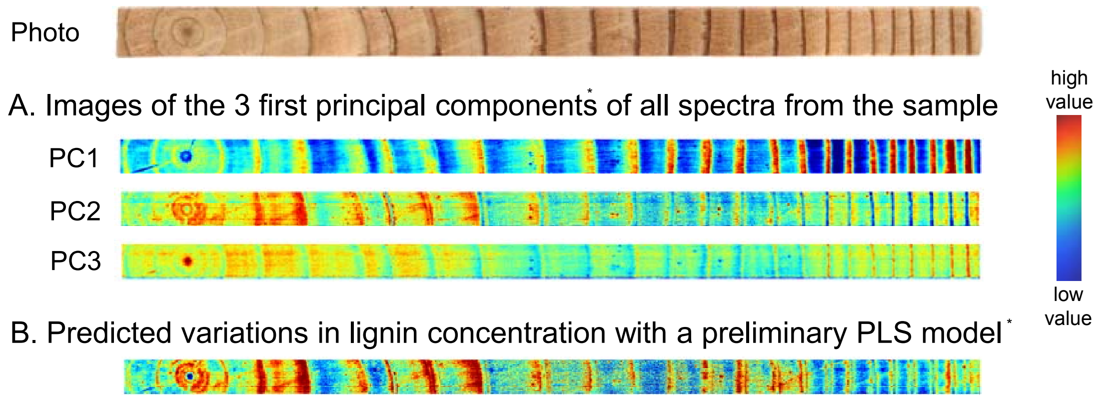

Figure 1. NIR imaging of a wood sample for detailed analysis of property variations within and between growth rings, as well as differences among trees of various origins: Photo of the sample; A: The 3 first principal components; B. Lignin concentration estimated with preliminary model. Pixel size 30 µm x 30 µm

Figure 2 shows a similar exercise on a fresh wood disc cut from a Scots pine. It had lost its bark on the side to the right, resulting in local drying and deterioration in the sapwood. The dot is a stain of chain oil.

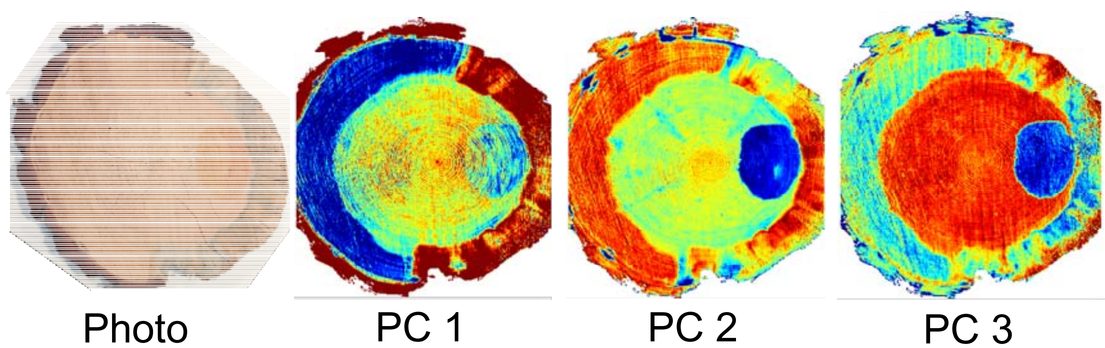

Figure 2. NIR imaging of a disc of diameter about 40 cm, cut from a Scots pine tree. Pixel size 1 mm x 1 mm

The starting point of this work was a need within the Swedish project Bio4Energy. Within this project, Innventia is managing the building of a database with information about a multitude of properties of 6000 trees of Norway spruce, down to the detail of variations between and within individual annual rings. The data will among other things be used for development of genetic markers for tree improvement. A small fraction of the increment cores sampled from the trees are shown in Figure 3. During the last years, Innventia has produced high precision sample strips from pith to bark from all the 6000 samples, and analyzed them for many wood and fibre properties with its SilviScan instrument. The ambition is to add analogous information about the chemical composition of wood to these data. The new imaging NIR system will be used to scan these samples, providing a spectrum for the wavelength interval 1000-2500 nm from each pixel. Models are now being developed to estimate radial variations in content of lignin, cellulose, hemicelluloses, etc.

Figure 3. Increment cores to be analyzed with imaging NIR for radial variations in chemical composition

In the Bio4Energy project, the NIR measurements will be performed on solid wood. The instrument has however already been used to build a model for estimation of lignin from a large number of samples of milled wood within the EU project Trees4Future. The samples were produced and previously analyzed for lignin content by Manfred Schwanninger, BOKU. They were poured into containers. Sets of these were scanned automatically at a time. Average NIR spectra were calculated for each sample and combined with the reference data. A model for estimation of lignin content with R^2 = 0.89 was developed, see Figure 4.

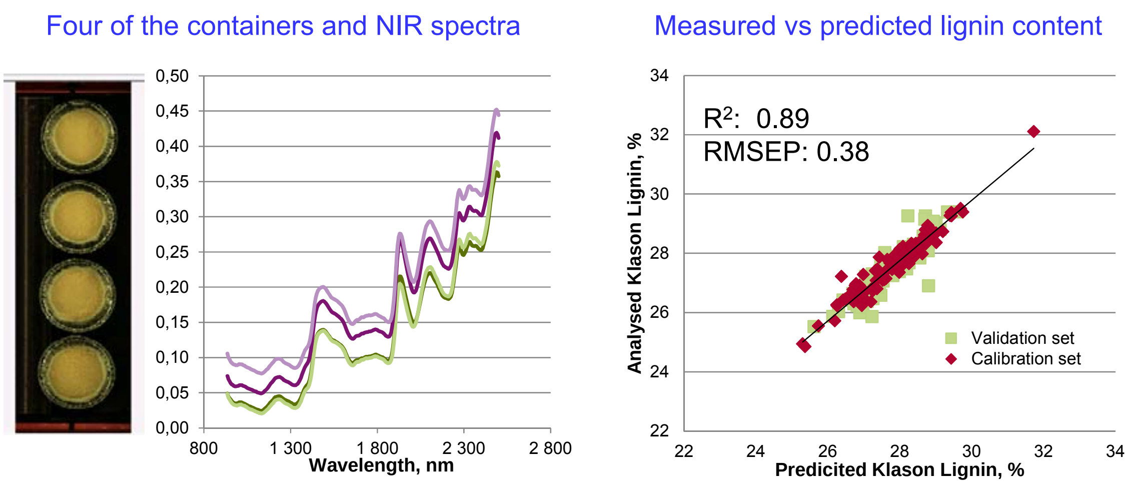

Figure 4. Four containers with wood meal, part of a set analyzed at a time with imaging NIR; Average NIR spectra calculated for the 4 samples; Illustration of the model developed for estimation of lignin content

A number of other potential applications in research, forestry and industry have been looked at. Some wood related examples are occurrence of compression wood, differences within and between trees and tree species, identification of tree species, impregnation, defects in wood, and property variations of pulp and paper.

Some reflections on problems and concerns with NIR based measurements in general and imaging NIR in special are summarized bellow:

NIR – general issues

Calibration

As for all indirect measurement method, reference data are needed to calibrate/build models for estimation/prediction. The reference data have to reflect all relevant variations in material properties, state of the objects and potentially influential factors of the measurement environment. It may not be easy to foresee what variations need to be covered. It is generally easier in a lab environment and more difficult in applications where you have less control. Properties and conditions may also change over time, due to changed product recipes, environmental factors, etc. These factors will often create difficulties and need for special concerns:

- Need for large calibration data sets
- Need for follow up of the models and updates based on additional reference data
- The model development is often dependent on the existence of accurate reference measurements
- Even if accurate reference methods exist, it may in cases be hard to apply them on the identical sample material as the NIR spectra are measured from
- Expensive reference analyses may make it difficult to obtain a sufficient number of reference samples

Reflection and depth of measurement

- Different measurement depth for different wavelengths
- Different measurement depth depending on material properties and structure
- Effect of background for thin samples
- Influence of surface structure and particle size
- In cases effects influences from the surrounding, i.e. the background, a sample vessel, reflected light, …

Additional issues for imaging NIR

- Resolution
- The pixel size is known, but what is the true resolution of the information obtained
- Light scattering within the material
- Reflection in different angles from the surface

Illumination

- Intensity variations – spatial and over time
- Scattered light outside the object

Edge effects

Non destructive characterization of wooden members using NIR

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On site characterization of wood members is a very challenging task, after considering all the variables affecting the whole structure itself and material used for construction. The up-to-date procedures are limited to few characterizations, and in general based on visual assessment supported by local drilling resistance analysis, stress-wave time of flight measurement and/or moisture content estimation.

The goal of this work was to promote near infrared (NIR) spectroscopy as a supplementary tool providing additional information for the expert assessing timber structures [1,2]. Several examples of successful NIR application in species recognition, physical properties prediction, evaluation of wood weathering and/or fungal degradation level is presented here [3,4]. Results obtained from NIR spectroscopy show close correlation with the reference data obtained by standard analytical methods. A novel approach for structures inspection and decision making regarding its state, by considering the supplementary infor provided by NIR spectroscopy is presented on Figure 1.

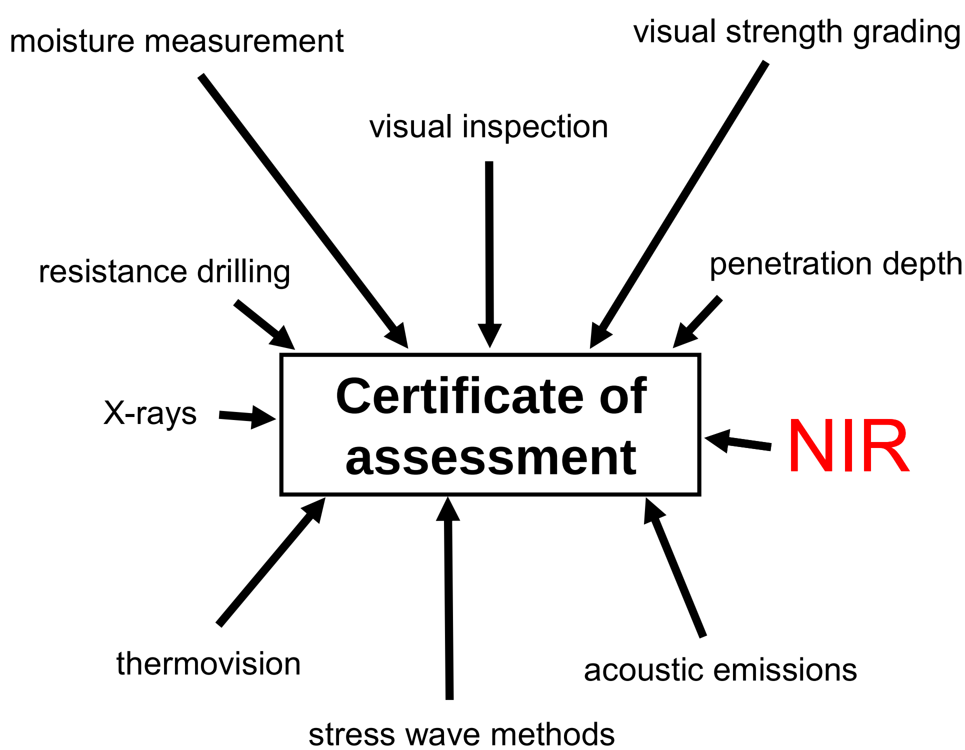

Figure1. NIR as one of tools for supporting expert decision

Problems:

- several phenomena can affect wooden members state, including moisture history, events (fire, flooding), ageing, weathering, biotic/abiotic wood degradation, mechano-sorption,
- implementation of NIR in routine assessment protocols requires prior preparation of a dedicated databases of high precision reference values to build reliable, flexible and sufficiently generalized models
- interpretation of the spectra collected in-field is often problematic

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Estimation of mechanical stresses with NIR

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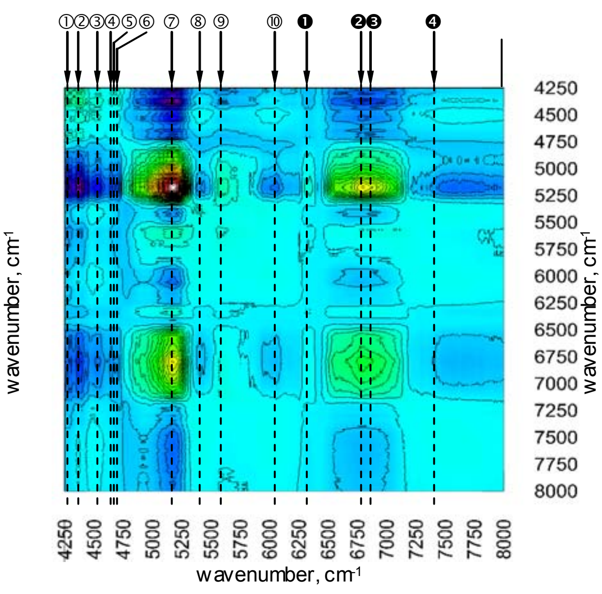
Significant scientific work has been dedicated for exploration of the infrared applications within wood science and technology. However, not much has been done linking it to the mechanical testing [1]. It is expected that due to mechanical stresses (and related deformations) the interaction between constitutive elements of wood changes, proportionally to the stresses applied. The response of the material to mechanical stresses (such as tensile loading) on the molecular level should be therefore detectable by means of infrared spectroscopy. Dedicated tests have been devoted for proving this hypothesis [2,3]. Self developed testing machine has been integrated with near infrared spectrometer in order to conduct a series fully controlled mechanical tests. It was possible to predict stress level of wood during tension by applying proper chemometric analysis (PLS models). 2D spectral correlation analyses were performed on the set of data collected during tensioning of the spruce samples. The disturbance was here the tension force, changing from 0N to 3000N with a step of 100N. The obtained synchronous chart of plastic deformations is presented on the Figure 1. Implementation of infrared spectroscopy in to timber engineering and mechanical testing of wood provides very essential supplement to the typical information collected during standard tests.



① cellulose (-CH, -CH_2)
② cellulose (-CH)
③ lignin (-CH, -C=O)
④ cellulose (?)
⑤ cellulose (?)
⑥ hemicelluloses (-CH, -C=O)
⑦ water (-OH)
⑧ cellulose (-OH, -CO)
⑨ cellulose (-CH, -CH_2)
⑩ hemicelluloses (-CH)
❶ crystalline regions of cellulose (-OH)
❷ cellulose (-OH)
❸ glucomannan (-OH)
❹ hemicellulose (-CH)

Problems:

- More additional tests and reference data are necessary in order to create more reliable and universal model suitable for routine on-site assessments
- Is this really reconfiguration of macromolecules of wood polymers measured with NIR in this case?
- Is it possible to estimate stresses on structures in use by means of NIR?

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Using NIR for wooden cultural heritage and waterlogged wood

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Proper conservation of original wooden objects is vital to meet the concept of authenticity of the cultural heritage. Wood is considered as a very friendly and useful raw material used extensively for various purposes from ancient times. Wood, as most of natural materials, is slowly changing/degrading on-site due to various biological factors. Bacteria, fungi, temperature, UV, pH, oxygen and water are the main factors influencing presence and speed of biological, physical and/or chemical deterioration. In a consequence of such degradation significant changes within physical, mechanical and chemical properties are occurring [1-3].

Rapid and accurate estimation of the degradation level is extremely important as it influences the selection of optimal restoration and conservation procedures. Nowadays a tendency for application of various analytical techniques into measurement of the ancient wooden artifacts is observed. The advantage of near infrared spectroscopy, beside of the nondestructive character, is the simplicity of measurement and possibility of numerous repetitions what significantly increases the reliability and accuracy of data. Some examples presenting the usefulness of NIR technique in the field of cultural heritage as presented on the workshop includes:

- identification of substances used for wood protection in the past [4]
- assessment of the chemical changes to the wooden surfaces before and after the restoration process
- evaluation of the degradation stage of waterlogged archeological wood

Figure 1 presents how wood surface treatments can be distinguished with NIR. The principal components analysis clusters samples into groups of no-treated, varnish coated and wax finished surfaces. It is also evident that the spectral distance between no-treated wood increases with the quantity of the coating applied and follows direction indicated by arrows.

It is expected that waterlogged wood changes its physical-chemical properties during storage in water. It can be confirmed by analysis of NIR spectra. As shown on Figure 2, the spectral distance between contemporary wood and waterlogged wood increases with the time. It was found, however, that in some cases (for example the sample stored for 8731 years in water) the NIR spectra seems to be very similar to that on contemporary wood. It was confirmed by means of visual assessment where the appearance of ancient wood was very similar to contemporary samples.

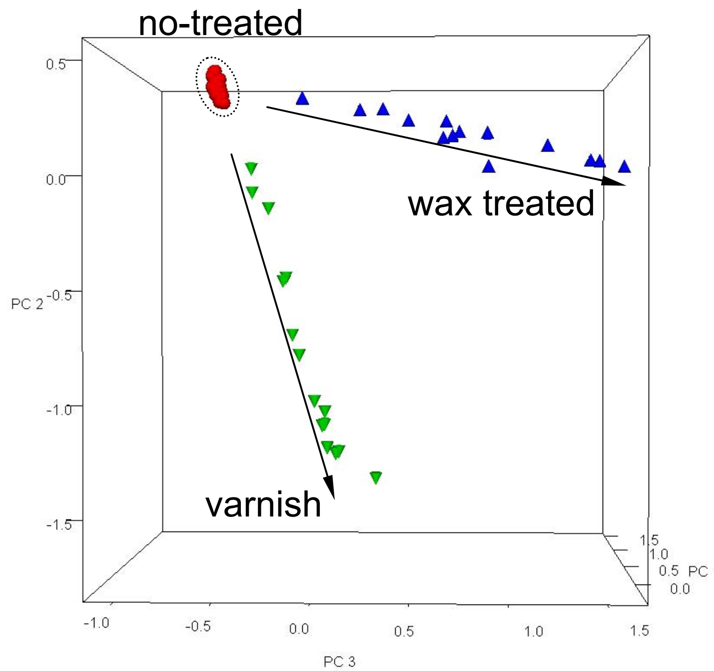

Figure 1. PCA of FT-NIR spectra scanned from pine wood (*Pinus silvestis* L.) surface coated with varnish and wax (2der 21smoothing points + vector normalization, spectral band: 4060-11900cm^{-1})

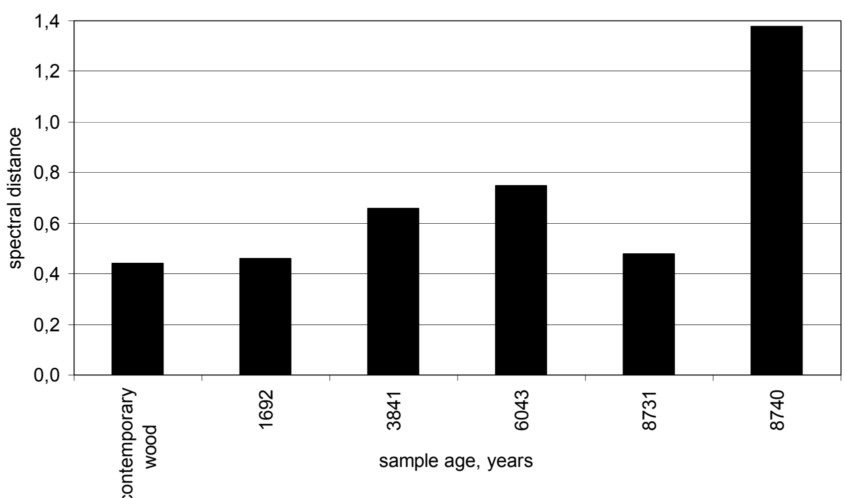

Figure 2. Distances between average spectra scanned with NIR (selectivity) on contemporary and different duration waterlogged woods.

Problems:

Challenges and problems related to use of NIR spectroscopy in the fields of cultural heritage and archaeological wood:

- limited sample that can be taken from historical objects for laboratory

- complex degradation factors, which in many cases cannot be known to the researcher
- formulation of general trends is a subject of error

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Application of NIR Spectroscopy to Archaeological Wooden Materials

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It is important to investigate the degradation mechanism of antique wooden materials used for historical temples, buildings and crafts from the viewpoint of wood science and archaeology. NIR spectroscopy is a powerful tool for measuring the physical, chemical and mechanical properties of wood whilst retaining the anatomical structure of the archaeological samples. In this presentation, we introduce two topics due to research for NIR-Archaeological wooden materials.

The change of crystalline structure in hydrothermally treated hinoki wood as an analogue of archaeological wood was investigated by means of FT-NIR spectroscopy with aid of deuterium exchange method and X-ray diffraction (XRD) method. Results were compared with that of dry-exposed archaeological wood taken from an old wooden temple. It is assumed from deuterium exchange experiment that several elementary fibrils are arranged in very close proximity, under 0.3 nm due to the expansion of crystalline width by hydrothermal treatment.

The diffusion process of deuterated water in washi (Japanese traditional paper) was investigated by means of a deuterium exchange method and FT-NIR transmission spectroscopy. The samples were the modern (AD 2003) hand-made washi and those from an archival collection of cultural artifacts (AD 1791 and 1615). It was suggested that during aging hemicellulose, which forms a composite with cellulose in paper, was progressively hydrolyzed, resulting in the expansion of inter-molecular distance between cellulose chains.

Project SLOPE: Integrated processing and control systems for sustainable forest production in mountain areas

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Mountains in Europe occupy ~35% of the land area and are mostly covered by forests. Forestry operations in mountain areas are seldom performed by the harvester/forwarder system, being the sector still characterized by manual felling and extraction of timber by cable cranes. Due to the limits posed by steep terrain conditions, poor road network of mountain areas, limited storage and operational room, harvesting and extracting systems are more expensive and less flexible compared to the cut-to-length systems based on wheeled machines, commonly found in flatland forests of EU Nordic Countries. Powerful and intelligent machines must be developed for forest works in steep terrain. This is the gap that SLOPE (Integrated proceSsing and controL systems fOr sustainable forest Production in mountain arEas) will try to fill in by developing an integrated system, from forest information system to logistic transportation, that allows optimization of the forest production in mountain areas. The project will integrate information from remote sensing, Unmanned Aerial Vehicles (UAV) and on-field surveying systems, to support macro and local analysis to characterize forest resources. Spatial information, including NIR and hyperspectral images, will be integrated with multi-sensor data in a model for Sustainable Forest Management and for optimization of logistics during forest operations. Intelligent technologies will be integrated in the cable crane/processor head to measure different data for the assessment of the assortment variety. Different Non-Destructive Testing methods, including NIR spectroscopy as well as pioneering chemometric analysis, will be tested during the project. Different traceability systems will be coupled to chemometric data, to trace the material, from the site throughout the supply chain. Information about material origin, quality and availability will be integrated in a unique system, accessible online and available in real time to a series of operators. The integration and post-processing of data collected by SLOPE will be used for further optimization of the "mountain forest models" and finally silviculture routines.

SLOPE in brief:

- Theme: Integrated processing and Control Systems for Sustainable Production in Farms and Forests
- Duration: 3 years
- Budget: app. 5M€
- Project Funding: EU 7th Framework Programme (FP7)
- Partners: Fondazione Graphitech (It), Cnr-Ivalsa (It), Kesla OYJ (Fi), Coastway Limited (Ir), MHG Systems OY (Fi), Universität für Bodenkultur Wien Boku (A), Flyby Srl (It), Greifenberg Teleferiche Sas (It), TreeMetrics Ltd (Ir), Instituto Tecnologico del Embalaje, Transporte Y Logistica (Es)

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http://www.slopeproject.eu/

Session 4

NIR & wood surface;

in collaboration with COST Action FP1006

Time-resolved diffuse optical spectroscopy for wood characterization

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We have recently proposed the use of picosecond Time-Resolved Diffuse Optical Spectroscopy (TRS) in the near infrared range (700-1300 nm) for the non-invasive in depth optical characterization of wood samples. TRS technique allows one to separately measure bulk absorption and scattering spectra, which can be related to chemical and structural composition of the sample, respectively. Different types of wood have been investigated (e.g. hardwood and softwood) in different conditions (e.g. previously degraded and subsequently consolidated). In particular the technique can be used both to assess waterlogged wood, and for the monitoring of dynamic changes in the moisture content of wooden samples. The application of TRS technique for monitoring the consolidation and conservation purposes of waterlogged wood in cultural heritage science will be discussed. It is worth mentioning that the rapid development of optical technologies (e.g. laser sources, detectors and optical components) opens the way to the realization of a compact and portable system to be used on the field.

Estimating wooden veneer moisture content using infrared technology

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The non-destructive testing (NDT) of wood veneer is industrially important in the manufacturing of several products including laminated veneer lumber (LVL) and/or plywood. Recently, a trend for monitoring of both, veneer moisture content (MC) and density during production at the mill is observed. The measurement of MC is essential since over-drying should be avoided to save energy and to maintain veneer quality. Whilst, a high MC must be avoided (especially moisture spots) because these can cause problems during hot-pressing. On the other hand, it is very important to properly estimate wood density as most wood properties are correlated with it. Particularly, the strength grading of veneers at the mill is of special interest as the relationship between wood strength and density is linear. Veneers of low density may cause further problems with the poor mechanical properties of the end product.

The resulting strength of LVL is therefore highly dependent on properly selected veneers assuring optimal MC and density. For these reasons, it would be of great interest if MC could be measured precisely and with sufficient spatial resolution already during production. Currently available on-line MC and density measuring sensors base either on measurement of wood's electrical resistance, or on measuring attenuation and phase shift of microwaves. Spatial resolution of these measurement methods is rather poor and not always sufficient for industrial needs. It is expected therefore that alternative methods, such as NIR-based, could offer other option when developing MC and density measurement systems. Several studies related to the usability of NIR spectroscopy toward estimation of wood moisture were already performed by numerous researchers including [1].

A newly developed compact sensor (MicroNIR by JDSU) [2] has been tested here in order to determine its capability for estimation of the wood moisture content. Series of dedicated tests were performed in order to validated the potentialities and limits for this approach. The settings of the sensor were set-up in order to assure highest signal level, maximum scanning frequency and minimum exposure time. As a result each spectrum has been measured with frequency of 80Hz and12.5ms integration time. A natural drying test if thin veneer (100µm thickness) of spruce (*Picea abies*) was conducted in order to test the sensors capability for estimation of water content. The initial moisture of wood was ~200% (fully saturated with water) and the drying was performed up to equilibrium moisture content (8%). The change of the NIR spectra (second derivative) is presented in Figure 1. A clear pattern of the 1414nm (7073cm^{-1}) can be observed suggesting its relation to the moisture content. This band is related to –OH functional groups of water and woody polymers. It can be even better visualized on the Figure 2 where the progress of the 2nd derivative at 1414nm follows the trend and was clearly dependant on the wood moisture content.

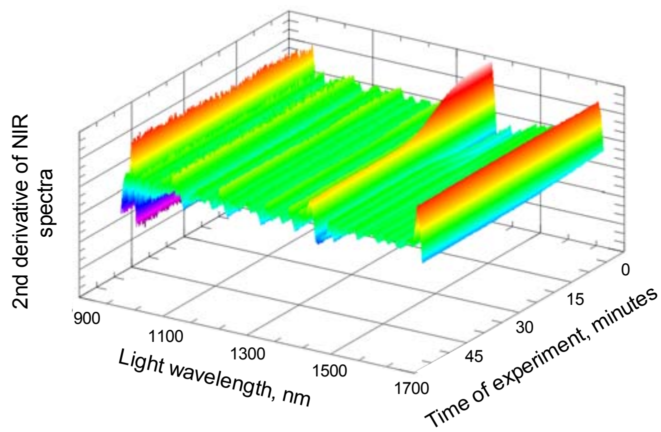

Figure 1. Pattern of changes of the NIR spectra (2nd derivative) during natural drying test as measured with MicroNIR sensor

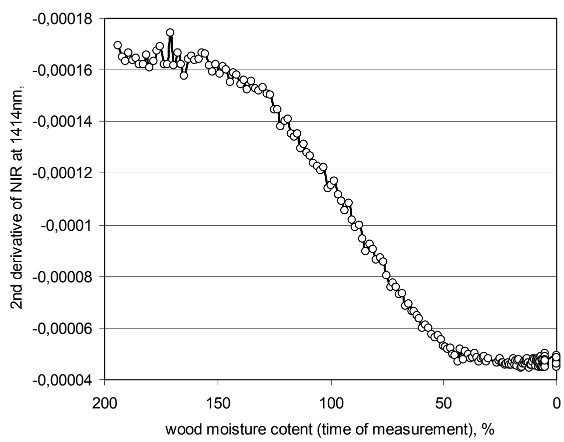

Figure 2. Relation between 2nd derivative of NIR spectra at 1414nm and wood moisture content as measured during natural drying test

Problems:

- What kind of signal (spectral range, resolution) is required to assure due performance of measurement system?
- Is the signal/noise ratio of the MicroNIR sensor sufficiently high to assure problem-less measurement of moisture during production?
- How will be the optical sensor performing in harsh industrial conditions, assuming measurement distance of few millimeters?

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NIR assessment of chemical pattern of wood after thermal modification

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Thermally modified wood (TMW) has become a new commercial product possessing a number of advantages over the natural wood. Even if several processes are already established on the market, there are no available standard procedures for quality control and/or unified product specifications. The unique document (UNI CEN/TS 15679 2007) contains examples of characteristics to be expected from TMW produced using some selected commercial processes. Since several methods for production of TMW exist, the quality of product might significant vary depending on the manufacturing procedure. Therefore, the emerging research need is a quality control of TMW with a special attention for non destructive, cheap and rapid methods [1-3]. The goal of this research was to exploit the potential of the near infrared spectroscopy into evaluation of the chemical changes to eight wood species (representing both soft and hard woods), exposed to thermal treatment in vacuum conditions. The other objective was to develop a robust prediction models for modified wood properties, such as mass loss due to treatment and equilibrium moisture content of modified wood [4].

An example of results is presented in Figure 1. It is clear that various wood polymers respond differently to the thermal treatment. According to NIR spectra analysis it can be seen that the most resistant for thermal degradation is lignin. Hemicelluloses and hydroxyl groups assigned to water are the most affected.

Such knowledge might be helpful for further optimization of thermal treatment procedure in industrial scale as well as certification of the TMW quality and further performance.

Problems:

- quality of the product might significant vary depending on treatment procedure applied, since several thermal modification processes are recently present on the market
- the specific properties of each species, including growth condition, provenance, defects, affects significantly the thermal modification process and further product properties
- measurement of the NIR spectra during modification process is problematic due to harmful environment (vacuum and high temperatures)

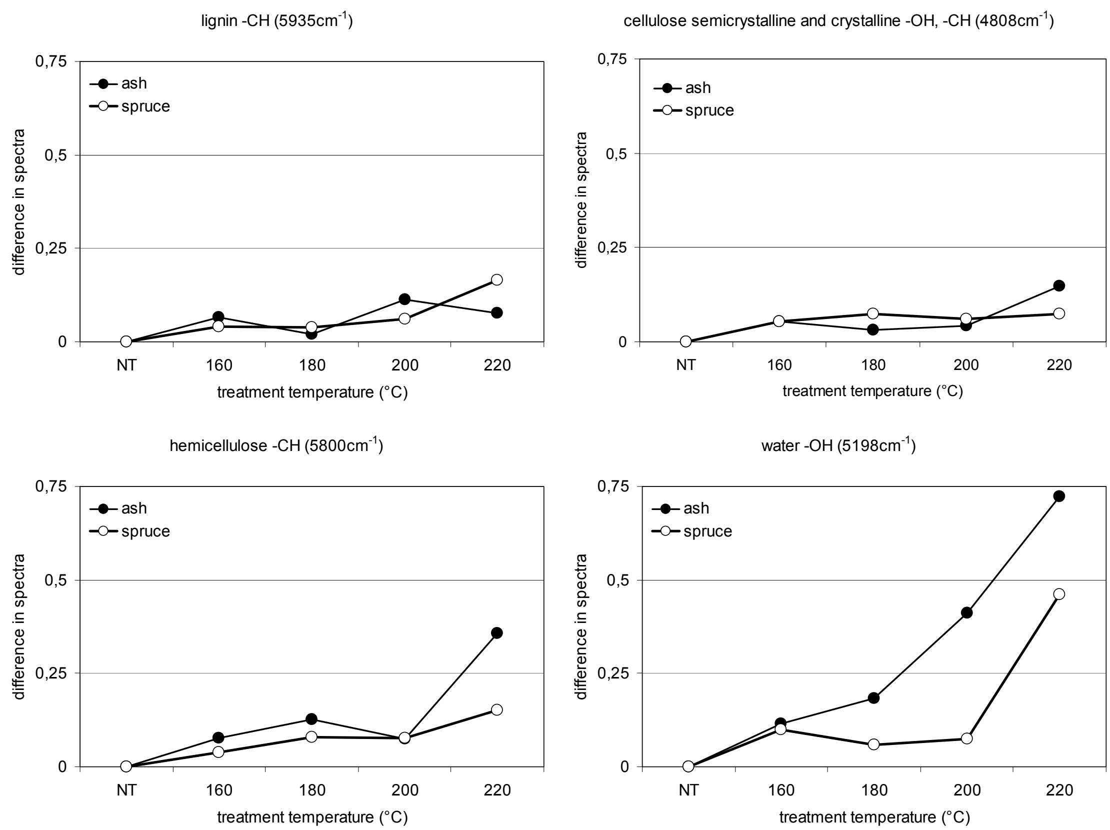

Figure 1 Pattern of changes of ash and spruce wood related to main chemical components in relation to treatment temperature

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Hyperspectral imaging: a new tool for detecting and quantify mould growth on wood surfaces

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Introduction

The degree of mould growth on the surface of coated and uncoated wood is a measure of the substrates (wood material and/or coating) susceptibility to infestation of mould fungi. It is a way of rating the substrates according to quality, treatment, time, climatic factors, workmanship, design etc. [1]. Assessment of mould growth coverage implies a need for a quantification that allows objective numeric validation without considerable subjective appraisal. Visual assessment is the current state of the art for evaluating mould growth coverage on the surface of coated and uncoated wood using standards such as: BS 3900 1989 [2], EN 927-3 2006 [3], ASTM D3274 2002 [4] and EN 152 2011 [5]. The objectivity of using visual assessment is often questioned [6-9] and there is an on-going interest and research for finding additional or alternative methods for more objective assessment.

In this study hyperspectral imaging technology was applied for evaluating and quantifying the growth of the discolouring fungi *Aureobasidium pullulans* and *Cladosporium cladosporioides* on the surface of coated and uncoated wooden samples. The aim was to determine if fungi can be detected, localized and quantified from hyperspectral images. Furthermore, we shortly present an ongoing field test to show a possible application of the method.

Experimental methods

Wood samples (50 x 50 x 2 mm) made from Norway spruce (*Picea abies*) were coated with a water-borne or solvent borne paint without fungicide, or left uncoated. The samples were put on malt agar growth medium in petri dishes and 500µl of a spore suspension of *A.pullulans* or *C.cladosporioides*, was applied to the upper face side of the samples. Samples without added fungal suspension were used as references. The samples were incubated at room temperature for one to six weeks to achieve a step-wise mould growth coverage.

Hyperspectral imaging was performed using a line scan Mercurium Cadmium Telluride (MCT) camera, covering the NIR wavelength region 900 – 2500 nm distributed on 256 channels. In average it takes 5 seconds to scan the sample. The spatial resolution of the setup was approximately 200 µm.

Principal Component Analysis (PCA) was applied to the hyperspectral image cubes in order to extract statistical relevant information from the data set by reducing the large number of correlated variables to a limited number of linearly uncorrelated principal components, using an orthogonal transformation. A more detailed description of the equipment and data analysis is given in [10].

Results and discussion

Signals from *A.pullulans* and *C.cladosporioides* could be seen on the infested samples selected for the images by visual inspection of the visible wavelength and the NIR hypercubes. The second component score images from PCA show how the *A.pullulans* and *C.cladosporioides* fungal biomass are revealed from a more uniform background (Fig. 1 and Fig. 2).

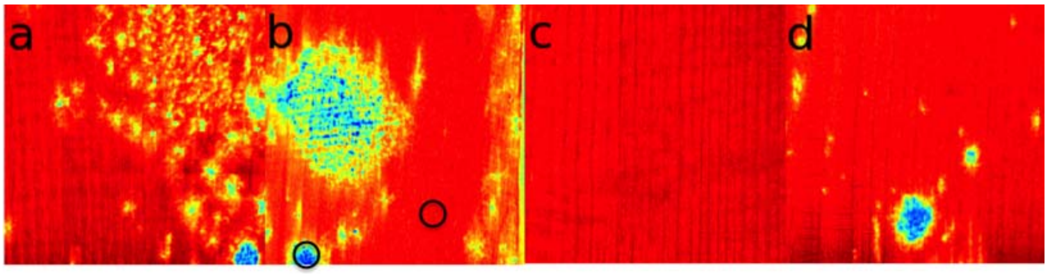

Figure 1. PCA second score image (with decluttering) of samples with waterbased coating and with *A.pullulans* growth observed with hyperspectral camera at NIR wavelengths. Black rings mark the spots where the spectras in Figure 3 was picked.

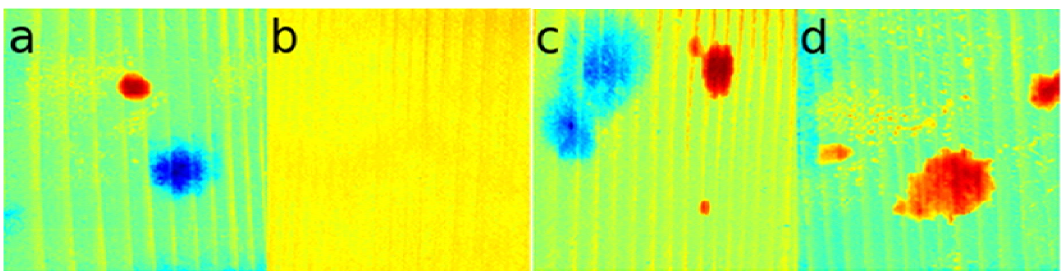

Figure 2. PCA second score image (with decluttering) of samples coated with solent borne coatin and with *C.cladosporioides* growth observed with hyperspectral camera at NIR wavelengths.

The NIR hypercubes show strong signals from water, particularly recognized by the spectral peaks at 1190 and 1450 nm, signals that are generally attributed to free water content in capillaries [11]. The PCA seems capable of extracting the water signal as a separate principal component, and the fungal signal as another component. Moreover, the spectrum extracted from areas with *C.cladosporioides* seems to systematically contain the signature peaks related to water, even in dry regions on the sample. The spectra from areas with *A.pullulans* however, show only absorption of the surface coating with no traces of the water spectrum. However, a more systematic study should be carried out to investigate if the *C.cladosporioides* always has a high content of water within the hyphae and if this can be used to identify the specie from *A.pullulans*.

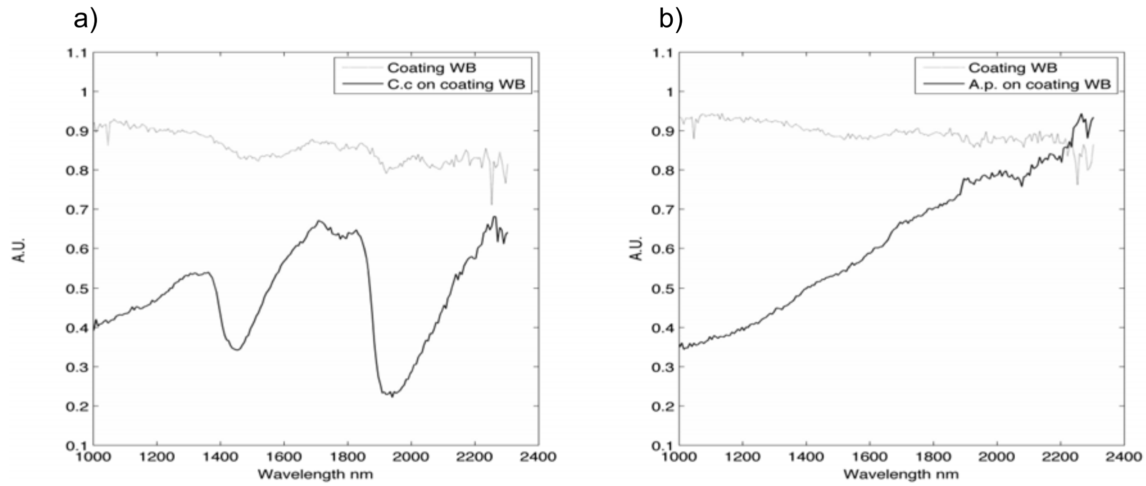

Figure 3. Mean spectra from 3x3 pixels cluster located on the sample; (a) without any fungi (grey) and on a spot with *C.cladosporioides* (black), (b) without any fungi (grey) and on a spot with *A. pullulans* fungi (black).

Conclusion and challenges

This laboratory study showed that hyperspectral imaging can be used for detection and quantification of fungal growth in a laboratory environment using mono culture of fungal species. A challenge for this technology is that the surface of mould is very thin and there is an uncertainty whether the observed spectra are due to decomposed wood or scattered light effects from the mould, rather than the mould spectral signature itself. A more detailed laboratory analysis will reveal more information about these issues. A time series of the mould growth on wood samples is recorded and the images will be analysed with advanced pre-processing algorithms such as Multiple Scatter Correction (MSC) in order to distinguish the nature of the different recorded signals.

There are several benefits using NIR hyperspectral imaging, such as the speed of measurement (5s to scan one sample) and the fact that the whole sample is scanned. The flexibility of hyperspectral imaging regarding sample size also makes it ideal for outdoors case studies. In another ongoing field study, we want to gather experience from using hyperspectral imaging of fungal growth on a variety of wood substrates exposed in an outdoor environment resulting in fungal surface growth caused by a mixture in species of mould and blue stain fungi. Five different wood substrates have been exposed vertically facing north and south at Sørås test field, Ås, in a specially designed test set-up. The wood substrates included in the study are: uncoated Aspen, uncoated Pine heart wood, uncoated Spruce heart wood, uncoated acetylated Southern Yellow Pine and coated Spruce heart wood. Sensors for wood moisture and temperature measurements were mounted within selected samples, and sensors measuring relative humidity and leaf wetness were placed on the test rig. An advanced nationally served meteorological monitoring station is placed in immediate closeness of the test rig, and data for a rage of meteorological attributes are available for further analysis. Start-up time of the test was August 2013 – and the test is still running. Hyperspectral images and RGB pictures of the samples are taken of the samples at fixed time intervals, as well as visual on-site evaluation according to EN 927-3 (2006). Challenges encountered in this outdoor experiment are the varying light conditions and the atmospheric absorption bands.

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NIR Spectral Imaging for the In-line Detection of Preservatives in Recovered Wood

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Introduction

The growing need for wood as a resource for the industry led to an increase of global wood harvesting. Nevertheless the demand for wood is not satisfied by this means. The difference between supply and demand results in higher cost for timber products. For this reason, currently substantially unused resources are interesting, for example recovered wood. Every year eight million tons of waste wood are generated in Germany, whereof only one third is used as material, especially for the production of particle boards and two thirds are incinerated to produce electricity and heat [1-2]. Due to e.g. national legislation, the existence of contamination and the local pricing pressure by scarcity of wood, the amount of recovered wood in particle boards can be different in other countries: Italy-89%, Denmark-66%, UK-55%, Austria-38%, Poland-10%, Finland-0%, Greece-0%.[1] So far mechanical separation techniques are mostly used to reduce the contamination level. Our goal is to increase the contemporary amount of recycled wood in Germany by using modern sorting machines equipped with sensors capable of chemical analysis to actuators removing foreign materials. Thereby even high contaminated fractions of waste wood could be substantially used, which contain e.g. wood preservatives, plastics, coatings or mineral pollutants and are incinerated almost unsorted at the moment. This procedure is against basic understanding of ecological und economical valuable carbon cycle, in which the organic material is used in a cascade and only the last resort is the elimination by incineration. The closer investigated analytical methods are X-ray fluorescence (XRF) spectroscopy[3], ion mobility spectrometry (IMS)[4] and near-infrared (NIR) spectroscopy[5-6], whereof the latter is considered in detail here. One important requirement is that the method must be integrated into existing industrial processes as an in/on-line measurement. Therefore it has to be fast, reliable and robust.

Material & methods

Various wood species were treated with typical organic and inorganic wood preservatives such as pentachlorophenol (PCP), lindane, tolylfluanide, tebuconazole, carbamates, copper salts, chromated copper arsenate (CCA), boric acid etc. The quantity of these active ingredients was determined by conventional reference analysis such as GC-MS, HPLC-MS and ICP-MS and is in the usual range for wood preservatives in timber products. The samples were transported on a conveyor belt and illuminated with halogen lamps (Figure 1). The diffusely reflected radiation was detected by a NIR hyperspectral imaging camera (SpectralEye NIR 2.2, inno-spec, Nürnberg, Germany). The system is sensitive in the wavelength range from 1100 nm to 2200 nm, has a cooled InGaAs detector with 256 x 320 pixel (spectral x spatial) and a frame rate of up to 330 Hz. The device applies the principle of pushbroom imaging [7]: the two-dimensional detector measures a row of 320 pixel perpendicular to the transportation direction. Every single point is split into 256 single wavelengths. Through the movement of the sample a picture of it is generated (Figure 2), containing one NIR spectrum for each single pixel of the region of interest. Because of the huge amount of information, it is necessary to automatize the assessment of the NIR spectra by using multivariate data analysis (MVA). Principal components analysis (PCA, as shown in Figure 3), the PARAFAC algorithm or Simplex Volume Maximization (SiVM) are used for data reduction. In some cases, additional data classification by means of Linear Discriminant Analysis (LDA) or Learning Vector Quantization (LVQ) is necessary. But at first several pre-processing steps are used to increase the signal-to-noise ratio and to avoid differences due to non-chemical information of the material. Methods such as standardisation, differentiation, spatial smoothing (median filter), spectral smoothing (Savitzky-Golay) and multiplicative scatter correction are applied.

Figure 1: NIR Hyperspectral Imaging System

Results & Discussions

At first the reflectance spectra of the pure active ingredients were measured. The organic wood preservatives showed comparatively large spectral differences. Except of the two water absorption peaks at around 1450 nm and 1900 nm, the inorganic compounds showed no signals in the near infrared region. Nevertheless previous laboratory experiments [8-9] demonstrated that wood treated with inorganic preservatives was distinguishable from natural wood with NIR spectroscopy. We can support those findings even for non-milled material. It is presumed that the inorganic salts bind to the wood matrix with its hydroxyl groups from its natural constituents cellulose and lignin. Therefore, the molecular vibrations change and the overtone and combination vibration of the wood matrix are different resulting in different NIR signals. This means that the inorganic wood preservatives can indirectly be measured with NIR spectroscopy. The discrimination of wood treated with organic preservatives from natural wood on the laboratory scale was demonstrated as well.

However, factors such as different particle surface features, noise, lighting variations, humidity etc. which cannot be avoided in an industrial environment and will influence the stability and accuracy of the classification algorithms. A possible way to work around this is to extend the spectral range used to longer wavelengths up to the thermal infrared (3 000 – 12 000 nm). Corresponding research activities are under planning.

Last but not least the variation of the concentration of the active ingredient is another important issue. NIR spectroscopy is not a method for trace analysis. Especially waste wood with long lasting exposure to moisture and heat can contain significantly less active ingredients because of washing out or evaporation effects. It needs to be tested with extra samples how low the measured concentrations can be to determine the limit of detection. Those values are hardly comparable with conventional analysis, because they refer to homogenous samples and not only to a surface layer which is investigated by NIR spectroscopy.

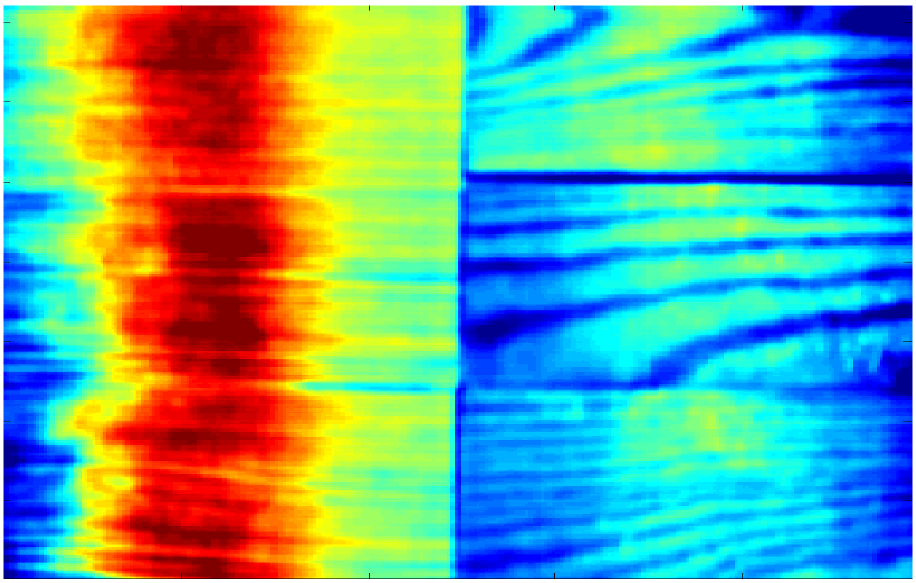

Figure 2: First principal component of the hyperspectral image of the natural pine wood (left) and pine wood treated with boric salts (right)

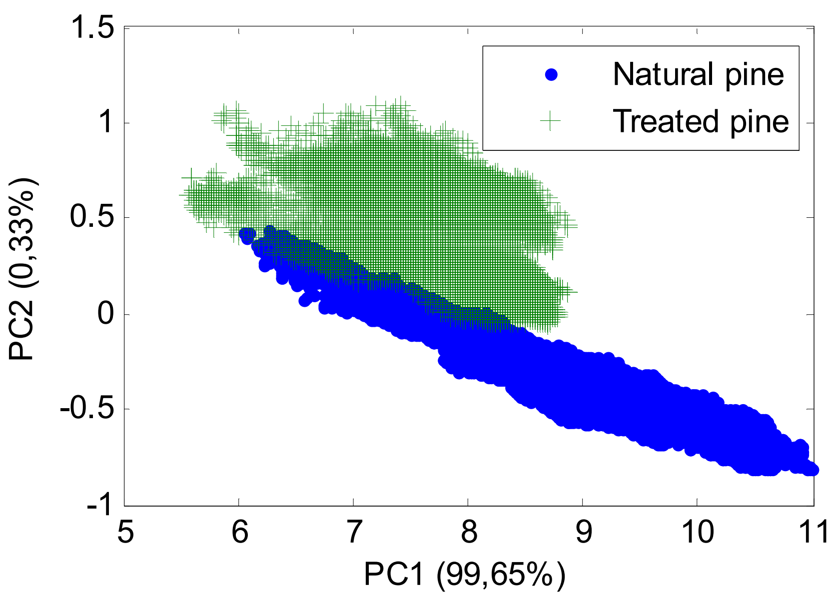

Figure 3: Scoreplot of principal component analysis of treated and natural pine wood

Problems:

- Why are there such differences in the amount of waste wood used for the production of particle boards in different European countries?
- Quantification:
    - NIR spectroscopy is not a method for trace analysis of contaminants
    - Which LODs for wood preservatives measureable?
    - Comparability with values by conventional methods?
    - In-/decrease of concentration of active compounds on the surface?
    - Penetration depth: measuring depth of NIR radiation for wood?
- Benefits by using extended spectral range (e.g. mid-IR)?

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Project SWORFISH: Superb Wood Surface Finishing

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The main goal of the SWORFISH project is to establish criteria for the optimization of manufacturing processes and mechanical, thermal and chemical modification of wood surfaces with selected physical and mechanical properties such as extra-durability, elevated abrasion resistance, fire self-extinguishing characteristics, anti-bacteria features, self-cleaning and accumulation of emissions/particles/pollutants.

The different parameters affecting the final quality of wooden surfaces depend on both material processing and modification and material original characteristics, such as the wooden species, moisture content, density, heterogeneity at the various anatomical scales.

Therefore, an innovative measurement line for assessing all important properties of the surface has been developed, with a special focus on exploring NIR sepctroscopy.

Correlations among the different properties and the processes involved in the surface formation are analysed by means of numerical models, which are validated in situ as well in vitro, in a laboratory environment.

All the novel surface modifications and treatments are carefully evaluated from the point of view of their impact on the human and environment. The project developments will take into account life cycle analysis, recyclability and hazard to the surroundings as integrated procedures.

SWORFISH in brief

- Theme: team 2009 incoming (CALL 2) and Trentino PCOFUND-GA-2008-226070
- Duration: 4 years
- Budget: app. 425k€
- Project Funding: European Union – FP7 – Marie Curie – Cofund – Progetto Trentino.
- Team: Jakub Sandak (coordinator), Anna Sandak, Mariapaola Riggio, Ilaria Santoni, Dusan Pauliny

References & more info

http://www.ivalsa.cnr.it/en/current-projects/tecnologia-del-legno/sworfish-superb-wood-surface-finishing.html?tx_wfqbe_pi1%5BPERSONA%5D=

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