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2nd Workshop on application of NIR spectroscopy for wood science and technology research

NIR & WOOD – SOUNDS GOOD! #2

In memoriam of

Dr Federico Prandi (1974 – 2016)

Book of abstracts

April 19-21, 2016

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

edited by:

Jakub and Anna Sandak

co-organized by:

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

Italian Society for NIR Spectroscopy (SISNIR)

COST Action FP1303 “Performance of Biobased building materials“

COST Action FP1407 “Understanding wood modification through an integrated scientific
and environmental impact approach (ModWoodLife)“

BI⁴ever



Workshop:
Application of NIR spectroscopy for wood science and technology research: 2nd edition

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sponsored by:
Bruker Italia, Viale V. Lancetti 43, 20158 Milano (IT)
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Workshop: Application of NIR spectroscopy for wood science and
technology research - NIR & WOOD – SOUNDS GOOD!

Book of abstracts

Workshop program

	Tuesday: April 19
13:30	registration
	Session 1: Instrumentation and hands-on
14:00	General presentation of NIR technology in aspect of wood, <i>Jakub Sandak</i>
14:30	Bruker presentation, <i>Paolo Belloni</i>
14:45	Viavi presentation, <i>Emiliano Genorini</i>
15:00	COFFEE-BREAK
	hands-on session
15:30	group 1: Bruker / group 2: Viavi
17:00	group 1: Viavi / group 2: Bruker
	Wednesday: April 20
08:30	registration
09:00	Welcome from IVALSA + SISNIR
09:10	Introduction of the participants
10:00	Keynote 1: Shedding light on wood: time-resolved diffuse optical spectroscopy to characterize absorption and scattering, <i>Cosimo d'Andrea</i>
10:45	round table discussions
11:15	COFFEE-BREAK + equipment presentation
11:45	Keynote 2: Probe technology for in-situ NIR applications, <i>Paolo Belloni</i>
12:30	round table discussions
13:00	LUNCH
14:30	Keynote 3: Near Infrared Hyperspectral Imaging – background and application for wood characterization, <i>Andreas Zitek</i>
15:15	round table discussions
15:45	COFFEE-BREAK + equipment presentation
16.15-17.45	Session 3 NIR & forest
	Using NIR spectroscopy for the discrimination between Eucalyptus Nitens and E. Globulus, <i>Guillermo Palacios</i>
	Detection and spectral characterization of resin pockets in spruce by FT-NIR Hyperspectral Imaging, <i>Andreas Zitek</i>
	Application of Near-Infrared Spectroscopy to determine the Juvenile–Mature Wood transition in Spanish conifers used in construction, <i>Antonio Ruano</i>
	Biomass characterization towards contaminants, <i>Martino Negri</i>
	Characterization of beech and oak wood supported with FT-NIR, <i>Agron Bajraktari</i>
	Identification of the origin of Big- leaf mahogany wood from five Latin American countries via NIRS and PLS-DA, <i>Tereza Cristina Monteiro Pastore</i>
	SPIRMADBOIS project : Use of NIRS for a sustainable management of forest resource in Madagascar, <i>Tahiana Ramanantoandro</i>
	RELATED PROJECTS: SLOPE: Integrated processing and control systems for sustainable forest production in mountain areas, <i>Jakub Sandak</i>
	round table discussions
19:00	DINNER
	Thursday: April 21
08:30	registration
9.00-12.30	Session 5 NIR & wood performance - COST FP1303
	Keynote 4: Biomaterials characterization with NIR, <i>Anna Sandak</i>
	Estimating the Weight Percent Gain of modified wood with Near Infrared and Raman spectroscopy, <i>Bernhard Tapken</i>
	Evaluation of wood veneers for furniture production by FT-NIR, <i>Mauro Bernabei</i>
	Application of NIR for evaluation of weathered samples from RR test, <i>Athanasios Dimitriou</i>
10:30	COFFE-BREAK + equipment presentation
	Weather degradation of thin wood samples assessed with NIR spectral imaging, <i>Ingunn Burud</i>
	The effectiveness of FT-NIR spectral analysis for determination the bio-based finishes, <i>Anna Rozanska</i>
	Characterization of wood surface degradation using FTIR, microtensile testing and colour measurements, <i>Vjekoslav Živković</i>
	RELATED PROJECTS: BIO4ever Bio-materials for building envelope - expected performance, life cycle costing & controlled degradation, <i>Anna Sandak</i>
	round table discussions
12:30	LUNCH
14.00-15.30	Session 7 Data mining & chemometrics
	Keynote 5: NIR & Chemometrics: how to handle NIR signals, recover information and build models, <i>Marina Cocchi</i>
	NIR hyperspectral imaging of Fire-Retardant-Treated Wood, <i>Ingunn Burud</i>
	Near-Infrared (NIR) hyperspectral imaging at high resolution and the difficulty to calibrate for the three main wood components, <i>Joris Van Acker</i>
	round table discussions
15:30	COFFE-BREAK + equipment presentation
16.00-18.00	Session 6 NIR & wood modification and environmental impact - COST FP1407
	Artificial weathering (QUV) of wood plastic composites made with thermally modified wood residues, <i>Edgars Kuka</i>
	Assessment of poplar veneers for plywood manufacturing after up-grading phase by vacuum thermal treatment, <i>Ignazia Cuccui</i>
	Some colour characteristics of thermally modified wood, <i>Dace Cirule</i>
	Evaluation of heat treated black alder wood by FT-NIR, <i>Emilia Salca</i>
	Pollutants detection on waste wood using FT-IR-ATR, <i>Marco Fellin</i>
	Monitoring thermally modified wood performance by NIR. Case of study: surface treatment, <i>René Herrera</i>
	RELATED PROJECTS: TV4NEWOOD - Thermovacuum: new process for generation of thermally modified wood, <i>Ottaviano Allegretti</i>
	round table discussions
18:00	closing remarks

Federico Prandi
(15.03.1975-14.04.2016)

It is a great sadness that few days ago we have lost Federico Prandi, our dear friend and extremely valued colleague. Federico was working as a senior researcher in Graphitech (Trento).

He was a scientific responsible for the SLOPE project.

Federico always had a smile for everyone and was highly regarded by everyone who ever had the opportunity to meet him. Regardless of the challenges he may have had along the way, everyone who'd ever had the privilege of working with him, always found him a special pleasure to work with.

He will be profoundly missed by many.

May he rest in peace...
Nevertheless, let us remember Federico each time, with a smile upon our faces.
We would like to dedicate the second workshop "NIR and wood: sounds good" for the memory of Federico.

Slope project team



Welcome from CNR-IVALSA

Our adventure on working with near infrared spectroscopy started ten years ago when a new “strange” instrument appeared in our laboratory. Being very sceptic 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 for a second time, a 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 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 29 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.

We would like to thank:

- SISNIR, especially Tiziana M.P. Cattaneo, Stefania Barzaghi and Roberto Giangiacomo for encouraging us to organize the 2nd NIR workshop on wood and for trust in us
- our sponsors, BRUKER Optics (especially Paolo Belloni) and VIAVI Solutions (especially Nada O'Brien and Emiliano Genorini) for financial support
- COST Action FP1303, especially Denis Jones, for support in organization and providing travel grants
- COST Action FP1407, especially Andreja Kutnar, for support in organization, encouragement and providing travel grants
- our colleagues from CNR-IVALSA, who helped us to organize this event

We are particularly grateful to our invited keynote speakers; Marina Cocchi, Cosimo d'Andrea, Paolo Belloni and Andreas Zitek for their kind acceptance of our invitation. It is our honour 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

Preface: SISNIR

The first edition of Wood workshop has been made at IVALSA-CNR in 2014. The event contributed to increase the NIR background on this interesting and active field.

On the basis of the success of the first edition, and taking advantage of the experience gained by Jakub and Anna Sandak and their network of scientists in this field, we are scheduling together the second edition.

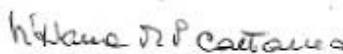
A new formula has been applied in scheduling the second edition, introducing a practical programme, the presentation of the latest developments of NIR instrumentation and dedicating more time to scientific presentations. Along three days communications will be dedicated to various aspects relating NIR with its application for the forestry, wood performance, modification and data mining. The keynote lectures will be presented by researchers having great experiences in varying scientific fields within NIR spectroscopy.

COST Actions FP1303 and FP1407 will support this year the organisation of the workshop, confirming the great interest and importance of this application field at the European and International level.

The “new” approach of this workshop is also highlighted by the use of round table formula to facilitate the interaction between participants and the exchange of experiences and ideas.

It is with pleasure that SISNIR supports this initiative with the aim to disseminate NIR knowledge and to promote cooperation between scientists.

SISNIR is very grateful to Jakub and Anna Sandak, the COST Actions FP1303 and FP1407, IVALSA-CNR, the Bruker Optics and VIAVI Solutions for their support to this valuable event.



Tiziana M.P. Cattaneo, President of The Italian Society for Near Infrared Spectroscopy – SISNIR

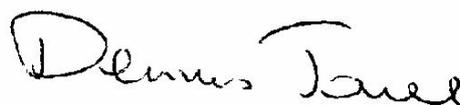
Preface: COST Actions FP1303 and FP 1407

COST Action FP1303 Performance of bio-based building materials and COST Action FP1407 Understanding wood modification through an integrated scientific and environmental impact approach are both addressing research topics that can significantly contribute to the societal challenges we are facing today. The climate changes we are fronting require immediate actions as well as new collaborative research and innovation activities. Bio-based materials and wood are recognized as the most important renewable material within the sustainable society and need to be applied not only to new applications, but to applications that we knew in the past but were replaced with fossil-based materials due to their low costs and short time superior performance.

Today the society and economy is well aware that also the environmental impacts in the whole life cycle of materials and products have to be taken into account when deciding for the materials in our surrounding, especially buildings where we spent most of our time. Although a lot is known on performance and characteristics of bio-based materials, there are still numerous questions science is searching for answer. At the same time the need for specific performance requests of bio-based materials is leading researchers in developing new processes, materials and applications. The NIR technique is a useful tool to support these research questions and developments. It can be used to detect chemical, physical, mechanical and anatomical properties of bio-based materials. Although NIR spectroscopic analysis is relatively new in the fields of wood science and technology, it is gaining importance in the characterization of wood as well as other bio-based materials.

This workshop, where researchers share their experiences, challenges and opportunities, will importantly contribute to the research work of Actions' members and goals of both Actions. It will ultimately lead to a greater acceptance of NIR as a powerful analysis tool for bio-based materials and aid in increasing the understanding of performance in service. We believe the workshop NIR spectroscopy will result in new collaborations and research that we are looking forward to learn more about at our future events.

We are wishing you a successful workshop with fruitful discussions.



Dennis Jones, MC Chair COST Action FP1303



Andreja Kutnar, MC Chair COST Action FP1407

Acknowledgment to COST

COST (European Cooperation in Science and Technology) is a pan-European intergovernmental framework. Its mission is to enable break-through scientific and technological developments leading to new concepts and products and thereby contribute to strengthening Europe's research and innovation capacities.

It allows researchers, engineers and scholars to jointly develop their own ideas and take new initiatives across all fields of science and technology, while promoting multi- and interdisciplinary approaches. COST aims at fostering a better integration of less research intensive countries to the knowledge hubs of the European Research Area. The COST Association, an International not-forprofit Association under Belgian Law, integrates all management, governing and administrative functions necessary for the operation of the framework. The COST Association has currently 36 Member Countries.

The Workshop "NIR and wood: sounds good #2" has been fortunate to be linked with two COST Actions: FP1303 "Performance of Biobased building materials" and FP1407 "Understanding wood modification through an integrated scientific and environmental impact approach (ModWoodLife)"

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:

COST FP1303

- Marina Cocchi, Italy
- Guillermo Palacios, Spain
- Anna Rozanska, Poland
- Antonio Ruano Sastre, Spain
- René Herrera Diaz, Spain
- Tom Moren, Sweden
- Zuzana Vidholdova, Slovachia

COST FP1407

- Ingunn Burud, Norway
- Dace Cirule, Latvia,
- Edgars Kuka, Latvia
- Emilia-Adela Salca, Romania
- Dick Sandberg, Sweden
- Bernhard Tapken, Germany
- Joris Van Acker, Belgium
- Wim Willems, Nederlands
- Andreas Zitek, Austria
- Vjekoslav Živković, Croatia

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General presentation of NIR technology in aspect of wood

Jakub & Anna Sandak

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The increased interest for application of NIR spectroscopy is observed in various fields, including forest and wood sciences. Vast amount of publication highlighted the potential of NIR in the characterization of wood in a wide sense. Journal of Near Infrared Spectroscopy published two special issues on wood, in 2010 and 2011, with a third number recently at the final editorial stage. Recently 548.000 results can be found in internet by searching "NIR & wood". Even if the technique seems to be well adapted to both scientific and industrial applications, there are still basic problems regarding instrument selection, correct measurement routines, and spectra interpretation. The most important limitation of near infrared spectroscopy is the complexity of NIR spectra that includes signals of overlapping overtones corresponding to different vibrating functional groups. The spectral resolution and measurement range of the modern spectrometers is still restricted, what complicates the spectra interpretation even more. Other factors, such as surface preparation, ageing, weathering and roughness may influence the spectra outline as well. Finally NIR is not self standing technology, which means that in order to predict material properties (chemical composition, physical/mechanical characteristics) reference method are indispensable. The goal of this presentation is to summarize state-of-the-art knowledge regarding recently available instrumentations, spectra acquisition and data mining in regard to forest/wood/bio-materials science and technology.

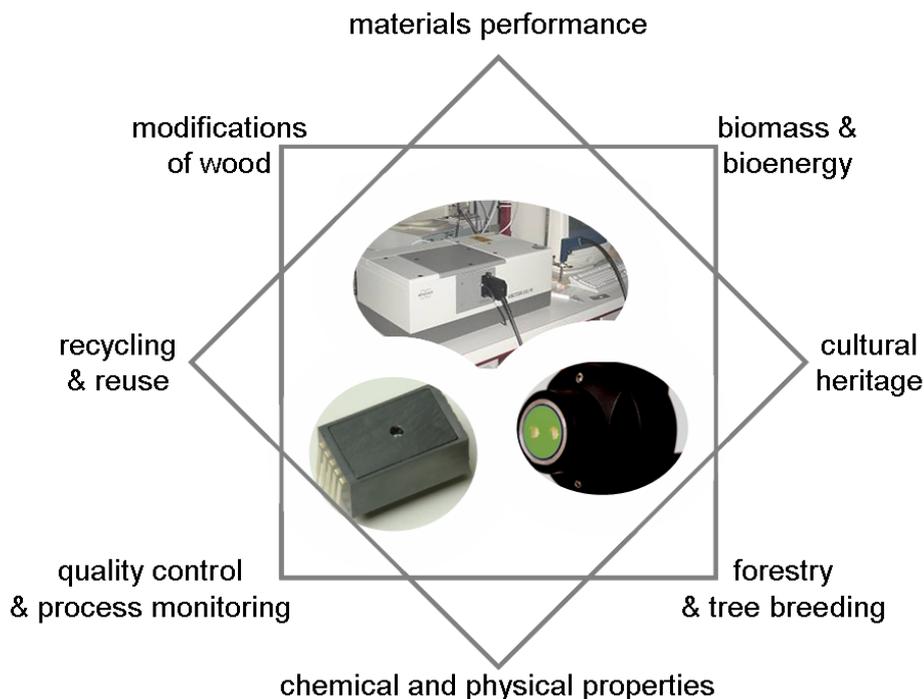


Figure 1. Major NIR spectroscopy applications within wood science and technology

Fit-for-Purpose MicroNIR Spectrometer

Emiliano Genorini

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Objective

The objective of this presentation is to provide an overview of the Viavi Solutions' MicroNIR™ Spectrometer family of the products and share application examples in the agriculture space. The MicroNIR spectrometer is the world's smallest near-infrared spectrometer suitable for on-line, at-line, and point-of-use applications in the field.

Introduction

The enabling technology of the MicroNIR Spectrometer is the novel thin-film linear variable filter (LVF) that separates the light into individual wavelengths (Figure 1).



Figure 1. Image of LVF

The LVF component is directly bonded to a multi-pixel indium gallium arsenide (InGaAs) detector array, which results in an extremely compact and highly reliable spectrometer with no moving parts. Additional information on the principle of operation can be found in reference [1]. All products cover the wavelength range of 950-1650 nm with excellent signal to noise ratio and photometric linearity making them suitable for classification or quantification applications in food, feed, agricultural, and pharmaceutical applications.

MicroNIR™ Pro

The MicroNIR Pro (Figure 2) is a general purpose analyzer that is equipped with user friendly software for data acquisition, method development, user management, and real-time prediction. The system also includes an embedded calibration development software based on CAMO's The Unscrambler® X (10.3.1) providing regression and classification algorithms and a complete set of chemometric tools for calibration model development and data investigation.



Figure 2. MicroNIR Pro

MicroNIR™ OnSite

The MicroNIR OnSite (Figure 3) is a ruggedized and ergonomic design of the instrument making it ideal for applications in the field or in a manufacturing environment such as raw material verification. It meets the IP65 standard for dust ingress and water spray protection. It is powered and controlled with a tablet computer-based software via a rugged USB connection and has an intuitive user interface that requires minimal training to operate. Additional software features are similar to those offered with the MicroNIR Pro.



Figure 3. MicroNIR OnSite

MicroNIR™ PAT

The MicroNIR PAT (Figure 4) is a compact, battery-powered and WiFi-equipped process analyzer. It meets the IP65 standard. It is ideally suited for end-point monitoring of a rotating blender, however, use on stationary processes is also possible. Powering from an AC power source as well as use of Ethernet connection for data communication are also possible and user-selectable. The spectrometer weighs less than 3 pounds (1.4 Kg) and is approximately 136mm x 136mm x 160mm in size. It contains an integrated 9-axis Inertial Measurement Unit (IMU) with gyroscope, magnetometer and accelerometer for consistent and reliable system triggering while installed on a rotating blender. The software includes real-time moving block standard deviation (MBSD).



Figure 4. MicroNIR PAT U (left) and MicroNIR PAT W (right)

Agricultural Applications

Several applications ranging from quantitative analysis of food and feed products will be shown demonstrating the high accuracy and repeatability of the instrument.

References:

1. N.A. O'Brien et al. "Miniature near-infrared (NIR) spectrometer engine for handheld applications", Proc. SPIE 8374, 837404–1-8 (2012).

Keynote 1: Shedding light on wood: time-resolved diffuse optical spectroscopy to characterize absorption and scattering

C. D'Andrea^{1,2}, A. Farina³, I. Bargigia², A. Nevin³, A. Pifferi^{1,3}

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Optical characterization of highly scattering media (or turbid media) is a novel research area which has found applications in many different fields ranging from biomedical optics (mainly for diagnostic purposes) to the assessment of fruit quality and pharmaceutical applications, just to mention few of them. We generally refer to this technique as Diffuse Optical Spectroscopy (DOS). In particular DOS is a bulk technique which aims at measuring both absorption and scattering spectra deeply inside the turbid medium. Wood can be considered as an isotropic highly scattering medium given by its complex system made of an intricate organized network of tracheids and vessels.

The basic configuration of DOS measurements consists of injecting light in one point of the sample and measuring the diffused light exiting from another point. Then by solving an inverse problem based on a mathematical model of photons diffusion, the optical parameters (absorption and scattering) can be recovered. By repeating the measurements at different wavelengths it is possible to obtain the absorption/scattering spectra while by changing the injection/detection points the imaging capability can be added. Generally, we refer to Diffuse Optical Tomography (DOT) when a 3D recovering of the absorption/scattering parameters is carried out.

Among different experimental DOS configurations, time-resolved spectroscopy (TRS) is quite promising because it allows one to separately recover the absorption and scattering properties by exploiting a single pair of injection/detection points. TRS basically consists of injecting a short light pulse into the medium and to temporarily sampling the light exiting the sample. In fact, changes of the absorption and scattering properties of the medium causes a modification of the temporal profile of the photons exiting the sample. It is worth reminding that absorption and scattering spectra are related to the chemical and structural composition, respectively, of the probe medium.

Recently, DOS has been successfully applied to the non-invasive in depth optical characterization of wood samples [1-4] and, in particular, we have proposed the use Time-Resolved Diffuse Optical Spectroscopy (TRS) in the near infrared range (700-1300 nm). First studies have been devoted to the basic characterization of different types of wood (e.g. hardwood and softwood) in different conditions (e.g. artificially degraded). Particular attention has been devoted to the study of waterlogged wood in order to correlate absorption and scattering variations with changes of moisture content both in a static and dynamic way.

Recently we have focused our attention to the non-destructive optical characterization of polymer/monomer uptake in wood samples. Also in this case the monitoring has been carried out both in a static and dynamic way in order to study the dynamical processes. In particular we improved the accuracy of the data analysis by accounting the changes of the refractive index during monomer uptake. We believe that the non-invasive monitoring of chemical compounds inside wooden samples can have important applications for both wood industry (e.g. polymer composites) as well as for monitoring of consolidation and conservation purposes in cultural heritage science. In particular the cultural heritage application of TRS technique for the consolidation and conservation monitoring of waterlogged wood will be discussed.

Finally, the technological aspects related to the development of TRS technique will be discussed. The rapid development of optical technologies, mainly fostered by optical communication and biomedical fields, allowed the design of novel devices (e.g. laser sources, detectors, optical components, fiber optics and wearable devices) which opens the way to the possibility to the

realization of compact, portable and low cost systems, which is fundamental to the application of TRS on the field or an industrial context.

References

1. C. D'Andrea, A. Farina, D. Comelli, A. Pifferi, P. Taroni, G. Valentini, R. Cubeddu, L. Zoia, M. Orlandi, A. Kienle, "Time-resolved optical spectroscopy of wood", *Appl. Spectrosc.* 62(5), 569 (2008).
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Keynote 2: Probe technology for in-situ NIR applications

Paolo Belloni

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NIR spectroscopy is one of the most powerful and non-destructive analytical technology. The use of remote probes allows a direct examination of large scale sample or process. Thanks to the use of NIR probes is no longer necessary carry samples to the spectrometer; fiberoptics cables can transmit NIR light up to distances of 100 m or more.

The principle is simple; the light of a halogen quartz source is coupled into a light fiber and transported to the probe. The probe is directly built into the process and allows the interaction of light to the matter. Three different kind of probe technology exists:

- Transmission probe (for liquid or transparent products)
- Reflection probe (for solid or non-transparent products)
- Transflection probe (for slurries, emulsion, semisolid products)

The interaction of light and matter generate different absorption depending on the composition of the sample. The modified light travels through a second light fiber back to the detector and the resulting absorbance spectrum is recorded.

In some cases light sources can be directly installed inside the probe body allowing non-contact measurement up to approx. 40 cm or more from the sample.

The quality of the fiberoptic used is a crucial parameter for the final spectra quality, in some case single fiber is sufficient, in case of low light flux, fiber bundle can be used to increase efficiency of light transportation.

Probe design, materials, dimension can be fitted to a lot of different process condition, including high temperature and pressure or the use in dangerous explosives areas.

References

1. Jörg-Peter Conzen and Tim Stadelmann (2004) Fiber Optic Probes for the Process Technology. Bruker Publication
2. <http://www.mypatprobe.com/>

Keynote 3: Near infrared hyperspectral imaging – background and application for wood characterization

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Hyperspectral imaging (HSI) in the near infrared has been recognized as a powerful technique for inspecting material surfaces, including wood, providing spatially resolved spectral information on chemical constituents. The term “hyperspectral” hereby refers to the number of wavelengths collected per pixel being larger than 10, the term “imaging” to the spatial, image-like representation of the surfaces. The advantage of hyperspectral imaging as compared to conventional spectrophotometers lies in the ability for remote sensing of surfaces. The result of hyperspectral imaging is the so-called “hypercube” that can then be processed by multivariate statistics to gather information on spatial differences in the chemical composition of the surface. HSI systems can be classified according to their measurement principle in whiskbroom - (mapping), staring - (2D wavelength scanning) and pushbroom - (line scanning with spectral dispersion) imaging, with pushbroom imaging being the most commonly applied principle.

A push-broom type hyperspectral imaging system typically consists of several components that need to be carefully interlinked to deliver the required performance: a camera including a matrix-detector able to obtain spectral and spatial information at the same time, a spectrograph mounted in front of the camera and dispersing the light of the inspected line into spectra, an objective lens for focusing the light beam, a lightning source delivering light in appropriate wavelengths (usually halogen-tungsten lamps for NIR studies), a y-table, and a computer with a dedicated software to interlink all hardware parts and control the whole imaging process. All components are usually mounted on a frame construction. The height of the spectrograph above the sample hereby defines the field of view, and by this, the spatial resolution that can be achieved (field of view divided by the number of pixels available on the detector).

Important issues to be considered before any useful measurements can be made, is an optimized lightning, the calibration of the camera itself including optimal cooling of the sensor to reduce the noise level and the setting of the acquisition parameters (e.g. integration time), the spatial and spectral calibration of the spectrograph, the adjustment of the objective lens to retrieve a sharp image, the reflectance calibration (white and black reference). Only if these parameters are considered carefully, useful hypercubes can be generated. These hypercubes then have to be preprocessed (removal of dead pixels, normalizing, segmentation of ROI area etc.) before multivariate statistics and image analysis can be performed.

This keynote lecture will give a general introduction into hyperspectral imaging, and highlight the relevant mechanical and technical features of a NIR hyperspectral pushbroom imaging system, including a basic introduction to the steps for system calibration and to the pre-processing and multivariate analysis of the hypercubes.

Using NIR spectroscopy for the discrimination between *Eucalyptus nitens* and *E. globulus*

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Introduction

Absorbance data from 780 to 2500 nm obtained from a NIRS instrument can be employed to conduct quantitative (i.e. chemical compounds prediction) and qualitative (i.e. classification of different quality groups) analysis of a specific product. Both analyses need the development of a multivariate prediction model to relate spectral data to quantitative attributes [1]. Among the advantages attributed to the NIRS technology, it can be highlighted the high response speed, the fact that it is a nondestructive method, with a low analytical cost per sample, with little or no need for sample preparation. Also, it is a multi-product and multi-attribute technique and the same instrument is suitable for different products and analytical parameters, is not necessary the use of chemical reagents and no polluting waste is generated, being able to predict chemical, physical and sensory attributes [2]. In the literature there are different successful studies in which the NIRS spectroscopy is applied to study different parameters in wood products. In particular, the NIR spectroscopy has been used for discrimination between different species [3], for the prediction of chemical parameters such as lignin, cellulose and extractives content [4], for determining characteristics associated with temperature effects [5], to measure wood density [6], among others. The objective of the present research was to evaluate the feasibility of the use of a handheld instrument based on NIRS for the discrimination between two different species of *Eucalyptus*, namely *E. nitens* and *E. globulus*.

Methodology

A total amount of 848 samples were analyzed of which 580 belonging to the species *globulus* and 268 samples to the species *nitens*. A handheld MEMS (micro-electro-mechanical system)-based NIRS digital transform spectrometer (Phazir, Polychromix Inc., Wilmington, MA, USA), working in reflectance mode in the spectral range 1600–2400 nm was used. Sensor integration time was 600 ms. Statistical treatments were carried out using the software Matlab 2015b (The Mathworks, USA) and the software PLS Toolbox (Eigenvector, USA).

Once imported the data, we proceeded to the spectral pretreatment in order to obviate spectral differences between samples due to physical characteristics (density, porosity, etc.). The combination of pretreatments was Detrend, second derivative and Mean Center. Multivariate prediction model was built using the algorithm PLS-DA (Partial Least Square Discriminant Analysis) that captures the spectral variability and produces correlates reference data. In this case, the reference data correspond to information on the membership of each sample to the species *nitens* or species *globulus*. The classification error was calculated through cross-validation and subsequently by predicting an external group of 124 samples excluded from the calibration set. The quality of the models was evaluated using different parameters: sensitivity (fraction of true positives or TP), specificity (fraction of true negative or TN), Matthew's correlation coefficient [7] and classification error in cross validation.

Results

Figure 1 shows the prediction of the samples classified as *globulus* and *nitens*.

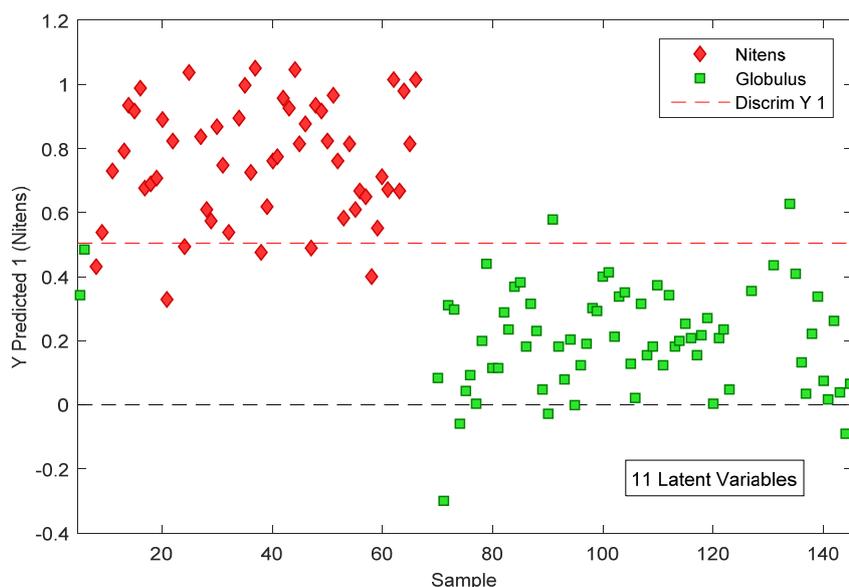


Figure 1. Estimated class values for discrimination of the class *nitens* from class *globulus* and (- -) discrimination threshold

From the figure can be seen that 48 samples out of 50 were correctly classified as *nitens* and 68 out of 74 samples were correctly classified as *globulus*.

Conclusions

In this study, the model built from the spectrum of 500 samples has been evaluated through the prediction of 124 "unknown" samples. The average prediction error obtained was 7 %. The study has confirmed the feasibility of NIRS as a fast and nondestructive method for the discrimination between different species of Eucalyptus. One of the main limitations encountered is the difficulty of including samples with very high humidity into the model. The water present in the samples has a high absorbance in the NIR range and this phenomenon doesn't allow the discrimination between different species of Eucalyptus.

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Detection and spectral characterization of resin pockets in spruce by FT-NIR and near infrared hyperspectral imaging

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Resin pockets in spruce represent an important characteristic of wood, impairing the wood quality according to the existing European grading norms. New approaches taking into account the full spatial dimension of a wood log like hyperspectral imaging are able to quantify the area and amount of resin pockets on wood logs. Especially the NIR spectra are promising for the detection of resin pockets in spruce. However, NIR spectra of spruce resin have not been studied in detail so far.

This presentation provides information on the spectra of resin from spruce collected by FTNIR and the application of NIR hyperspectral imaging (900 - 1700 nm) using a push-broom system for the detection and classification of resin pockets on freshly cut wood logs.

Application of near-infrared spectroscopy to determine the juvenile–mature wood transition in Spanish conifers used in construction.

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Wood has been by far, the most used and older building material because of its strength workability and availability. It has being replaced by new materials, but none of them it's renewable, has the lowest carbon footprint when they are processed and can be used as a carbon sequestration method, as they sunk atmospheric CO₂ while growing. Despite this, one of the biggest problems of wood used in construction is its variability. Wood is not homogeneous and its physical, chemical and mechanical properties vary a lot among species, within the same species and even along each tree.

There are important changes along the stem in radial (pith to bark) direction and vertical (axial) direction, even between earlywood (springwood) and latewood (summerwood) and along the length of the same ring. This variation in wood properties is produced by several factors interacting with the tree, like climate (rainfall, temperature, soil moisture...), site (orientation, slope, soil...), silviculture (size, stand density, crown size and position, fertilization, pruning, age of harvest...) and genotype among others [9, 10, 3, 14, 15]. Nowadays due to the trends of harvesting using shorter rotation, an old problem has focused more and more attention; the Juvenile Wood (JW).

The Juvenile Wood can be defined as a still imperfect, wood present in the inner part of the bole near the pith, in which wood characteristics undergo rapid and progressive changes in successively older growth rings. Although this definition it's true, we have to emphasize that it has been called by different names depending on tree position or development: corewood, crown-formed wood, crown wood, inner wood...; because JW from the upper part of the bole differs to the lower part, this is due to the "physiological aging" [8, 9, 10, 11, 14, 19]. It is characterized by having a cellular structure with lower tracheid length, lumen diameter and wall thickness; lower densities, specific gravity, transverse shrinkage, stiffness and cellulose/lignin ratio; higher spiral grain and microfibril angle; and usually wide growth-rings and high levels of compression wood, although if the last two are not present it doesn't mean it is not juvenile wood or vice versa.

All these variations have deep negative effects on physical-mechanical and technological properties of the material [6, 9, 15, 19] and also can result on different drying distortions as spring, bow, crook and twist (Figure 1); resulting on a lower possibility of final use and economic return in the wood industry.



Figure 1. Problems related with the presence of Juvenile wood

Usually, it has been used basic specific gravity and density to determine the JW extent, because it's easier than other techniques. Also the more accurate measures with micro X-ray densitometry and microscopy is another reason along with that density it's highly related with the wood physic properties, being a great predictor of the modulus of elasticity and rupture (MOE and MOR) [14]. But there are also other techniques like microfibril angle that it's used in many papers, and less common, tracheid length and wall thickness. In our case, we'll be using micro X-ray densitometry to adjust a model to set the juvenile-mature wood transition point.

As the micro X-ray densitometry method is laborious and time-consuming we are going to evaluate the potential of Near Infrared Spectroscopy (NIRS) for identifying juvenile and mature wood zones in wood boles samples. For this purpose quantitative calibration models will be developed for predicting density from the latewood using micro X-ray densitometry as the reference method in order to obtain the transition point. Also we are going to assess the possibility of developing a qualitative model for identifying juvenile or mature wood which would not be easy since there is an area called transition wood, in which wood is still changing so there should be an overlapping area difficult to differentiate.

Cores and disks taken along the stem will be scanned using a Fourier Transform Infrared Spectroscopy (FT-IR) BRUKER MultiPurpose Analyzer equipped with an external optical fiber probe with 3mm of path length, a spectral resolution of 2cm⁻¹ and a spectral range that varies from 780 to 2.777 nm. In order to scan only the latewood part of the ring a black methacrylate sheet with a hole will be used to reduce the measured area.

Since the scanned section, the spectral range, the sample preparation the moisture content and the number of PLS latent variables vary a lot among the previous studies [2, 4, 7, 12, 13, 16, 18] we are still assessing this parameters. The samples will be acclimatize in a climatic chamber at 20°C and 65% RH, so the wood moisture content should be homogeneous around 12,1% when measured.

About the scanned section, for density it's better to measure in a transversal way because it has more variation and higher signal [1]. Although the micro X-ray densitometry method was done using the radial direction, Giroud et al. [5] doesn't take it into account and measured in a transversal way based on research [17], who concluded that the differences between the two directions are small.

Although the original idea was to use multiple spot NIRs, we've been searching information about near-infrared hyperspectral images and found out a lot of promising information. So we're weighing the idea of including this method using a processing image program, to select the part of the image that interests us, then calibrate and compare results with the original method.

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Biomass characterization towards contaminants

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Abstract

The biomasses are a strategic energy source for European countries since they are locally produced, renewable, carbon neutral, exploiting agricultural and forestry residues, able to active rural communities, etc. Nevertheless the few critical issues related to the thermal exploitation of biomasses should also be mentioned, such as the particle and gaseous pollution normally produced by high quality biomasses even in efficient and high-tech plants, and the additional pollution due to the quality of ordinary and/or unsorted biomasses.

Monitoring the presence and the concentration of unwanted pollutants, particularly preservatives and hydrocarbons, is fundamental for an accurate characterization and selection of the best management of wood biomasses.

The primary goal of this research is the control of most common hydrocarbons in wood residues and biomass. The main benefits are: sorting the polluted from non-polluted wood; sub-selection of polluted residuals in order to sort each category in an appropriate way; reduction of recycling of dangerous pollutants; profitable use of non-contaminated wood.

This goal is reached by using FT-IR-ATR technology by creating a spectra database of pollutants and by monitoring the occurrence, typology and concentration of unknown polluted wood residues. The biomass pollutants database under construction is constituted by oils and fuels.

Goals

The primary goal of this research is the control of pollutants in wood biomasses from both forest-wood chain and industrial processes. According to the current laws, the biomasses harvested in forest are labelled as "virgin", meaning that no contaminations or relevant pollution are expected; also the by-products of solid logs sawmilling are normally labelled as above, even if coming from an industrial process. But other wood industrial processes can be critical towards the potential contact among wood and pollutants; it is the case of inappropriate stoking or manipulating or transport processes. Moreover, even the "virgin" timber harvested in forest could accidentally get in contact with unexpected pollutants.

Therefore our goal is to discriminate the proper virgin wood from the polluted wood, limiting the investigation framework towards the hydrocarbons pollutants.

Testing specimens and testing method

After a series of preliminary testing sessions, we realized that the concentration of the reference pollutants was a troubling issue. In fact providing different amount of reference pollutants onto a wood surface did not provide different concentrations, but simply larger polluted areas of the specimens with the same concentration.

Therefore we reduced the wood into thin powder through successive mechanical chipping and grinding processes and then by sifting and sieving, in various (4) sieving particle sizes. Same amount of such wood powder were added by rising amount of reference pollutants such as lubricating oil, gasoline and diesel fuel in the mass ratio of 1:1, 1:2 and 1:10 (regardless to the mass losses of the evaporation of the most volatile compounds).

The reference specimens "wood powder + given concentration of chosen hydrocarbons" were mixed up and then measured on a Bruker ATR-MIR with a diamond crystal measurement window; note that for this particular test the original zinc selenide was initially damaged by the footprint of the small wood particle pressed on the window. Each specimen was then measured with a scanning sessions of 60 seconds, three times repeated.

Conclusions

The principal components analysis (PCA) provided the results shown in Figure 1. The MIR spectra are able to discriminate the occurrence of lubricating oil, gasoline and diesel fuel in the mass of wooden media. Moreover the signal of these hydrocarbons lasts along the time and even 20 months after the contamination, some differences were still ongoing.

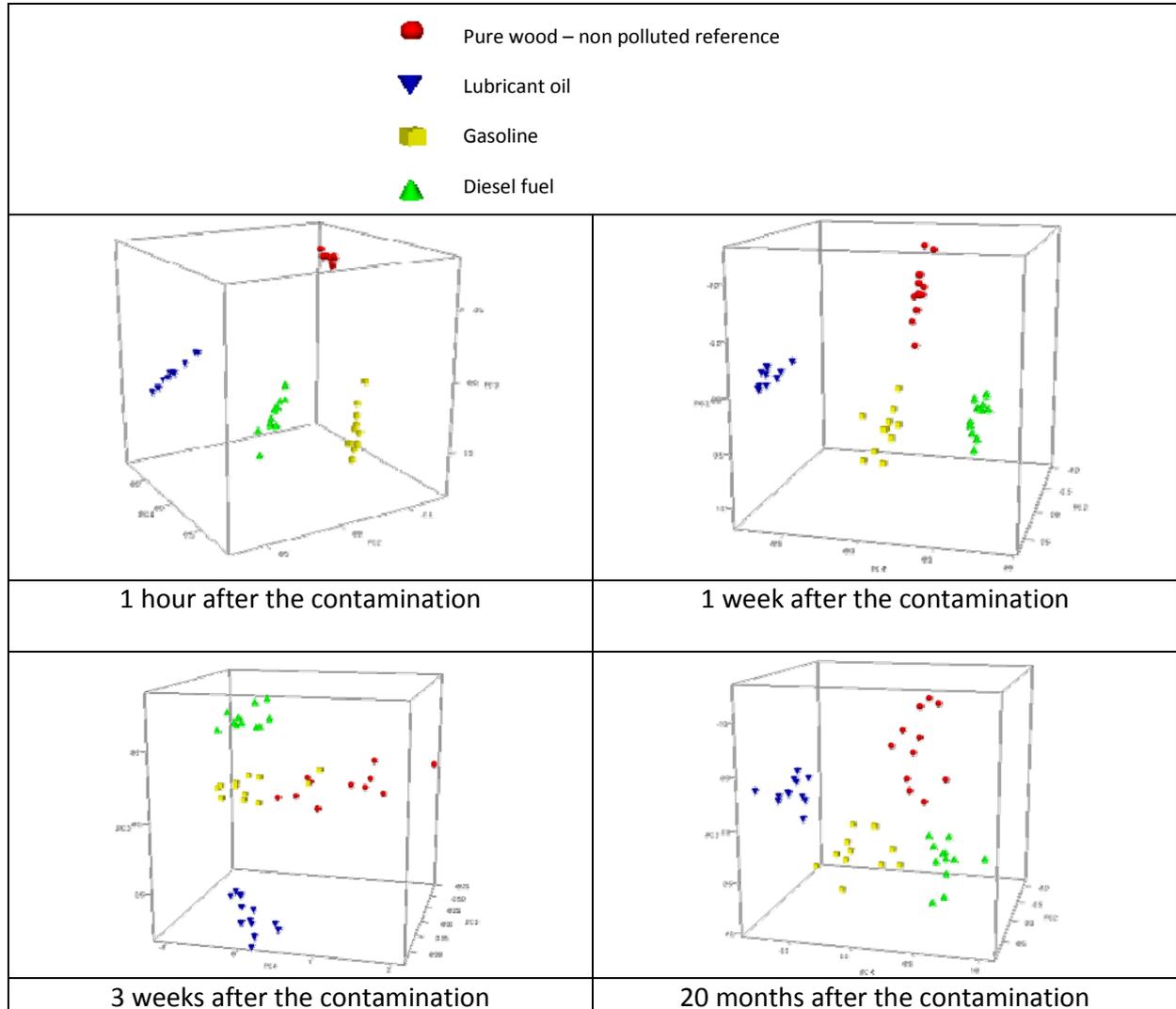


Figure 1. The PCA discriminated the occurrence of lubricating oil, gasoline and diesel fuel in wood

Characterization of Beech (*Fagus sylvatica* L.) and Turkey Oak (*Quercus cerris* L.) wood supported with FT-NIR

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Abstract

Almost 50% of Kosovo's land is covered by forest. The varied elevations, climatic influences and diversity of soils within the country provide a wide variety of micro-habitats to which tree species are adapted. The annual amount of felling is currently slightly below 1 million m³, with beech and oak being the main species [1].

European beech is a species widely spread across Europe, and its wood is highly appreciated by the wood industries due to its hardness, wear-resistance, strength, and excellent bending capabilities. As beech wood is of relatively low price, it makes this resources commonly used for various applications.

Turkey oak is native to an area from south east France across to the Balkans and Turkey. It is commonly used as firewood, as the timber splits and/or warps during seasoning. The raw wood has relatively little market value; therefore several modification processes are developed recently in order to improve its properties [2].

Comprehensive valorisation of bio-resources is essential in order to assure the optimal use of wood and rural development in underdeveloped areas. Novel analytical tools may be of a great advantage to assist such sustainable way of resources usage. Presented work is an attempt to demonstrate usefulness of NIR spectroscopy toward characterization of the raw materials originated from Kosovo. The preliminary approach was focused on the automatic recognition of two (beech and oak) wood species. Figure 1 presents an outline of the averaged second derivative spectra of both species. It can be seen that slight difference in spectra can be noticed, especially in -CH groups of lignin (band 11 and 12), and -OH groups of cellulose (band 16). It confirms that beech and oak slightly differs chemically and that NIR spectroscopy is capable of detection even such minor differences in the chemical composition.

Principal components analysis (PCA) and Identity test were additionally performed on the obtained spectra in order to evidence the capability of NIR measurement system to discriminate both species. It is apparent on Figure 2 that beech and oak were clearly separated and created two not overlapping clusters. Thanks to encouraging preliminary results, a further work is foreseen to be taken under common collaboration. It will be dedicated to analysis of same wood material with complementary methods and to predict other material properties. Other wood species are also considered for evaluation with the overall goal of supporting local wood industries and to assure optimal utilization of native wood species in Kosovo.

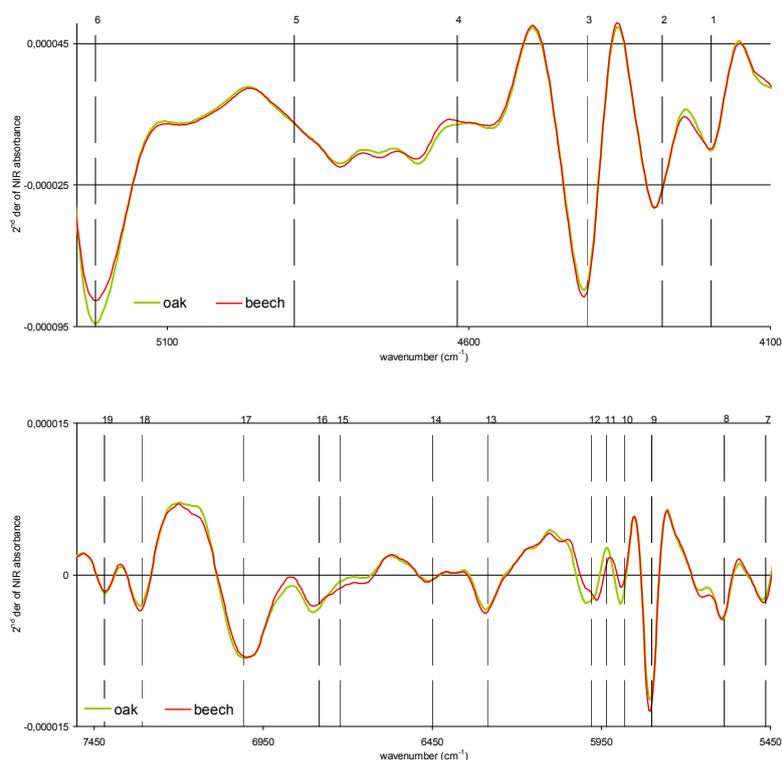


Figure 1. Second derivative of NIR spectra of beech and oak wood

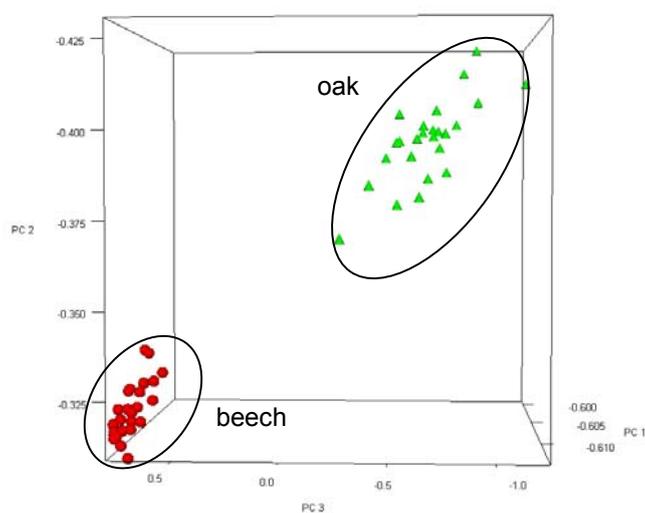


Figure 2. PCA discrimination of beech and oak wood with NIR

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Identification of the origin of Big- leaf mahogany wood from five Latin American countries via NIRS and PLS-DA.

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Introduction

The Big-leaf mahogany (*Swietenia macrophylla*) is a woody forest species that occurs naturally from North to South America starting in Mexico and ending in Bolivia and South of Brazilian Amazon [1]. Its wood has several desirable features such as beauty, workability and moderate resistance to pests. After decades of extensive selective logging and predatory exploration *S. macrophylla* (along with its congeners *S. humilis* and *S. mahagoni*) was included on Appendix II by the Convention on International Trade in Endangered Species - CITES as a species with potential risk of extinction. Thereby, the wood extraction is now gaining increased regulatory protection. The wood of the big leaf mahogany is often confused with *Swietenia humilis* and *Swietenia mahagoni* in Central America countries where it was found even hybrid species. In Brazil, there are many native species whose woods are visually similar to *S. macrophylla*, and there is evidence that *S. macrophylla* has been smuggled under the guise of other species. Thus wood species identification procedures are essential to avoid illegal exploitation and trading and to ensure conservation. To botanically identify a species it is necessary to look at all its features including leaves, fruits, seeds and flowers. However, in everyday practice of timber species identification, the wood has already been logged and typically only boards are available. So, alternative methodologies are needed for correct wood identification. A method currently employed for this purpose is a combination of macroscopic and microscopic identification, where many wood characters such as density, color, smell, brightness, texture, growth rings, vessels, fibers and porosity of an unknown sample are compared with candidate species. When an expert wood anatomist is available to perform the identification this method can provide very forensically reliable results. Unfortunately, there are not enough experts to meet the demand. Therefore, the development of technological methods for wood identification that not require specialist knowledge can help to improve field-level inspections and identification as well as forensic level wood identification.

In this paper, Near Infrared Spectroscopy (NIRS) and Partial Least Squares for Discriminant Analysis (PLS-DA) were applied to identify the mahogany origin from five American countries: Bolivia (BOL), Brazil (BRA), Guatemala (GUA), México (MEX) and Peru (PER). Besides alleged identity, all samples were identified by a wood anatomist from the Forest Products Laboratory from Brazilian Forest Service. Small wood blocks totaling 279 were used for this analysis, being 79 from México, 38 from Peru, 28 from Guatemala, 99 from Brazil e 35 from Bolivia. Sample surfaces were sanded with P80 grit sandpaper in order to homogenize samples. The grain direction (longitudinal, radial, and tangential) was at random. Each block sample was scanned three times, on different spots. Diffuse reflectance measurements were made in absorbance scale ($\log(1/R)$) using the portable devise MicroNIR 1700 with 6.2 nm of resolution and 908 to 1672 nm spectral range [2].

Data analysis was carried out in Matlab 7.12.0 (R2011a) software with PLS_toolbox 6.5. Spectra were preprocessed with first derivative using Savitzky-Golay smoothing (13 points and second order polynomial). Data were mean centered and replicate spectra were not averaged. PLS-DA

calibration models were built using two thirds of the samples, selected from the 279 block samples using the Kennard-Stone algorithm. For each country a specific PLS-DA model was developed.

Results and Discussion

Table 1. Figures of merit data for Big-leaf mahogany discrimination by origin.

Figures of merit*	Country				
	BOL	BRA	GUA	MEX	PER
FPR (%)	0	6.7	2.6	4.9	0.4
FNR (%)	0	4.2	0.0	1.3	0.0
EFR (%)	100	89.1	97.4	93.8	99.6

*FPR: false positive rate, FNR: false negative rate and EFR: efficiency rate

Verifying the results in Table 1, it can be observed that each country presented a high efficiency rate (EFR) ranging from 89.1 to 100%. Brazil was the country that showed the lowest EFR, where the false positive (FPR) and negative rates (FNR) were very close. Some factors that can explain the lower efficiency observed for Brazil is its large territory and ecological diversity.

In addition, there is a great variability of the set of samples originating from different regions of the country and measured in field conditions. Mexico and Guatemala had high values of EFR, despite similar natural conditions and territorial neighbourhood between these countries (Figure 1).

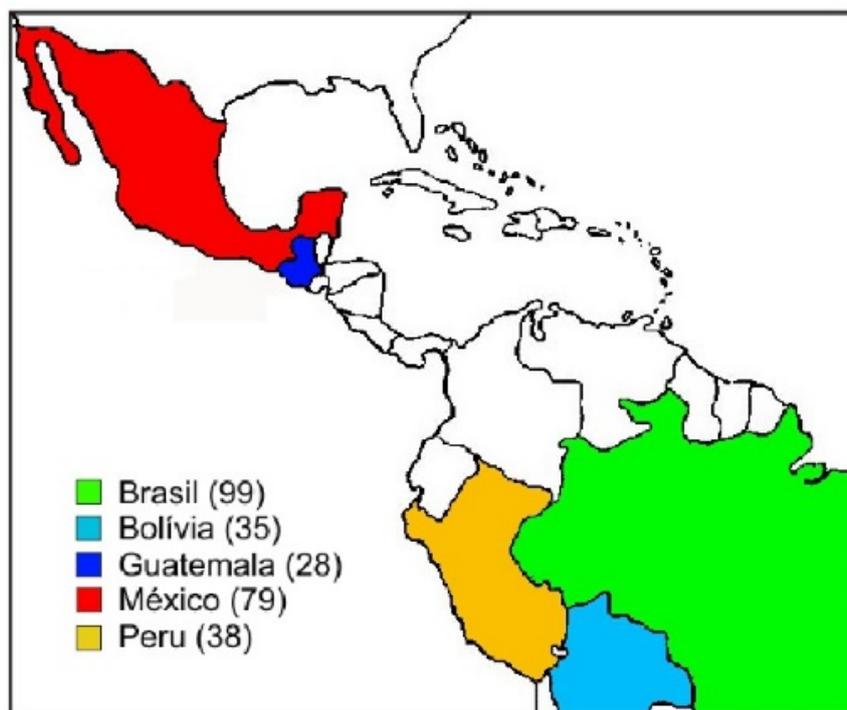


Figure 1. Map of America with the number of samples by country

Conclusions

The method developed with NIRS and PLS-DA offers exact and precise results for the identification of the origin of *Swetenia macrophylla*. The origin identification from five countries was possible using a very simple sample preparation and small blocks of wood. The efficiency for the used portable device MicroNIR's and the PLS-DA developed models for this wood were demonstrated. It is noteworthy the technical potential: it is possible to expand the model to other mahogany producing countries and use it in control and inspection activities.

Acknowledgements

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SPIRMADBOIS project: Using NIR spectroscopy for a sustainable forest resources management in Madagascar

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Forests in Madagascar are rich in wood species and exhibit a remarkable endemism rate. However, forest loss is a major environmental issue mainly driven by land conversion for agricultural purposes, the growing domestic demand in wood products and the illegal trade of precious wood for international markets.

Forest sustainable management requires a better knowledge of forest and wood properties and uses as well as specific tools to deal with. Unfortunately, few studies have been done regarding properties of Madagascar timber species. Only 200 out of the 4000 existing species have been described to date in terms of their mechanical, durability and physical properties. Some wood species like *Dalbergia* sp. (48 species) cannot be identified from log observations. A quick and reliable method to estimate wood properties and tools to ensure traceability for legal timber control purpose are therefore essential to improve the management of Madagascar's forest resources.

This paper introduces the SPIRMADBOIS project that aims to contribute to the sustainable forest management, plantations and natural forests alike, by providing tools based on NIR spectroscopy analysis. Specific objectives to use NIR spectroscopy are:

- to estimate wood properties of one plantation species (*Eucalyptus robusta*),
- to determine geographical provenances of *Eucalyptus robusta* from Madagascar using wood and/or leaves samples,
- to estimate wood properties of 60 natural forest species,
- to distinguish between rosewood and palissander (both belong to the *Dalbergia* genus) using wood samples.

This paper will present on-going progress focused on NIR spectroscopy. By participating in the "Application of NIR spectroscopy in wood science and technology" workshop, we grasp opportunities to create an efficient network amongst NIR specialists, to exchange experiences and to discuss our preliminary results.

NIR spectroscopy as a tool for in-field determination of log/biomass quality index in mountain forests – SLOPE project approach

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Current in-field methods for logs grading are based on visual rating, which is operator-dependent, time-consuming and subjective method. Different wood defects such as knots, resin pockets, rot, compression wood, among others, affect the quality and potential usage of the log. An early detection of these defects and an adequate wood quality classification helps to optimize the resource use along the whole production chain. Therefore, the specific target for the development of an efficient in-field grading approach was defined within the project Integrated processing and control systems for sustainable forest production in mountain areas – 604129. The grading is conducted by means of automatic measurements of selected wood properties with diverse sensors, including NIR spectrometers. Series of tests were conducted on wooden discs by means of laboratory equipment (FT-NIR spectrometer) and by a portable device (MicroNIR). In-field measurements on standing trees and harvested logs were also performed using MicroNIR. PCA models for identification of logs defects were developed on the base of the spectra collected with both, portable and laboratory instruments. Such models will serve for the automated determination of quality indexes to be used for log grading. It is foreseen that the NIR quality indexes will be integrated with the expert system under development within the SLOPE project and combined with quality information derived from other sensors. The overall goal is to provide a reliable technology for automatic log quality grading advancing recent practices present in the forest industry.

Acknowledgments

This work is conducted within project SLOPE receiving funding from the European Union's Seventh Framework Programme for research, technological development and demonstration under the NMP.2013.3.0-2 (Grant number 604129).

Keynote 4: Biomaterials characterization with NIR

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The trend for rapid deployment of novel/advanced material solutions at reduced-costs through predictive design of materials and innovative production technologies is observed nowadays. Such materials are optimized for specified applications, assuring at the same time expected properties and functionality at elongated life, minimizing the environmental impact and reducing risk of product failure. As a consequence, higher numbers of well performing (also in severe environments) materials are available on the market. In the same time new developments in the field of wood modification offer innovative products with enhanced properties of natural. These include novel bio-based composite materials (fiberboards, particleboards), as well as more effective and environmentally friendly protective treatments, e.g. thermal treatment, densification and chemical modifications (acetylation, furfurylation). The same revolutionary progress is observed with surface treatments including innovative coatings, impregnations or integration of developments of nanotechnology for wood protection.

Biomaterials due to its biological nature can undergo alteration during their service life, that can be caused by mechanical, environmental or biological agents (bacteria, fungi, insects). The lecture presents an overview on the current applications of near infrared spectroscopy applied for the characterization of wood and wood-based products and evaluation of their degradation during time. A state-of-the-art report is complemented by results of original research carried out by presenter.

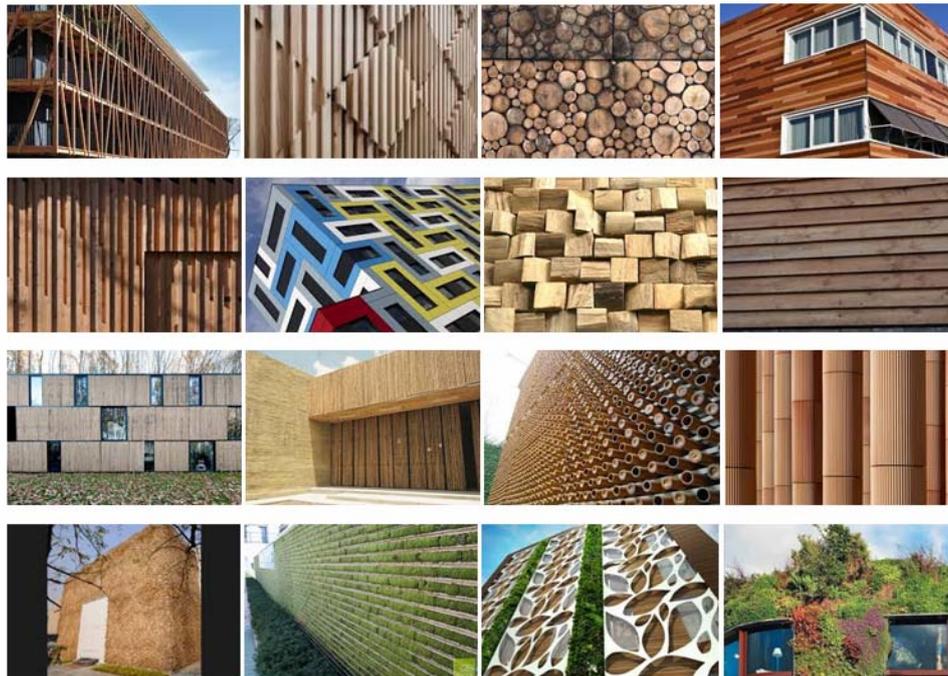


Figure1. Various biomaterials used in for building facades

Estimating the weight percent gain of modified wood with near infrared and Raman spectroscopy.

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Wood modifications getting more and more recognized to improve the durability and dimensional stability of non-durable wood. In the production of modified wood, a certain Weight Percent Gain (WPG) is targeted which must be controlled in the running process regularly. Near-infrared (NIR) and Raman spectroscopy could serve as a useful tool of WPG determination and replace costly chemical analysis method. Therefore the aim of the PhD's is to investigate if it's possible to predict the WPG of known wood modifications with NIR and Raman spectroscopy. The targeted wood modifications are acetylation, furfurylation, phenol resin, melamine- and DMDHEU-treatment.

Wood samples (30x30x6 mm) of pine (*Pinus sylvestris*) and beech (*Fagus sylvatica*) are treated with the different modification chemicals. Ten different WPG steps are adjusted (4, 10, 16, ..., 52, 58). 8 radial and 8 tangential specimens are modified for each WPG step. All specimens are measured in conditioned state (20°C; 65% RH) and oven dried (103°C). Both surfaces of each specimen are measured, so that for all specimens two spectra are recorded. The infrared spectra is measured with a spectrometer from Polytec (System 1750), Germany, with a wavelength range of 845 to 1.654 nm. The measurements were taking 2 seconds with an accumulation of 10. The Raman measurements are pending.

The data analysis is done with the software Unscrambler X from Camo, Norway. The spectra are normalized by SNV (Standard Normal Variation) and then a PLS regression (Partial Least Square) is performed.

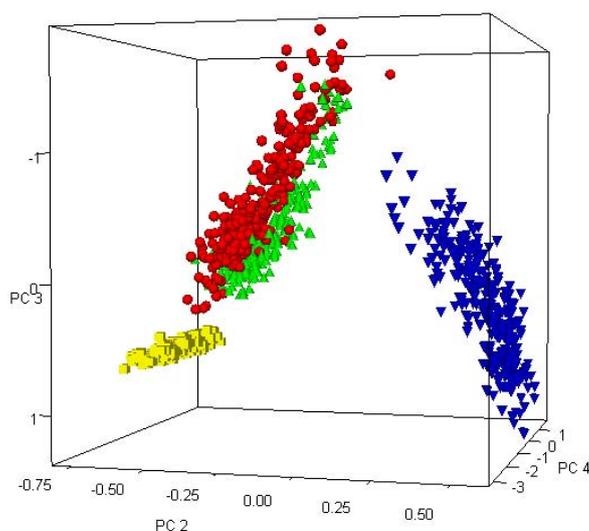
The first results for the phenol resin treatment are promising, but further improvements are needed.

Evaluation of wood veneers for furniture production by FT-NIR

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The case study reports collaboration with industrial partner who requested evaluation of four types of oak veneers. The set of samples included natural oak (sample code: 3) and three veneers with similar - dark appearance (sample code: 1, 2 and 4). NIR spectroscopy was used for analysis of the chemical composition in order to discriminate investigated samples. The main objective was to create data base of NIR spectra that might be used for future discrimination of veneers with similar appearance, but of different technological properties. Totally 600 samples (150 wood veneers of each type) were measured with VECTOR 22-N produced by Bruker Optics GmbH. The spectral range measured was between 4000 and 12000 cm^{-1} . Each spectrum was computed as an average of 32 successive measurements, in order to minimize the measurement error. Two separate spectra were measured on each sample. As a result, a data base of 1200 spectra (300 of each veneers type) was created. The PCA model presented in Figure 1 was built on the base of raw NIR spectra covering the whole spectral range (11500 - 4100 cm^{-1}). It can be seen that separation of the samples 3 and 4 (natural oak) from the others is most evident. On the other hand, some overlapping of clusters corresponding to samples 1 and 2 is noticed.



**Figure 1. PCS scores of investigated veneers in the spectra range from 11500 to 4100 cm^{-1} (no spectra pre-processing, method: factorization (3 factors)).
Samples coding: ● 1, ▲ 2, ▼ 3, ■ 4**

After confronting the results with industrial partners it was concluded that both veneers from group 1 and 2 were obtained from archaeological/waterlogged oak logs, but of different provenance. On the other hand, the clearly discriminated veneer of sample 4 was a contemporary wood prepared industrially to imitate a darkened oak. Developed data base might serve in the future for fast and non destructive comparison of veneers, and might be used for quality control in industrial applications.

Application of NIR for evaluation of weathered samples from RR test

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Wood exposed to weathering is degraded by various environmental agents such as solar radiation, cyclic wetting, atmospheric temperature and relative humidity changes, environmental pollutants and certain micro-organisms. The rate of weathering varies within timber species, but depends most of all on the microclimatic conditions. There is a lot of research dedicated to studies on the weathering mechanisms of diverse materials. However, the comprehensive understanding and detailed models are hardly available for weathered wood surfaces. This research is a part of activities within the Round Robin test, the common initiative of the COST Action FP1006, where 28 sets of samples were exposed in 18 locations in Europe and Brazil (Figure 1).



Figure1. RR test locations

Two types of experimental samples (“thin” and “thick”) were prepared from one log of Norway spruce tree (*Picea abies* L.). The efficient surface exposed to weathering was 30 x 35mm (width x length respectively). The thickness of samples was ~100 μm in case of short weathering (thin) and 5 mm for long weathering (thick) samples. One piece from the thin samples set was collected after 0, 1, 2, 4, 7, 9, 11, 14, 17, 21, 24 and 28 days of weathering. Long term weathered sample was collected in a monthly base, assuming total exposure for twelve months.

The list of surface characterization methods applied for assessment of RR test samples includes: colour CIE Lab, VIS, NIR and MIR spectra, imaging, gloss, XRF and roughness.

Two alternative NIR spectrophotometers were selected and intensively tested with this work, including laboratory and portable devices. Near infrared spectra were acquired by FT-NIR spectrometer Vector 22N (Bruker GmbH, Ettlingen, Germany) in the spectral range from 12.000 to 4000 cm^{-1} . Fiber optic probe was used for the illumination sample and for the collection of the reflected light. The diameter of the probe was ~3.5 mm and corresponding measured area was ~10 mm^2 . The compact sensor MicroNIR 1700 (VIAMI, Santa Monica, USA) was used for complementary measurements. Each spectrum was measured as an average of 10 consecutive scans. The scanning frequency was 50Hz, corresponding to 20 ms of integration time. The spectral range was from 10526 to 6060 cm^{-1} .

NIR spectroscopy was used for identification of the chemical changes in wood due to weathering. Besides routine spectra interpretation, calculation of weathering index was performed by regressing two points defining the border values for the weathering state. Such algorithm allowed direct comparison of weathering location on the intensity of wood degradation.

Acknowledgment

This work has been conducted within the Round Robin test initiative of the COST FP1006, STSM funded by COST FP1303 and project BIO4ever funded within a call SIR by MIUR (RBSI14Y7Y4). Special thanks to all participants of the RR test.

Weather degradation of thin wood samples assessed with NIR spectral imaging

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Introduction

Untreated wood is frequently utilized as cladding in modern buildings and there is a trend moving towards less surface treatment of the wood to obtain a naturally weathered result. The elements of façades made of wood often present non-uniform degradation patterns. Various architectural solutions are generally the main reasons for heterogeneous appearance since these affect the microclimate on the surface. The microclimate on a wooden façades differs from the exterior climate and can be modelled using ray tracing to account for micro scale variations of the solar irradiance on the wall, as presented in [3]. In order to foresee the degradation on the different parts of a wall, accurate prediction models for wood degradation as a function of the main climatic factors are crucial.

Near infrared spectroscopy and hyperspectral imaging are well suited scientific tools for rapid and non-destructive characterization of wood surfaces [2]. The goal of this work was to assess the degradation of wood due to weathering, and to model its kinetics on the base of thin wood samples exposed outdoors by means of NIR hyperspectral imaging. The work has been focused on the influence of UV radiation on the weathering of wood, and its perceptibility in NIR-spectra images of the exposed wood samples.



Figure 1. Left: Samples exposed outdoor facing South. Right: image of three samples collected in this experiment after exposure for 1, 10 and 20 days

Materials and methods

A set of experimental samples were prepared from a single board of Norway spruce (*Picea abies*) wood by slicing it on the planner (Marunaka) to a thickness of $\sim 100\mu\text{m}$. The surface area exposed to weathering was of $30 \times 35 \text{ mm}^2$. Five sets of 21 samples each were exposed outdoors facing south for varying durations. A single set of 21 samples was firstly exposed, then after seven days a new set of 21 samples were exposed. The procedure was repeated for the following sets. One sample was collected each day from each set. Additional samples were exposed to artificial weathering in UVTest (Atlas) machine following 1 to 10 cycles of 2.5h UV radiation followed by 30 min water spray.

Thermogravimetric analysis was performed on selected samples using a Simultaneous Thermal Analyzer coupled with a Fourier Transform InfraRed spectrometer (STA FF9 F1 Jupiter, NETSCH, Germany) to estimate the lignin and holocellulose (cellulose + hemicellulose) contents.

Hyperspectral images in NIR range from 1000 to 2500 nm were obtained from the samples with a push-broom hyperspectral camera SWIR (Specim) in transmission mode. A segmentation of early- and late- wood was performed by factorizing the hyperspectral images with Principal Component Analysis (PCA). The changes in the spectra due to weathering were modelled by means of diverse regression techniques.

Results and discussion

Spectra corresponding to the early and latewood zones at varying degradation stages were successfully extracted from the hyperspectral images. Changes in the spectra were assessed separately (for early- and latewood) due to significant differences to the morphological, chemical and physical structures of these woody components.

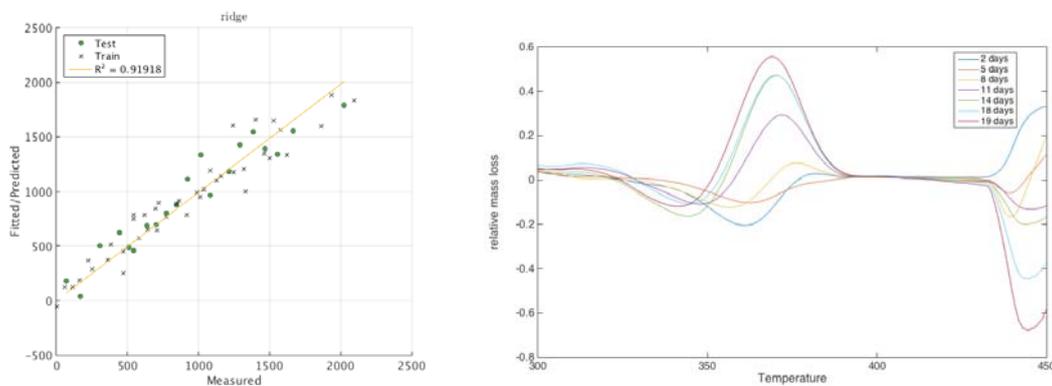


Figure 2: Left: measured and predicted UV radiation on wood samples (earlywood) exposed outdoors. Right: 1st derivatives of the thermogravimetric curves highlighting differences in kinetics of earlywood mass loss as related to reference sample

The deterioration kinetics due to weathering and the corresponding degradation of lignin was modelled by means of diverse regression techniques. It was found that the Ridge regression algorithm was relatively faster and more efficient than Partial Least Square (PLS) algorithm.

The best regression fits ($r^2 = 0.92$ for earlywood and $r^2 = 0.87$ for latewood) were obtained when modelling the spectra with the amount of UV-radiation as a response variable (Figure 2). It indicates that the amount of UV radiation is a prominent factor for wood degradation. The mass loss curves (first derivative) from the thermogravimetric analysis, as shown for some of the earlywood samples in Figure 2, indicate that the mass loss at different temperatures varies with the amount of weathering of the samples. Further analysis of the weather data along with improvement of the degradation assessment will be carried out in order to link the degradation progress with the defined weather doses.

Acknowledgement

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The effectiveness of FT-NIR spectral analysis for identifying bio-based finishes

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Wood durability is depending on wood species and specific environmental conditions related to the place where the wood is applied. The effectiveness of finishes is related to surface resistance parameters. However in many cases surface finish is affecting also wood appearance and its aesthetic properties.

In case of decorative antique parquets made of various, often very effective and expensive wood species, the choice of surface finish is crucial for parquet durability and its visual characteristics. The finishes used on floors had to create a hydrophobic layer on the surface, protect it against dirt, stains and discolorations, reduce its wear rate and be resistant to removal or scratches. They also needed to provide favourable roughness and gloss, with a minimum change in colour. Such change was especially undesirable in case of intarsia parquets made of many different wood species with different appearance.

Traditional, bio-based finishes included: wax and varnish made on the basis of linseed oil. Those substances leave the appearance effect of natural wood structure, with a beautifully highlighted yearly ring patterns. However they have different properties and performance, therefore precise identification is necessary for future conservation and restoration works. In case of antique buildings being still in use, the major requirement for the optimal method for identification of finishes is the possibility to be used in situ and assuring lack of negative effects for the antique material due to measurement. Therefore, utilization of non-destructive and portable instruments is favourable.

The purpose of this research consisted in assessing the effectiveness of FT-NIR spectral analysis for identifying bio-based finishes. Preliminary measurements were realized on well-characterized model samples of oak (*Quercus* sp), elm (*Ulmus* sp), ash (*Fraxinus excelsior* L) and pine (*Pinus sylvestris* L.), in order to calibrate the system and to develop identification algorithm. Procedures for finishing classification were subsequently validated with antique decorative wooden parquet samples collected from two manor houses in South-Eastern Poland.

FT-NIR spectroscopy highlighted differences between various natural surface finishing methods. Even if algorithm work well for classification of contemporary wood, evaluation of antique floor was more problematic, due to samples aging, natural weathering and necessary preparation of historic samples that might lead to partial or complete removal of the investigated finishing substances.

Characterization of wood surface degradation using FTIR, microtensile testing and colour measurements

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Numerous studies of wood weathering phenomena led to good general knowledge regarding principle breakdown processes during exposures. However, results obtained using different methods are incoherent and dependent on the particularities of the testing method applied. In order to better understand the processes which happen on and under the wood surface during weathering as well as to identify the most influential wavebands of light causing photodegradation of wood, several weathering tests (artificial and natural) were performed. Batches of thin strips of fir wood, assembled in packs of matching three strips (thus forming a 240 μm thick surface layer), were exposed to irradiation behind a set of cut-on glass filters that transmitted selected regions of the UV and visible spectrum in the range of wavelengths from 295 up to 515 nm. Weathering regimes included different light sources and various levels of climatic conditions (temperature and relative humidity of air).

Selected indicators of weathering - colour, microtensile strength at zero and finite initial span, as well as chemical changes developed with different intensities in various stages of exposure.

Typical FTIR spectra of wood surface reveal the two simultaneous processes which occur during weathering – decrease of peak around 1508 cm^{-1} , attributed to changes of aromatic skeletal (C=C) of lignin, and an increase of peak around 1736 cm^{-1} , attributed to carbonyl groups of hemicellulose. The third peak, around 1374 cm^{-1} , is assigned to carbohydrates in cellulose and hemicellulose, and was chosen to be a reference peak. Relative intensities of two longer peaks (around 1508 cm^{-1} and 1736 cm^{-1}) to the peak at 1374 cm^{-1} enable the semi-quantification of chemical changes and their correlation with colour and micromechanical changes. The above described processes were found to be orderly profiled in the surface, with proportionally lower intensities in deeper layers of wood surface.

Microtensile changes at finite (10 mm) span were more pronounced than at zero span. This testing setup should be more sensitive to delignification of the compound middle lamella, with reduced ability to transfer and distribute stresses. In addition to that, second and third strips (the layer down to 240 μm) exhibited significant strength loss measured over finite span. Prolongation of weathering resulted in inclusion of ever longer wavelengths into the active spectrum of light causing damage to the surface. The mechanical disintegration demonstrated by a loss in tensile properties follows the same pattern as the changes in colour, with development in depth of the surface and more active role of visible light in time. This process is not so pronounced as colour changes at the early stages of weathering, but it develops with some delay. However, the strength changes are more gradual in spatial and chronological aspects than the changes in colour.

The changes in yellowness (+ Δb^*) predominantly controlled the overall (ΔE^*) changes. Generally, after short term the changes are most pronounced in the top strips, much less in the second strips (80-160 μm) and are very small in the third strips (down to 240 μm).

Initial colour changes reflect the chemical alteration of wood more subtly than the micromechanical properties. As natural exposure develops in time, the effect of visible light becomes proportionally more expressed, since the ΔE^* curves shift in the visible range.

Discoloration developed disproportionately faster in early phases of exposure; finite span microtensile strength reached saturation in the middle of the exposure period, whereas zero span strength and chemical changes reached their maxima at the end of exposures. This practically means that, different methods revealed different nature and extent of "damage" in specific phases of exposure and only the combination of results can give a complete image of the process.

BIO4ever - Bio-materials for building envelope - expected performance, life cycle costing & controlled degradation

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The Bio4ever project is a multi-disciplinary research dedicated to fulfilling gaps of lacking knowledge on some fundamental properties of novel bio-based building materials. The overall goal is to assure sustainable development of the wood-related construction industry, taking into consideration environmental, energy, socio-economic and cultural issues. This can be achieved by developing original, trustworthy tools demonstrating advantages of using bio-based materials when compare to other building resources. A comprehensive understanding of the physical/chemical properties and their connection with the material's structure will be obtained as a result of a combination of analytical/experimental methods and numerical modelling.

The experimental samples include around 100 various types of bio-materials that might be used for building facades. Spectroscopy will be directly implemented for in-situ measurements of investigated materials with portable MicroNIR 1700 (VIAVI, Santa Monica, USA). The series of samples will be also measured in laboratory conditions with Vector 22N (Bruker GmbH, Ettlingen, Germany). Detailed characterization before and during degradation by biotic and a-biotic agents is foreseen in order to produce experimental data used for development of the numerical models simulating the material deterioration in a function of time and exposition.



Figure 1. Appearance changes to wooden facades due to weathering

Acknowledgment

BIO4ever is ongoing project funded within a call SIR by MIUR (RBSI14Y7Y4).

Keynote 5: NIR & Chemometrics: how to handle NIR signals, recover information and build models

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Near infrared spectroscopy (NIRS) coupled to chemometrics is a story of long success. In fact, thanks to chemometrics it has been possible to use NIRS as a quantitative methodology. In general, we may say that typical NIRS drawbacks, such as strong band overlap, low absorption coefficients, strong variability due to sample/environment conditions, very limited linearity of the response, could only be handled by multivariate data analysis.

Nowadays, NIRS is becoming a standard for properties calibration in routine analysis, in many different fields such as food control, chemical laboratory, chemical and pharmaceutical industries, also the use of NIR sensors at the production plant is constantly increasing. However, most of the applications deal with the development of multivariate calibrations as it is usually provided with the instrumentation software.

Notwithstanding, the synergy between chemometrics and NIR may offer a much wider perspective.

The objective of this presentation will be to give an overview of chemometrics tools applicable to all stages of analysis from experimental planning to knowledge gain (Figure 1).

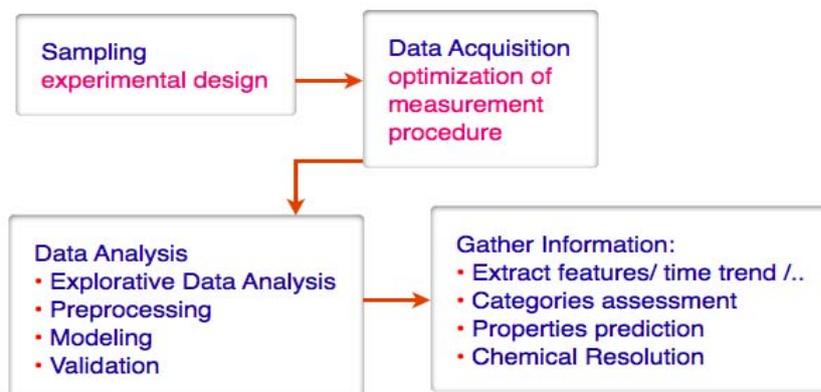


Figure 1

In particular, taking into account the peculiarities of NIR signals and the main applicative contexts, focus will be on signal pre-processing, process monitoring (at/on/in-line), highlighting time trends, assessment of raw material variability, evaluation of treatment effects in designed data, development of multivariate control charts for quality control.

Both bilinear and multilinear chemometrics methods will be presented discussing the most relevant issue for an effective data analysis strategy.

NIR hyperspectral imaging of Fire-Retardant-Treated Wood

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Fire-retardant-treated wood is wood which has been impregnated with fire-retardant chemicals in solution under high pressure. The treatment reduces surface burning characteristics, such as flame spread, rate of fuel contribution and smoke contribution. A challenge is often to evaluate whether a wood sample has been successfully treated and if the treatment has reached through the sample.

NIR hyperspectral imaging has been carried out to see if this technique can be used to detect the fire retardant treatment on wood samples. Various methods have been explored here to estimate the amount of fire retardant chemical present through the thickness of the samples.

Near-Infrared (NIR) hyperspectral imaging at high resolution and the difficulty to calibrate for the three main wood components

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Abstract

Structural and chemical mapping can contribute to selection and breeding support. Infrared scanning can be used to map density, tension wood and chemistry without the need for more complex, expensive and/or time consuming methods, yet with less accuracy and lower resolution, thus being applicable as a single tool for breeding selection. Based on poplar disks for early selection, data were collected in the NIR in a high-throughput mode.

Results of NIR-based hyperspectral mapping illustrated that tension wood zones were denser and contained more alpha-cellulose and less lignin which corresponds with the existence of the G-layer as described in literature. Unfortunately it remains difficult to calibrate for absolute figures on the three main wood components (hemicellulose, cellulose and lignin).

This paper would like to open discussion on the importance exact data for mapping wood chemistry.

Near-infrared analysis is another technique also widely used in many research fields, mostly to derive chemical information from the object under study. The traditional method for chemical analysis of wood is wet chemistry. Numerous wet chemical analytical methods are based on the fractionation of biomass samples with further isolation of purified fractions that can be quantified using conventional analytical instruments. These methods are however time consuming, labour intensive, expensive and destructive, and therefore not widely implementable. Moreover these analyses need a certain amount of material which hinders mapping of the chemical properties at higher resolution.

Artificial weathering (QUV) of wood plastic composites made with thermally modified wood residues

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Thermal modification is one of the most commercially developed wood modification method, and it is widely popular in Europe. The process involves wood controlled degradation in oxygen absent atmosphere, where the furnace temperatures (160-220°C) are higher than the conventional drying temperature of wood. In the year 2014 by using *ThermoWood* process 145 000 m³ of thermally modified wood was produced. The amount each year is increasing. In thermal modification process like in almost every process there are residue products. These residues are sawdust, damaged boards, shavings and others. Because of the chemical changes of wood (more hydrophobic nature) these residues could be a great filler in thermoplastic polymer matrix. Wood plastic composites as well as thermally modified wood, are well known materials.

It is known that WPC are not durable to UV light, because of the polymer matrix and wood sensitivity of the radiation. So UV stabilizers or UV absorbers should be used to protect the material from UV degradation. In our research we did not use any additional additives, so we could determine: what is the situation for WPC with thermally modified wood. WPC consisted of 50 wt% polypropylene and 50 wt% wood fibers (unmodified or thermally modified). Samples for artificial weathering were 1.5 mm thick sheets made by compression moulding. The colour changes ΔE_{ab} (CIE Lab) and surface properties were determined after artificial UV weathering (no water cycle).

Colour changes in both cases were significant after 280 h of artificial UV weathering (Figure 1), where ΔE_{ab} value for WPC with unmodified wood fibers was 12.9 ± 2.1 units, but for WPC with thermally modified wood fibers it was 31.2 ± 1.7 units. These significant colour changes are mostly, due to degradation of polypropylene matrix, where in the result white, crystalline degradation products are formed on the material surface.

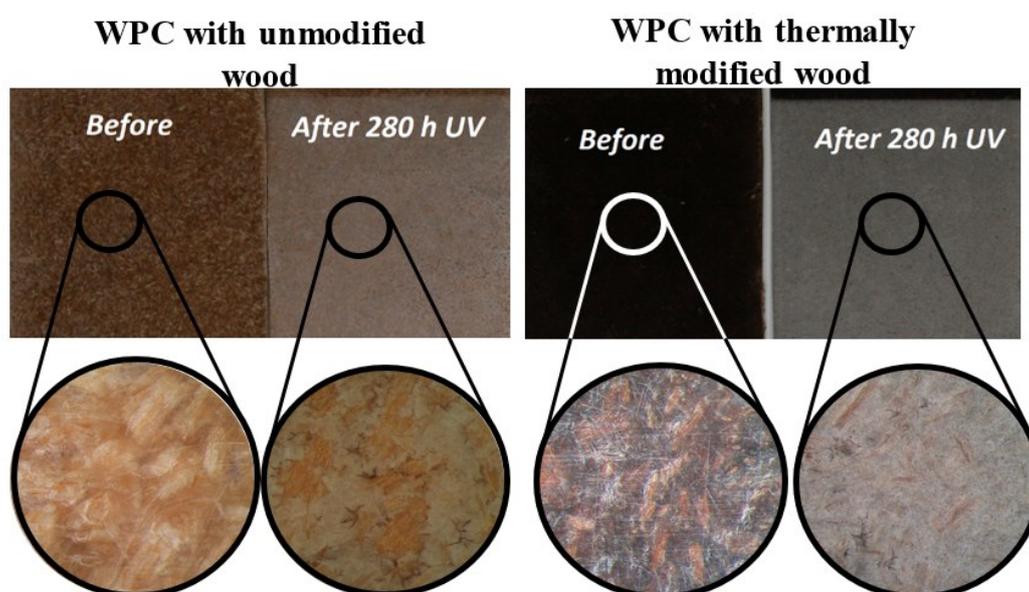


Figure 1. Optical microscope pictures of WPC before and after artificial UV weathering

Optical microscope pictures with 50x magnification (Figure 1) shows that in both WPC cases after 280 h of artificial weathering, the material surface is shattered in small pieces. However, there is an important benefit for WPC with thermally modified wood fibers: the erosion effect is less noticeable. For WPC with unmodified wood fibers after 280 h UV we can see that most of the fibers that were

near the surface are exposed due to surface erosion, but in case of thermally modified wood fibers there are no such things noticeable.

The research results show that UV absorbers or UV stabilizers are necessary also for WPC with thermally modified wood fibers. However, there is a benefit comparing to WPC with unmodified wood fibers, which is that surface is less subjected to erosion. That means that WPC with thermally modified wood fibers are more durable against UV radiation despite the significantly larger colour change.

Acknowledgements

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Assessment of poplar veneers for plywood manufacturing after up-grading phase by vacuum thermal treatment

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Poplar is a fast growing species, cultivated in plantations with short rotation period, important for several industrial and ecological purposes. One of the main problems noticed during industrial manufacturing and further use of the derived products is the high hygroscopicity of the wood material.

Vacuum thermal treatment is proposed here for veneers up-grading that might be used for manufacturing of multi layer parquets and panels with reduced hygroscopicity, improved durability and dimensional stability. The goal of this research was to evaluate the influence of temperatures, time and pressure on principal indicators of thermal modification process such as mass loss (ML) and equilibrium moisture content (EMC) of investigated veneers.

Totally 38 batch processes with various treatment conditions (temperature T ranging from 150°C to 240°C, pressure p : 100, 250 or 1000 mbar and treatment time t from 0.5 to 22.5 hours) were performed. Selected chemometric techniques: Principal Component Analysis (PCA), Identity Test (IT) and Partial Least Squares (PLS) were used for spectral data evaluation. PCA allowed direct comparison of the effect of process conditions on chemical composition of poplar veneers (Figure1). Identity Test was used here for classification of the veneers according to treatment intensity. Partial Least Square (PLS) models were developed for prediction of ML and EMC. Preliminary results support the assumption that PLS model of NIR spectra are suitable for quality control of vacuum thermally treated veneers and might be used at industrial scale.

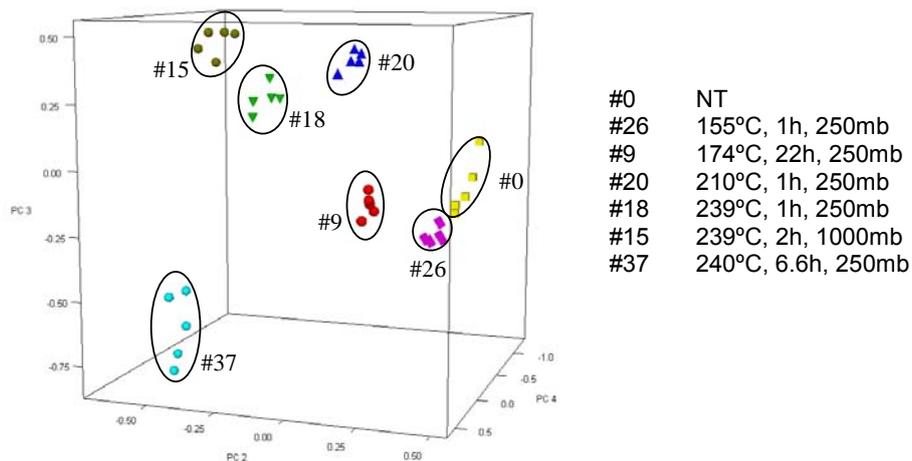


Figure 1. PCA used for direct comparison of the effect of process parameters on the chemical composition of thermally modified veneers (TMV)

Acknowledgment

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Some colour characteristics of thermally modified wood

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Thermal modification of wood has gained not great but stable niche in the wood-processing sector. Initially the main focus of thermally modified wood promotion was put on its improved biodurability and enhanced dimensional stability without use of harmful chemicals. However, as the thermal treatment commonly results in substantial loss of wood strength, thermally modified wood is mostly recommended for non-structural usage. Its typical applications are garden furniture, flooring, cladding, decking and similar. In all these applications wood serves as a functional as well as a decorative material and its altered colour is very important feature for attracting the attention of consumers. However, another substantial aspect of a material choice is its potential working life which is largely determined by the ability of the material to maintain aesthetic features during its end-use.

Better understanding of how the characteristic colour of wood is formed during the thermal treatment as well as its discolouration processes caused by weathering are important aspects for maintenance of wood decorative properties during its application.

A series of experiments are performed covering different aspects related to thermally modified wood colour formation and changes due to weathering under different conditions. Experimental specimens were prepared from four hardwood species (*Betula pendula*, *Populus tremula*, *Alnus incana*, *Fraxinus excelsior*) and modified at three temperatures (140°C, 160°C, 170°C) in a water vapour medium under elevated pressure. Spectrofotometrical measurements of reflectance spectra and conversion them into colour parameters (CIE Lab colour model) are used for colour characterization and evaluation of the changes. To assess the colour stability of modified wood, discolouration of specimens exposed to different environments (dark, indoors, outdoors, under filters transmitting definite wavelength ranges, in the weathering chamber) are monitored.

The results show that all studied woods acquire quite similar colour with almost identical reflectance spectra during thermal treatment. Analyses of reflectance changes show that rise of treatment temperature results predominantly in formation of chromophores with characteristic absorption in the longer wavelength range. Moreover, the chromophores responsible for the typical colour of thermally modified wood are not parts of low molecular, extractable substances, as wood extraction causes hardly any changes in its colour. However, the surfaces were found to be remarkably darker than the cores for all tested specimens.

Although prolonged outdoor exposure results in grey surfaces for both wood types, there is substantial differences between modified and unmodified wood regarding colour stability during exposure to solar irradiation. The very first indicator of differences in weathering processes is fading of thermally treated wood contrary to darkening of unmodified wood. Moreover, our results show that visible light causes essential discolouration in modified wood while UV radiation is the dominant factor provoking changes in unmodified wood colour (Figure 1).

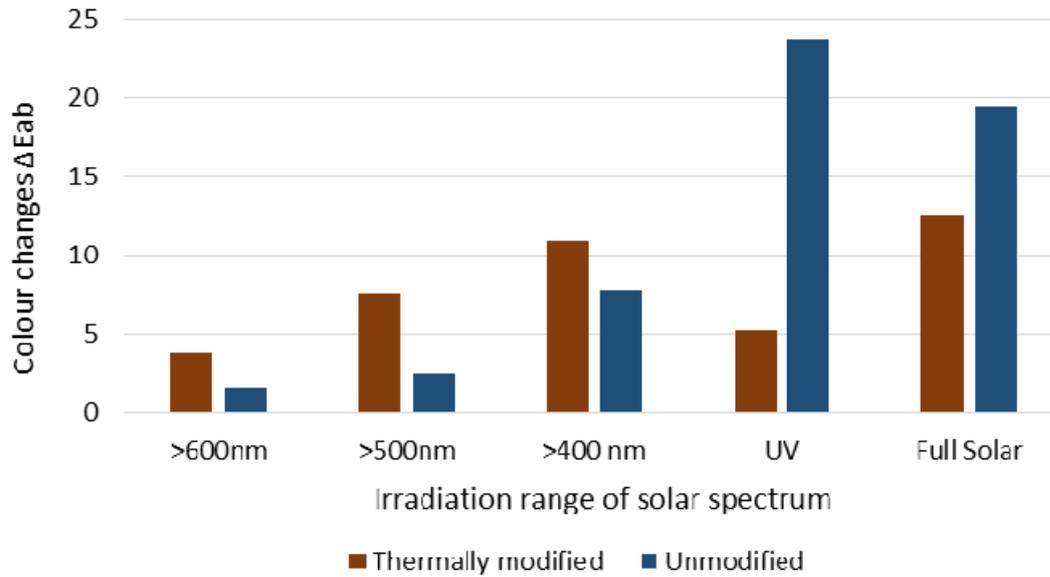


Figure 1. Colour changes of thermally modified and unmodified wood shielded by filters with different transmittance wavebands

However, monitoring of colour or reflectance changes do not provide information about chemical processes related with these changes. Moreover, not all wood transformation and degradation processes manifest in discolouration. Therefore testing of specimens by tools of spectroscopy (FTIR-ATR, FT-NIR) is now in progress.

Acknowledgments

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Evaluation of heat treated black alder wood by FT-NIR

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Heat treatment is the process in which wood is subjected to high temperatures from 120°C up to 250°C for several hours. Under these conditions some chemical changes of wood main chemical components occurs with further influences on its properties, such as: hygroscopicity, dimensional stability and resistance to biodegradation. This way without any additional chemical substance and under high temperature exposure, wood becomes a more adequate material for outdoor uses (claddings, facades, landscaping and park structures, pedestrian bridges), but also for indoor applications as well (panelling, furniture, flooring, stairs).

According to data in specialty literature, the decomposition of wood main chemical components takes place at temperatures of 180°C for hemicelluloses, 270°C for lignin and 340°C for cellulose [1]. NIR is a non-destructive and useful method used to determine chemical composition, to detect and predict physical and mechanical properties of wood material [2, 3].

Most of the research work and studies have been performed during last decade on resinous species which are less sensible to temperature and may be easily treated. But also hardwoods were modified by heat treatment, such as aspen, ash, birch, oak and beech. The potential of NIR analysis was widely exploited to give essential chemical information on such modified wood species [4, 5].

The demand for valuable wood species has increased over their cycle of regeneration and the phenomenon became a difficulty for the forestry sector. Such a situation imposes a new direction to the research trend to find new ways for the capitalization of wood species which are considered less valuable but fast growing ones. And black alder (*Alnus glutinosa* L. Gaertn.) is one of them. In Romania quite less interest is shown to black alder wood, in spite of its good potential for furniture industry especially. Apart of its nice appearance as natural wood, alder may get a more pleasant dark colour when heat treated. Valuable wood species may be successfully replaced by alder in various restoration works. Chemical changes of wood material need to be evaluated in such applications.

Therefore the purpose of the present work was to evaluate the chemical changes caused by heat treatment when applied to black alder wood. Samples were subjected to heat treatment in a regular oven at two temperature levels (120°C and 190°C) and two time spans (3h and 6h). NIR method was used to evaluate the chemical changes caused by heat treatment.

It was found that heat treatment affected mostly the hemicelluloses as the exposure temperature and duration increased. Crystalline and semi-crystalline cellulose were less influenced by heat treatment than amorphous regions of cellulose. A higher content of lignin in treated wood was noticed. Large spectral variation at absorptions due to water and hemicellulose were noticed.

Figure 1 presents the differences in spectra based on second derivative related to main chemical components of black alder wood as function of heat treatment.

Results of this work might be useful for work developed by restorators of old art furniture. Moreover, such data may be used for further optimization of heat treatment procedures in wood industry sector.

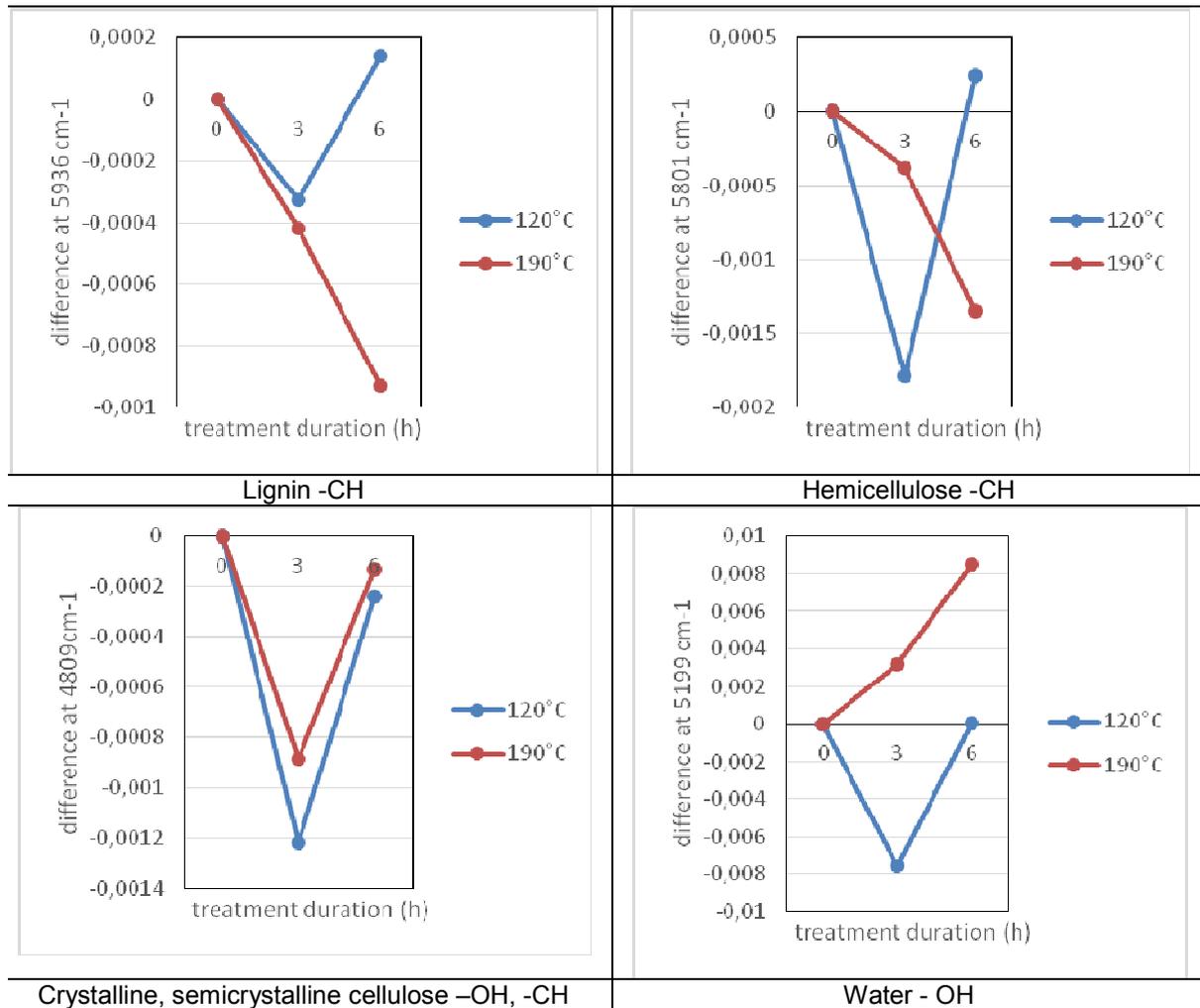


Figure 1. Changes of main chemical components of black alder as function of heat treatment

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Pollutants detection on wood waste using FT-IR-ATR

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Abstract

Nowadays in Italy the wood residues from building demolition and deconstruction process, from timber treated processes, from wood-based panel industry and finally from waste management platforms are mainly selected, processed and delivered to particleboards enterprises; the residues rejected by the selection processes are either combusted in specialized plants either land-filled. These residues are constituted by widely heterogeneous materials including treated/non treated wood, boards and other wood-based and non wood-based materials (such as plastics, plastified paper, paper, melamine, urea, formaldehyde, etc.).

Monitoring the occurrence and the concentration of pollutants (coatings, preservatives, glues, hydrocarbons) on wood residues is fundamental for an accurate characterization and selection of the best management for each material.

Goals

The primary goal of this research is the control of pollutants in wood residues from industrial processes. The main benefits are:

- sorting the polluted from non-polluted wood;
- sub-selection of polluted residuals in order to sort each category in an appropriate way;
- reduction of recycling of dangerous pollutants;
- profitable use of non-contaminated wood.

This goal is reached by using FT-IR-ATR technology by creating a spectra database of pollutants and by monitoring the presence, typology and concentration of unknown polluted wood residues. The industrial pollutants database is constituted by known coatings, glues, and preservatives which have been analyzed wet and once dried.

Preparation of sample

Wood residues with unknown contamination were sampled from different categories of wood processing enterprises and recycling platforms; the single specimens were not prepared except than lightly brushing the surface from dust and removing the coarse incrustations. The most planar surface was then pushed onto the crystal. Due to the surface resistance of diamond, the original window of zinc selenide has been removed and the diamond window has been used for such kind of application despite the lower optical performances.

The specimens were then measured with a scanning sessions of 60 seconds, three time repeated. After each analysis, the MIR spectra were compared to the ones in the commercial pollutants database purchased by S.T.Japan Spectra DataBase.

Data and spectra

The commercial databases provide a list of compounds scored according a hit quality list. The results can be very variable. In the two following images are shown opposite results. In Figure 1, the inner layer of a plywood panel has been classified as glue, thanks to the automatic comparison made by the software between the spectrum of the plywood panel (in red) and the reference spectrum - chosen within the library (in blue), providing the hit quality list with a quite high score. In Figure 2, a particleboard has been erroneously identified, being at the first place the compound "codeine"; in this case it should be noted that the hit score was quite low.

Conclusions

The occurrence and qualification of pollutants were frequently successfully traced with FT-IR-ATR, but the occasional occurrence of false negative and false positive affected the overall reliability of the method. Nevertheless the technique has been found effective for classification of wood-based polluted materials, provided that cross checking based on the knowledge of both waste materials and of wood based products is deeply managed by the researcher.

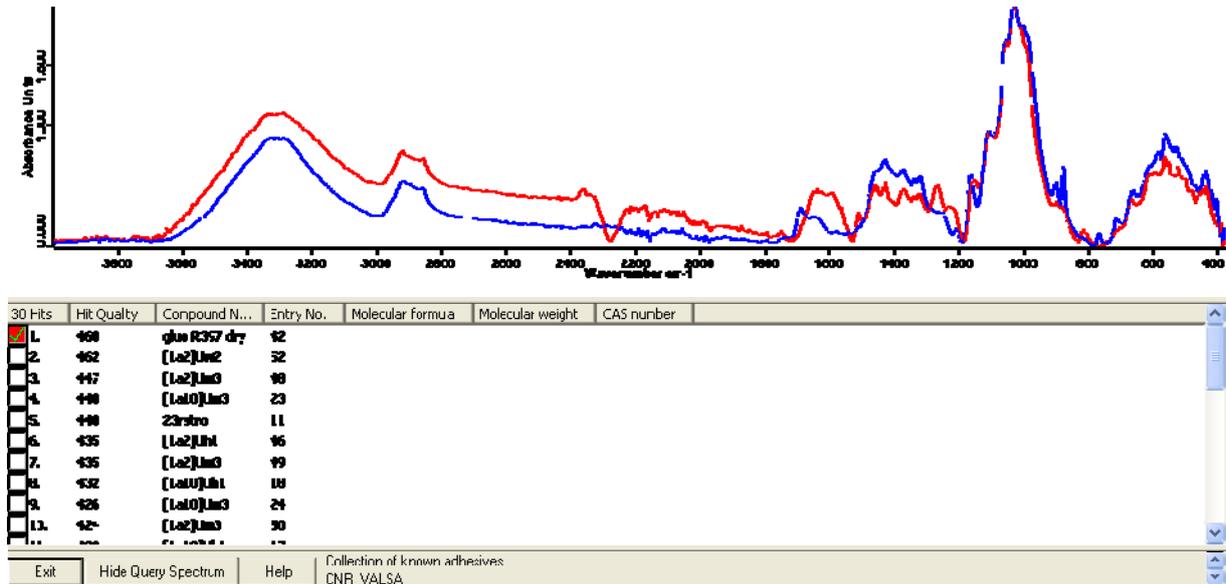


Figure 1. The spectrum of the plywood panel (in red) compared to the reference spectrum chosen within the library (in blue)

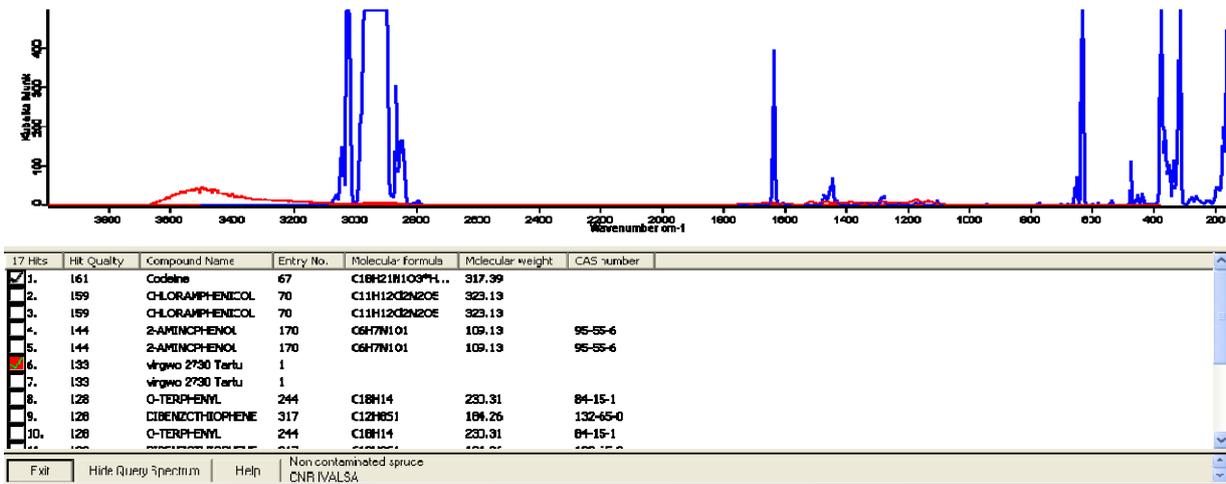


Figure 2. The particleboard is not in the first place of hit quality list proposed by using the available database; moreover the two spectra does not match at all.

Monitoring thermally modified wood performance by NIR. Case of study: surface treatment

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Abstract

To preserve surface features of thermally modified products during outdoor exposure it is necessary to protect it against natural weathering factors and thus to avoid surface degradation and changing tones. Thermo-Ash (*Fraxinus excelsior*) samples treated at 192, 202 and 212 °C were finished with water-borne and UV-Radcure coatings to preserve the surface and extend aesthetic features. Afterwards, coated samples were subjected to accelerated weathering in a SOLARBOX chamber (M/S Erichsen, model 522) with changing cycles of UV radiation, condensation and water spray to simulate conditions of temperature, direct solar radiation and precipitation. Finishing and monitoring methods are summarized in Table 1. The samples were examined after 1000 and 2000 hours of aging cycles and colour, gloss, contact angle and chemical changes by NIR were measured.

Table 1. Coating parameters and evaluation methods

Sample	Finishing procedure				Monitoring Surface changes			
	Coating product	Finishing method	Drying	Curin g	Color	Gloss	Wettability	Perfor mance
Ash	Water-borne → Brush → YES							
Ash 192°C					CIELab ¹	GM ²	CA ³	NIR ⁴
Ash 202°C	UV-Radcure → Industrial roller → YES							
Ash 212°C								

¹ CIE method L*a*b*;² Gloss-meter measurement;³ Contact Angle; ⁴ Near-Infrared Spectroscopy

The preliminary results indicated that degradation in all samples was higher in surfaces finished with UV-Radcure compared with water-borne finishes. UV-Radcure samples showed greater colour shade variations and gloss reductions, though all samples maintained acceptable protection values. On the other hand, surfaces finished with Water-borne after accelerated weathering maintained the photostability with steady colour and gloss values after several cycles. Regarding surface hydrophobicity, both coating products maintained high values of contact angle in concordance with hydrothermal wood features.

These raw results do not suggest a clear link between thermal treatments and the effect obtained neither have they directed to a predictive model over time. NIR spectroscopy was used firstly to track chemical differences between samples, and due to weathering test; and secondly as a tool to analyze the multivariate data set created and generate predictive models to classify the material according to the general trends applying the principal components analysis (PCA). The preliminary analysis of variables on NIR spectra integrates the data set to present its heterogeneity or homogeneity using algorithms for clustering data (Figure 1). This step is followed by PCA to reduce the dimension of the data set. Moreover, the identity test distinguishes samples into defined groups from NIR data analysis (Figure 2). Finally, Partial least Squares (PLS) magnify PCA showing linear models of the input variables to obtain a cross- or test-set validation (Figure 3). These results are a simple visualization but it is the basis of a predictive analysis, and it is necessary to add more input data and to validate every statistical step.

Table 2. Data set of chemical and physical properties

Analysis	Lignin	α -Cellulose	Hemicellulose	Extracts	Density	Acidity	pH	WWA	Contact angle
Ash	28.88	45.38	28.88	4.69	0.69	0.0674	4.55	54.65	61.31
Ash192	29.13	43.81	29.13	6.11	0.68	0.0435	4.55	34.59	99.96
Ash202	35.12	43.93	35.12	8.06	0.65	0.0279	4.75	33.13	99.19
Ash212	35.54	38.14	35.54	9.49	0.63	0.0159	5.32	33.03	97.22

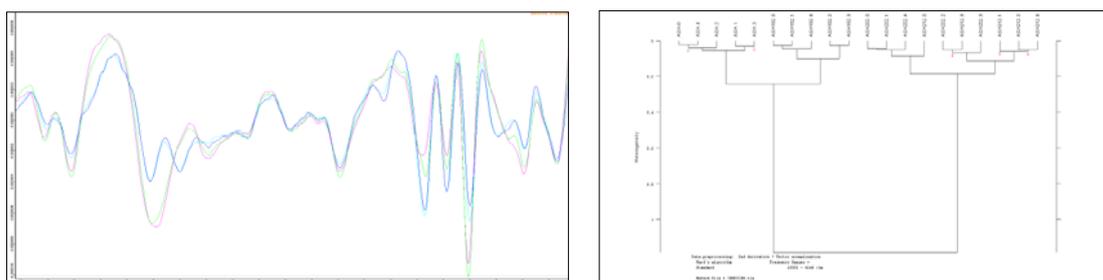


Figure 1. NIR spectra of samples and cluster analysis of similarity between treatments

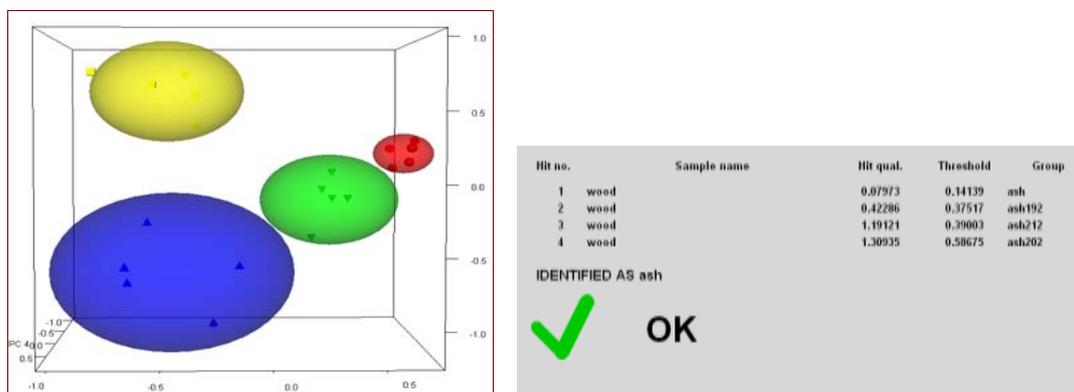


Figure 2. PCA and identity test of thermally modified samples

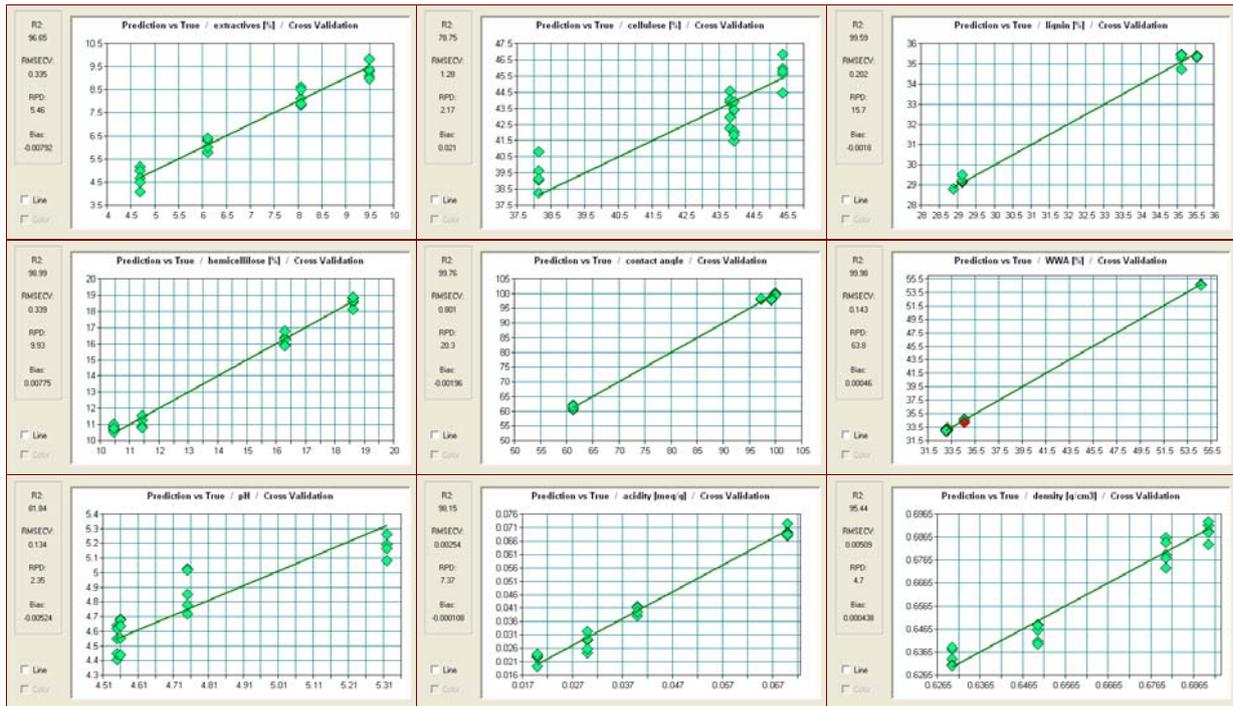


Figure 3. PLS of thermally modified samples data set

TV4NEWOOD – Thermovacuum: new process for generation of thermally modified wood

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Tv4newood project, financed by European Commission with Eco Innovation programme, has the aim to create and disseminate in European market a new process for generating thermally modified wood. The product timber, having special properties of strength and durability, is created without adding chemicals and with a considerable saving in energy. This is possible with a technology that operates "under vacuum".

The project investigates the innovative thermal treatment process of seven European wood species considered today as economically unattractive. The process improves durability and aesthetic characteristics of local resources in order to obtain resistance and appearance similar to tropical woods.

NIR spectroscopy and selected chemometric techniques provided new highlights for understanding the modification process kinetics. Principal Component Analysis (PCA) allowed direct comparison of the effect of process parameters on chemical composition of investigated species. Partial Least Squares (PLS) prove potential of near infrared spectroscopy for development of robust prediction models for mass loss and equilibrium moisture content, considered as reliable indicators of the wood modification advancement.

Experimental results prove that NIR spectroscopy have great potential for quality control of vacuum thermally treated wood and trials for further implementation toward on-line process control are under development.

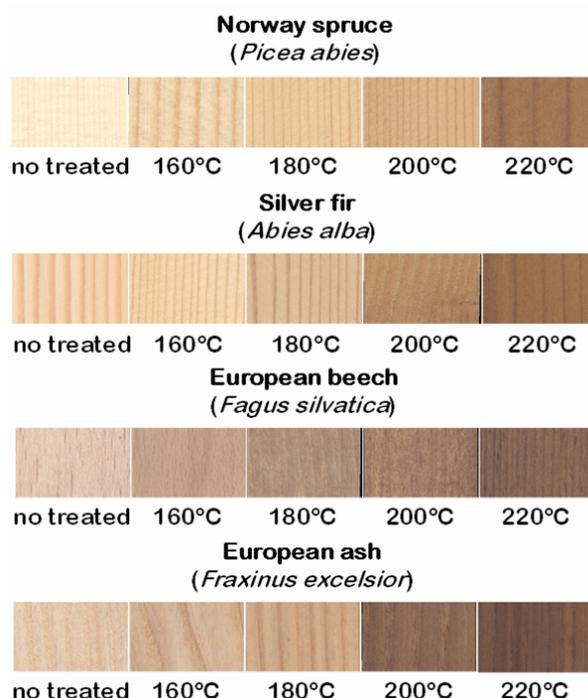


Figure 1. Appearance of wood after Thermovacuum process

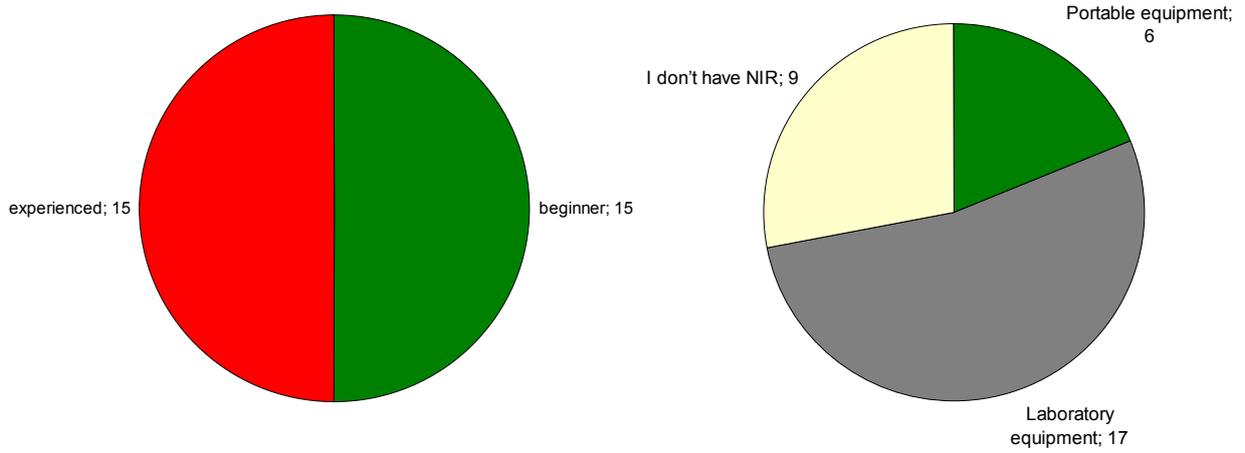
Acknowledgment

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List of participants

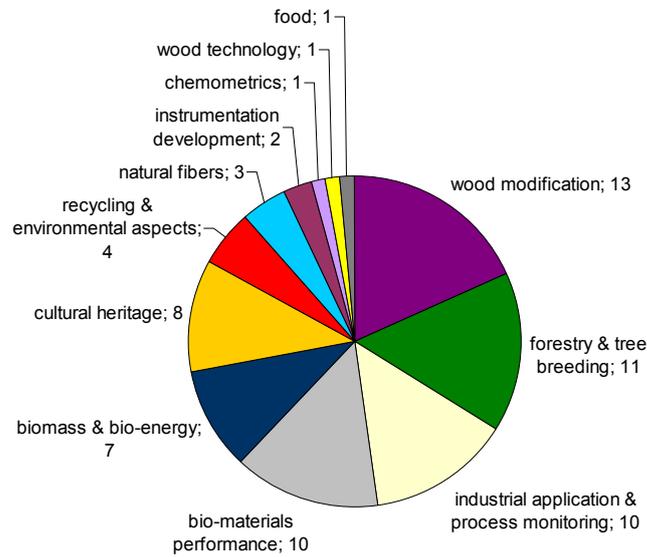
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Some facts about NIR&wood: sounds good #2 participants



Experience level in NIR

Used/possessed NIR equipment



Research interests

equipment \ research interests	wood modification	forestry & tree breeding	industrial application & process monitoring	bio-materials performance	biomass & bio-energy	cultural heritage	recycling & environmental aspects	instrumental development	others
portable equipment	● ●	● ● ● ●	● ● ● ●	● ●	● ●	● ● ●	● ●	●	● ●
laboratory equipment	● ● ● ● ● ●	● ● ● ● ● ●	● ● ● ● ● ●	● ● ● ● ● ●	● ● ● ● ● ● ● ●	● ● ● ● ● ●	● ● ● ● ● ●	● ● ● ●	● ● ● ● ● ● ● ●
no NIR instrument (yet)	● ● ● ● ● ● ● ●	● ●	● ● ● ● ● ●	● ● ● ● ● ● ● ●	● ●	● ● ● ● ● ●	● ●		

● - beginner ● - expert

