Scientific report
STSM COST FP1006

Host person: Marion Noel

Host Institution:Bern University of Applied Sciences – Architecture, Wood and Civil Engineering, Biel/Bienne, Switzerland

Period: from 11th of January to 7th of February 2015

By: Maria-Cristina Popescu, 28.02.2015

The aim of the above mentioned STSM was to establish scientific collaboration between “PetruPoni” Institute of Macromolecular Chemistry of Romanian Academy (Dr. Maria-Cristina Popescu) and BFH (Dr. Marion Noel). As a result of the discussions, we established themes for possible joint EU projects.

The scientific work has been focused on NIR spectroscopy, Scanning Electron Microscopy (SEM), IR Imaging and ASE tests on different impregnated wood samples.

Description of the work carried out during the visit

- ✓ materials: birch and pine wood impregnated with CNC based polymer
- ✓ preparation of the wood sample for ASE tests
- ✓ evaluation of weight and dimmensional stability of the impregnated wood sample during ASE tests
- ✓ training in operating the NIR, SEM and IR Imaging instruments
- ✓ recording of the NIR spectra, SEM images and IR imaging
- ✓ processing of the NIR spectra and of the IR imaging
- ✓ finalization of the data processing, interpretation and writing of a co-authored paper will be continued in Romania after the completion of the STSM

Description of the main results obtained

IR imaging

FTIR spectroscopy in combination with microscopy has the potential to resolve and quantify the distribution of organic components in different blends. Infrared radiation is absorbed by the molecular bonds in the sample, such as C–H, O–H, N–H, C=O, C–C, resulting in stretching, deformation or/and twisting of the bonds and leading to characteristic transmittance and reflectance patterns at specific wavenumbers. When infrared spectroscopy is combined with microscopy and imaging techniques, the spatial resolution of the chemical composition is achieved [Naumann, A., Navarro-González, M., Peddireddi, S.,Kües, U., & Polle, A. (2005). *Fungal Genetics and Biology,42,* 829-835.]. FTIR Microscopy was used e.g. to detect fungi [Naumann, A., Navarro-González, M., Peddireddi, S.,Kües, U., & Polle, A. (2005). *Fungal Genetics and Biology, 42,* 829-835]or for evaluation of differences in the chemical composition of transgenic aspen [Labbé, N., Rials, T. G., Kelley, S. S., Cheng, Z.-M., Kim, J.-Y., & Li, Y. (2005).*Wood Science and Technology, 39,* 61-77.].

FTIR imaging maps were recorded for simple polymers and impregnated wood using a AutoIMAGE FTIR Microscope (PerkinElmer) in the wavenumberrange from 4000-700 cm^{-1}. A spectral resolutionof 4 cm^{-1} and a number of 64 scans per measurementwere used. A number of 600 spectra are recorded for a sample area of 145 µm x 145 µm.

The experiments were done in a period of 7 days (see Gantt diagram).

A typical example of the obtained IR image is presented in Figure 1, where the distribution of the components in the blend can be evaluated. As it can be seen the different color from the IR image reflects the localisation and the amount of different compounds. Thus, the blue color can be assigned to CNC and the red color to acrilic polymer while the green colour reflects the blending between the components.

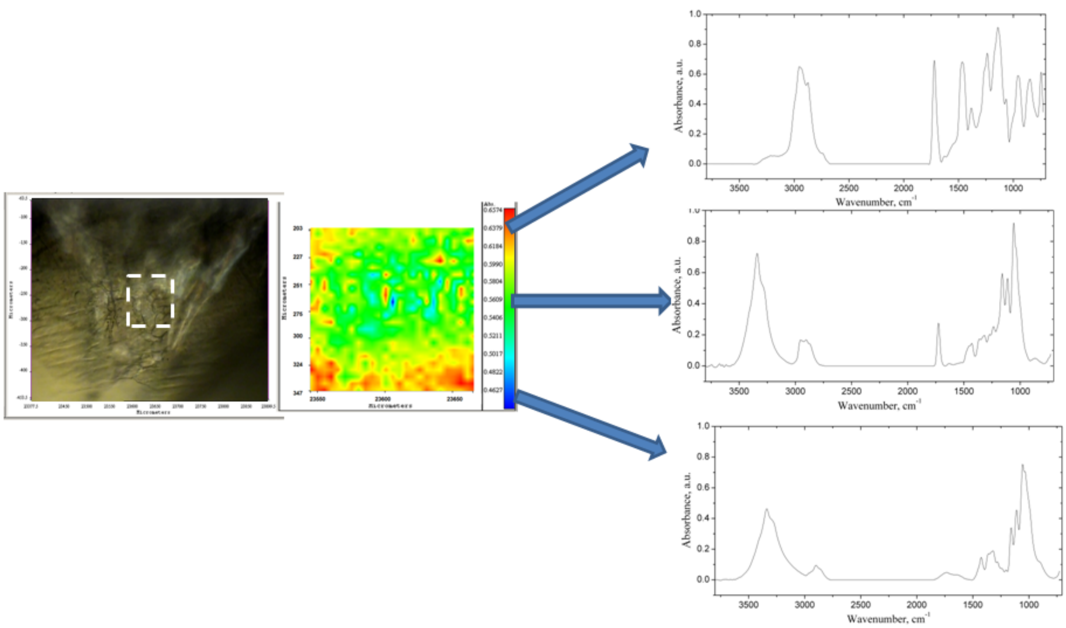

Figure1. The IR image of the indicated region and the spectra coresponding to each color from the IR image

NIR spectroscopy

Near infrared spectroscopy (NIRS) is a nondestructive evaluation technique for organic materials and has found wide spread use in the food, agriculture, pharmaceutical, petroleum, polymer, and pulp and paper industries.

It is an analytical technique that has been used for the prediction of product properties since the 1960's. However, the NIR region of the electromagnetic spectrum was rarely utilized and was considered that it contained no relevant structural information due to the absence of sharp bands, the abundance of overlapping and shoulder bands, a dramatic loss in sensitivity (2 to 3 orders of magnitude relative to the mid infrared) and the difficulty of making band assignments owing to the presence of numerous overtone and combinations bands.

At the beginning of 1990's the analytical applications of NIR spectroscopy were widespread with different domain like agriculture, food, paper, petro-chemical industry, polymer and textile industry, etc. The popularity of this type of spectroscopy can be attributed to the emergence of high precision spectroscopic instruments with very high signal to noise ratios to detect minute differences in the reflectance spectra and high-speed computers to carry out the complex calculations involved in multivariate analysis.

This method can be used for evaluation of materials that contain –CH, -OH, and –NH chemical functionalgroups, because the NIR region contains absorptionbands corresponding to overtones and combinations of vibrations of these chemical groups. Forthis reason, NIR techniques have shown promise as a nondestructive evaluator of the propertiesof wood based materials.

By this method birch and pine wood impregnated with CNC based polymers were analysed in order to evidence the interactions between the wood components and coating materials.

The experiments were performed in a period of 7 days (see Gantt diagram).

For each sample, a number of 10 spectra were recorded in a 800-2600 nm spectral range. The average spectra and their second derivatives of the studied samples are given in Figure 2.

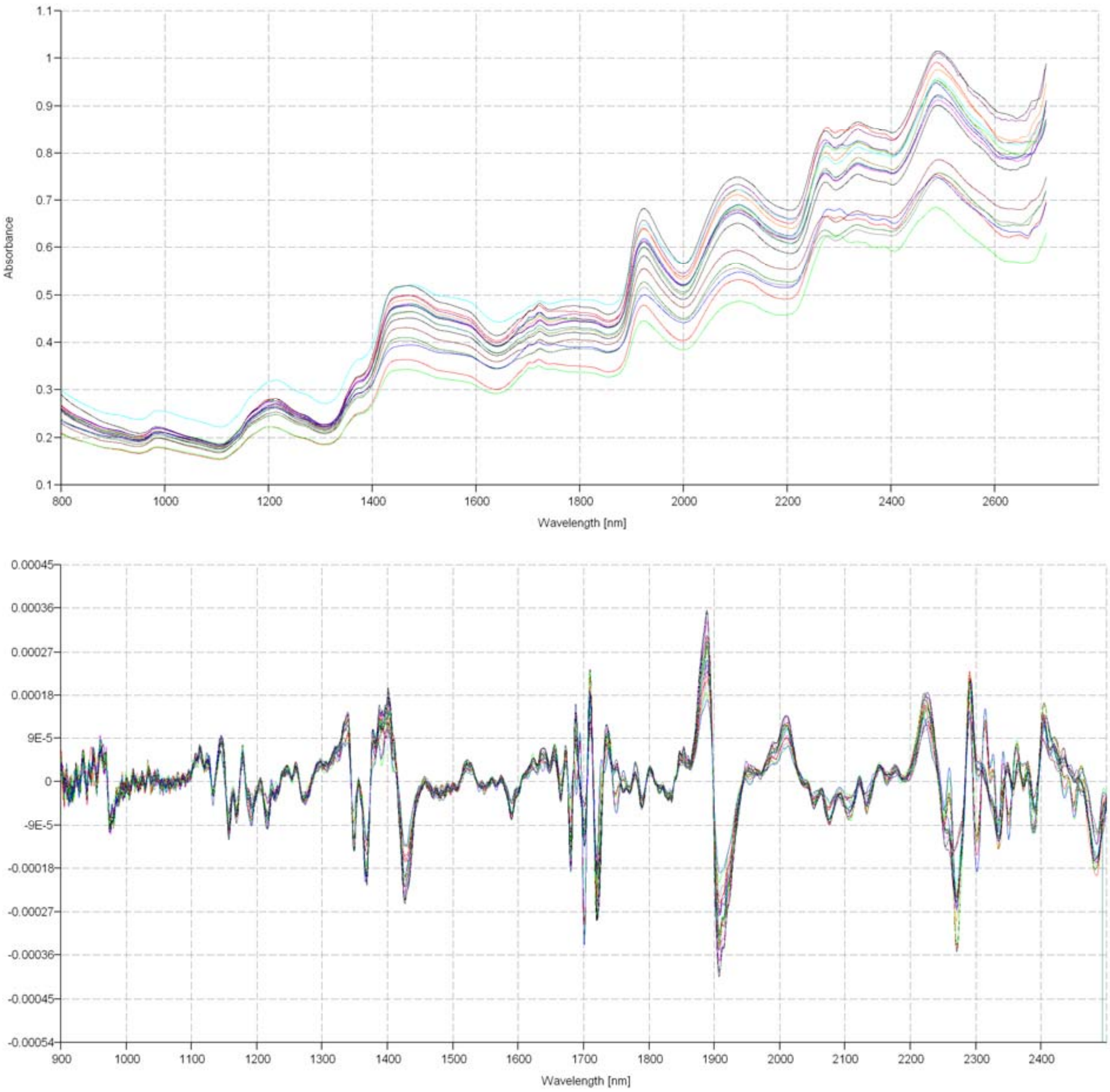

Figure 2. NIR spectra and their second derivatives of the birch wood treated samples

Significant differences can be observed in the 1600-1800 nm and 2000-2400 nm spectral regions. The 1100-1640 nm region correspond to first and second overtones of O-H, first overtones of C-H combination bands and second and third overtones of C-H, and Car-H stretching vibrations, 1640-1850 nm region correspond to the first overtone of C–H or CH_2 stretching vibrations from aromatic groups in lignin and semi-crystalline or crystalline regions in cellulose, whereas the bands in the 1850-2000 nm region are assigned to the combinations of O-H stretching and bending vibrations and second overtone of C=O stretching vibrations. The last region (2000-2600 nm) is more difficult to assign due to high number of possibilities for the coupling of vibrations [M. Schwanninger, J.C. Rodrigues, K. Fackler, (2011), *J. Near Infrared Spectrosc. 19, 287-308*].

Scanning Electron Microscopy

An example of SEM images of wood treated with CNC based polymers are presented in Figure 3. After impregnation, the wood cell wall structures are covered with a thin layer or polymer.

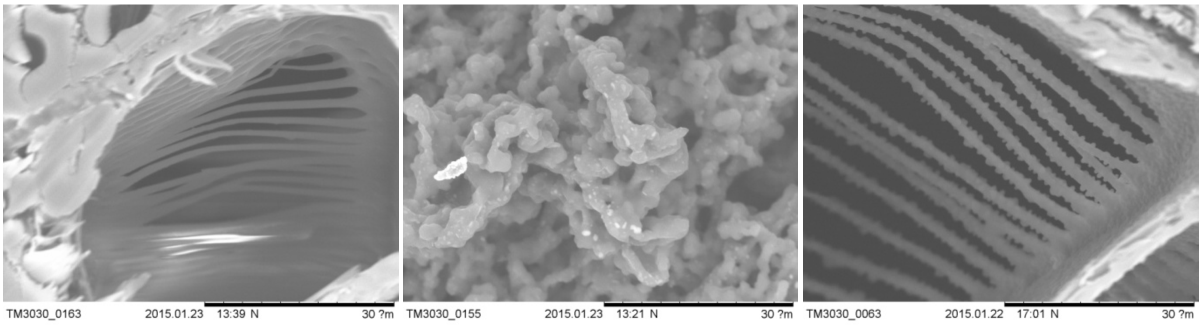

Figure 3. SEM images for birch wood, CNC based polymer and impregnated wood

ASE tests

The ASE tests were performed during all the STSM period. The tests were carried on in 4 steps for a number of 108 samples. In the first step, the samples were dried until no mass loss change was observed. This stage was followed by the impregnation of samples with 2 organic acids and their dry until no mass changes were observed. After this stage, the samples weredivided into 3 sets – 36 samples for each experiment. One set of samples was kept at 90 %RH and 23 ^{o}C, one was soached in water and kept there for 1 week at 23 ^{o}C and the third set was imersed in water and the weight was continuously monitored during 240h at 23 ^{o}C. After all tests, the weight and dimensional stability were evaluated.

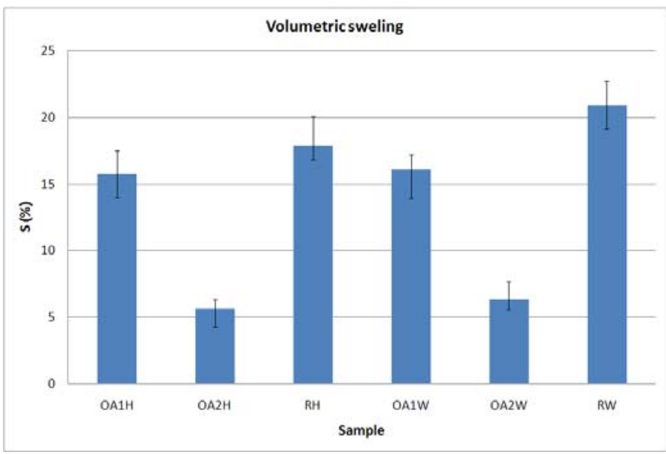

Figure 4. Volumetric swelling of wood sample treated with 2 organic acids

The samples treated with organic acids are more stable borth in weigh and dimensions when compared to the untreated sample (Figure 4). In addition, only small amounts of organic acids are lost during the tests, evidencing a good compatibility between wood and the organic acid.

Future collaboration with host institution

My collaboration with the host institution (BFH-AHB), especially with Dr. Marion Noel, in the near future will result in a co-authored paper, which will be sent to a peer-reviewed journal. We intend to continue collaboration both during COST FP1006 period and COST FP1303 and also by other projects in the HORIZON 2020 framework.

Other comments

From my point of view, the scientific mission has been a fruitful research experience which also opened several possibilities for further cooperation. It gave me an invaluable possibility to discuss various problems of my research area.

I wish to express my sincere gratitude to Marion Noel for the hospitality and the organization of my visit at BFH-AHB. It was a great pleasure for me to work with her and to work in the friendly atmosphere at the BFH-AHB.

I am also grateful for the financial support by the COST organization, which made this Short-Term Scientific Mission possible and for opening this opportunity to collaborate with BFH-AHB. This collaboration allows us to progress in our studies regarding the wood treatments and wood characterisation.

The Gantt diagram illustrates the distribution of the activities / day.

Work/analysis		Day																											
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
General	Travel from Iasi to Biel/Bienne	x																											
	Inspection of the facility and preparation of experiments		x																										
Modified wood	ASE test, humidity and water, of chemically modified samples		x	x	x	x	x			x	x	x	x	x			x	x	x	x	x			x	x	x	x	x	
Coated wood	Performing the micro-IR measurements			x	x	x	x			x	x	x																	
	Performing the NIR measurements and processing of the spectra											x	x	x			x	x	x	x									
	Optical microscopy measurements																				x			x	x	x	x		
	Scanning electro microscopy measurements																				x			x	x	x	x		
General	Laboratory work will be completed, acquired data will be compiled and results will be discussed																											x	
	Travel from Biel/Bienne to Iasi																												x
	Data processing, interpretation of the results and writing co-authored paper	will be carried out in Romania																											