

The use of NIR spectroscopy as a quality marker of hydrothermally treated wood

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Fast, non-destructive and efficient markers are currently required in the field of wood modification industry in order to be used for the estimation of the thermal treatment degree of wood (Willems *et al.* 2015). Spectroscopic methods were proved as valuable tools for non-destructive determination of many wood properties (Sandak *et al.* 2016). Hardness, mass loss as well as surface elasticity are physical properties that can be determined with minimal impact on wood and could also be used as markers of the thermal modification degree (Willems *et al.* 2015). Purpose of this collaboration was to evaluate the potential of NIR spectroscopy for non-destructive evaluation of hydrothermally treated wood. Beside of NIR, also other spectroscopic techniques were tested within this collaboration. The main steps of the research are presented along with some indicative results, since the data processing and analysis are still ongoing.

Beech (*Fagus sylvatica* L.) specimens with dimensions of 40mm x 40mm x 22mm were produced from defect-free sapwood. In order to facilitate comparison between treated and non treated wood, the specimens were cut in adjacent pairs, as explained in other works (Hansson and Antti 2006). Consequently half of the specimens were hydrothermally treated in a closed system using saturated steam at 110, 140, 170 and 200°C for intervals of 10, 30, 60, 120 and 240min. 5 specimens per treatment were used. All specimens were then conditioned in a constant climate of 65RH/20°C and used for the determination of Brinell hardness, surface elasticity. Hardness and surface elasticity were determined according to methods used by Niemz and Stübi (2000) and Rautkari *et al.* (2011). Spectra were acquired by using FT-NIR VECTOR 22-N (Bruker Optics GmbH) at 5 spots over each sample surface; four around corners of the sample surface containing the indentation mark of and one directly on the indentation area. A dedicated sample holder was constructed in order to assure measurements on the corresponding regions of both treated and reference specimens. The same procedure was performed on the respective paired samples and comparative spectra corresponding to treated and non-treated wood were acquired. The spectral range measured was between 4000cm⁻¹ and 12000cm⁻¹. Halogen lamp served as the source of infrared light. The system was able to measure the spectra with a resolution of 8cm⁻¹ and each spectrum has been computed as an average of 32 successive scans, in order to reduce the measurement error. All measurements were performed in the same climate conditions (climatic chamber 65RH/20°C.) In addition to the above measurements, FT-IR (in reflectance mode), UV-VIS (with probe and with integrating sphere) as well as custom made hyperspectral imaging system were used in order to acquire complementary information.

Spectra were preprocessed (extended multiplicative scatter correction) for multivariate analysis. Various chemometric methods were implemented for data analysis and mining. These included Principal Components Analysis, Partial Least Squares, 2D spectral correlation, among others. Particularly, PLS modeling was utilized to develop models predicting hardness, surface elasticity and mass loss change on the base of NIR spectra. The possibility for developing quality control routines based on NIR spectroscopy was also explored.

From the up to now analysis of the data very promising results came up concerning the correlation of treatment conditions against hardness, surface elasticity and mass loss. Multiple Linear Regression (MLR) analysis showed strong correlation of the predicted against the reference values. The results will facilitate better understanding of the degree of wood thermal modification. Proposed non-destructive methods might be used for on-line process control and for further optimization of the thermal treatment. It is expected that developed quality markers might assure well-defined improvement of material properties and contribute to the estimation of overall performance of wood as a building component. It should be clearly noted that due to the large volume of data acquired the analysis are still ongoing. The outcomes presented in this paper are only a small indicative part of the results that will come up upon completion of the analysis using all methods for all reference and estimated parameters.

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