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Quality control of vacuum thermally modified wood with near infrared spectroscopy

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A B S T R A C T

The thermo-vacuum process is an alternative technology for thermal modification of wood where reduction of oxygen concentration inside the reactor, necessary to avoid wood combustion, is obtained by applying vacuum. The goal of this research was to exploit the potential of near infrared spectroscopy for the quality control of TMW (prediction of mass loss and equilibrium moisture content in both softwoods and hardwoods), exposed to the thermal treatment in vacuum conditions. Applied chemometric techniques confirm great potentiality of the near infrared spectroscopy. Important parameters characterizing quality of TMW in vacuum conditions were predicted with high accuracy. Prediction errors of chemometric models based on near infrared spectra were relatively small; 0.9% and 0.36% in case of mass loss and equilibrium moisture content respectively. Such methodology might be helpful for development of on-line process control and for further optimization of the thermal treatment procedure at industrial scale.

The trend for substitution of traditional resources with novel of improved characteristics is observed nowadays in the wood-based industry. Thermally modified wood (TMW) is an example of such innovation that replaces timber in several applications. TMW is a timber in which the chemical composition of constitutive woody polymers and wood physical properties are modified by the exposure to high temperatures in conditions of reduced oxygen availability [1]. The typical range of the treatment temperatures ranges between 160 and 230 °C, and depends on expected treatment intensity. Thermally modified wood possesses superior durability against decay and weathering, enhanced dimensional stability, constant color, reduced thermal conductivity, lowered equilibrium moisture content and increased hygrophobicity.

Several techniques for production of TMW are commercially available nowadays. The thermo-vacuum process is a recently patented alternative technology suitable for thermal modification of wood. The reduction of oxygen concentration inside the reactor, necessary to avoid wood pyrolysis or combustion, is obtained by applying vacuum. Heating transfer from heaters to wood is provided to by forced convection. The thermo-vacuum is classified according to Hill [2] as a dry process in an open system, where the vacuum pump continuously removes all volatile compounds from the reactor. Volatiles (mostly polysaccharides) and the water vapour are catalysts accelerating the degradation phenomena in wood and their absence ensures a lower rate of wood mass loss (ML). Moreover, the removal of volatiles assures high energy efficiency, less corrosion and avoiding rust problems, when compare with alternative processes using superheated steam [2]. Preliminary results indicate that reduction of mechanical properties of vacuum TMW is not as significant as in alternative treatment technologies [3].

The production volumes of TMW in 2012 reached 315,000 m^3 in Europe and 100,000 m^3 in North America. The market is currently in a phase of intensive growth, with a number of new products, producers and users arising year by year. The main obstacle for the further advance of TMW is related to its certification, standardization and quality assurance. In this context the high variability of the product quality is an important issue, especially considering natural heterogeneity of wood highly affecting the treatment efficiency.

Fourier transform near infrared spectroscopy (FT-NIR) is a technique capable of fast and non-destructive measurement of organic materials. The important advantages are accuracy, simplicity, and ability of performing very high number of tests without needs of any destruction to the material. Quality assessment of thermally treated

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wood by means of NIR was previously researched by Esteves and Pereira [4]. Schwanninger et al. [5] obtained very good clustering of thermally modified beech wood and suggested possibility of spectroscopy utilisation for quality control. Analogical conclusions were presented by Bächle et al. [6] by applying Soft Independent Model Class Analysis (SIMCA) classification for three wood species. Attempt to understand chemical changes occurring during heat treatment of wood were made by several authors [7–9], but are often limited to a small number of species or narrow treatments conditions. Esteves and Pereira [4] obtained good Partial Least Squares (PLS) models based on NIR spectra for mass loss (*ML*) and equilibrium moisture content (*EMC*) but independently for two species: pine and eucalypt. Bächle et al. [10] developed PLS models for prediction of mechanical properties for thermally treated spruce and beech. Todorovič et al. [11] used color parameters (*CIE L*a*b**) for prediction of mechanical and physical properties of heat treated beech wood. Brischke et al. [12] proposed to use color measurement of homogenized samples for implementation in industrial quality control. Most of previously obtained prediction models are valid for single wood species. However, the development of single generalized model, capable of predicting modified wood properties for several species, and independently of the modification processes and/or producers is of great interest for both research and industry [13]. The goal of this research was, therefore, to develop such robust models for prediction of mass loss and equilibrium moisture content of soft- and hardwood species thermally modified in vacuum.

Eight wood species, representing softwood and hardwood: Norway spruce (*Picea abies*), Silver fir (*Abies alba*), European larch (*Larix decidua*), European beech (*Fagus silvatica*), oak (*Quercus* sp.), European ash (*Fraxinus excelsior*), Wild cherry (*Prunus avium*) and Black locust (*Robinia pseudoacacia*) were used as experimental samples. Five small blocks (50 mm × 40 mm x 10 mm, length x width x thickness respectively) from each batch were cut out from treated boards.

The thermal treatment was performed in the prototype plant, developed at the Drying Laboratory of IVALSA/CNR in collaboration with WDE-Maspell company. The prototype is based on a standard vacuum drying kiln heated by diathermic oil. The kiln is designed to work in high temperature ranges (up to 250 °C). The schema of thermo vacuum system is presented in Fig. 1 and consists of the following elements:

- A cell of treatment (1) vacuum-tight (autoclave) made of a stainless steel cylinder (Ø 1,7 m), a loading carriage (2) on which the pile of wooden sawn board (1 m^3 gross volume) (3) is deposited;
- A vacuum tight door (4);
- A heating system based on diathermic oil circulating in the double wall gap (5), oil heater/cooler, oil pump and the control RTD placed inside the chamber close to the internal wall (not shown on the schema);
- A ventilation system composed of high efficiency fans (speed: 1930 rpm, flow: 1 m^3 s^{-1}, static pressure: 50 Pa at 200 °C 100 mbar) (7), related external 4 kW electric motor (6) and motor-fan transmission shafts equipped with lubricated and cooled mechanical seals (8) used to transfer, via circuitry indoor air, the thermal energy from the reactor wall to the timber disposed on the pile;
- A group of vacuum pumping, composed of the suction pipe (9), the condenser (10), a recovery tank and storage of condensed vapours (11), the water ring vacuum pump (3,6 kW, ultimate pressure 50 mbar) (12), the reservoir (13) of recycling water of the vacuum pump (14) and the gas drain (15);
- A water cooling system of the vacuum pump consists of the recycle pump (16), the water–water exchanger (17), the evaporative tower (18), the water circulation pump of the tower (19), the cooling fan (20) and the cold water storage (21);
- A system for the evacuation of the condensate recovery tank (11), composed of the water pump (22), the electro-pneumatic valves (23) and (24), the tank (25), where the condensate is stored before its proper disposal;
- The cell (reactor chamber) is isolated from the external environment through the thermal insulation (26). The treated wood pile is formed by layers of boards and/or semi-finished wood separated by spacer strips to allow the passage of the heating fluid.

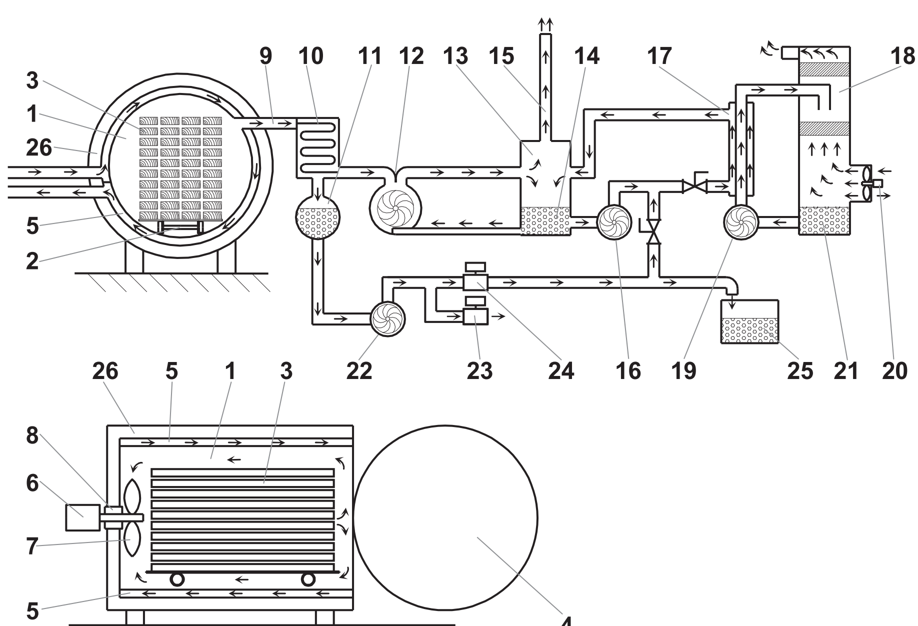

Fig. 1. The schema of thermo-vacuum system (detailed explanation of components described in the text).