



Comparison of chemical composition and decay durability of heat treated wood cured under different inert atmospheres: Nitrogen or vacuum

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ABSTRACT

Wood heat treatment is an attractive alternative to improve decay resistance of low natural durability wood species. Nowadays, several types of thermal treatments of wood exist. These treatments differ mainly by the nature of the inert atmosphere used to avoid wood combustion which may correspond to: nitrogen, vacuum, steam or oil. Decay resistance is strongly correlated to thermal degradation of wood cells wall components and consequently of treatment conditions from which depend chemical modifications. The aim of this study is to determine the effect of a new generation of treatment performed under vacuum on the chemical modifications occurring during treatment. For this purpose, one hardwood (beech) has been treated under different conditions (vacuum, nitrogen, steam). All treatments were performed at 220 °C for mass losses resulting from wood thermodegradation of approximately 12%. For each treatment condition extractives, Klason lignin, hemicelluloses and α -celluloses content were determined as well as monosaccharide composition. Intensity of thermodegradation was also evaluated by TD-GC-MS. Results show that extracts content were lower in the case of wood samples treated under vacuum, while lignin, hemicelluloses and α -celluloses contents were higher in the case of samples treated under steam and nitrogen, indicating lower wood degradation under vacuum treatment. These results were confirmed by chromatography analyses according to the nature and quantity of thermodegradation volatile products formed during TD-GC-MS analyses.

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1. Introduction

Wood heat treatment by mild pyrolysis is used to improve wood properties such as its decay durability and dimensional stability [1,2]. These new properties are the result of chemical modifications of wood cell wall polymers occurring during treatment which confer to wood new properties [3–5]. Different thermomodification processes are currently used in Europe and all over the world: Thermowood®, OHT MenzHolz®, Plato®... The main difference between these processes is the nature of the inert atmosphere and the curing conditions used during the heat treatment. Previous studies have shown that degradation of wood depends on heat treatment intensity directly connected to treatment temperature and duration conditioning the final properties of heat treated wood [6]. Elemental wood composition has been reported as a good marker of treatment intensity and consequently of mass losses issued from the different degradation reactions allowing further

prediction of decay durability of heat treated wood [7–9]. According to our previous experiment, mass losses comprised between 10 and 14% are generally required to reach full durability of treated wood towards classical brown rot and white rot fungi usually tested in wood durability standards [8–11]. However, even if different studies have been performed on the effect of treatment intensity (time and temperature) on conferred properties of the material, much less has been reported on the effect of inert atmosphere utilized during the process. Indeed, it is obvious that inert atmosphere used during treatment may impact directly thermodegradation reactions and consequently the final properties of the material. More recently, some studies reported a new thermodegradation process based on the use of vacuum as inert atmosphere [12–14]. Even if the effect of vacuum has not been yet fully investigated, it is postulated that vacuum removes all volatile degradation by-products limiting acidic accelerated degradation of polysaccharides and leading to an odorless material at the end of the treatment. Allegretti et al. described that vacuum thermally modified wood presents quite similar behavior compared to classical thermally modified wood with important correlations between treatment intensity, mass loss, EMC and color [14].

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Contrary to the conventional convection processes, heat treatment under vacuum is generally performed by conduction allowing a better homogeneity of the process. Risks of wood deformation are reduced due to two metallic plates surrounding the piece of wood [15]. However, even if some papers have been reported on heat treatment under vacuum, no data have been reported on the effect of such treatment on wood chemical composition. The aim of this study is to determine the influence of vacuum or nitrogen on the chemical modifications occurring during treatment. For this purpose, beech wood has been treated to reach similar mass loss of 12% under different conditions vacuum or nitrogen and the effect of treatment conditions evaluated on chemical and elemental composition of wood and on its decay durability.

2. Material and methods

2.1. Wood samples and heat treatment protocols

Heat treatment was performed simultaneously on two boards of beech (*Fagus sylvatica*) of 110 × 650 × 25 mm (T × L × R) in a 0.25 cubic meter laboratory autoclave by conduction between two electric heated metallic plates equipped to record dynamic mass loss as a function of time and temperature (SEIR, Charmes France). Each board was initially dried at 103 °C for 48 h and placed in the oven between two metallic plates. The oven is closed and placed under nitrogen or vacuum (200 mbar). The plate temperature was slowly increased by 0.3 °C min⁻¹ from ambient to drying temperature (103 °C) until complete stabilization of the mass of the boards. After this period, the plate's temperature was increased by 0.3 °C min⁻¹ from 103 °C to 170 °C and the temperature maintained for 2 h. The temperature was then increased by 0.2 °C min⁻¹ from 170 °C to 230 °C to perform thermomodification of wood until a mass loss of 12% was obtained. Heating system is then stopped and wood samples allowed to cool down to room temperature under inert atmosphere.

2.2. Determination of chemical composition

All experimental procedures concerning extractive, holocellulose, α -cellulose and Klason lignin determination were adapted from literature procedures with minor modifications [16].

2.2.1. Extractives content

A part of the board was ground and passed through different sieves to obtain particles ranging between 0.2 and 0.5 mm. This sawdust was successively washed in a Soxhlet extractor with a toluene/ethanol (2:1, v/v) mixture (6 h), followed by ethanol (6 h) and dried at 103 °C for 48 h.

2.2.2. Holocellulose content

500 mg of extracted sawdust were placed in a 100 mL flask containing 30 mL of distilled water and heated at 75 °C. Acetic acid (0.1 mL) and 15% aqueous sodium chlorite (2 mL) were then added each hour for 7 h. The mixture was filtered on a Büchner funnel and the residue washed with water, Soxhlet extracted for 2 h with ethanol and dried at 103 °C to a constant mass.

2.2.3. Hemicellulose and α -cellulose contents

The dried holocellulose prepared as above was placed into a 250 mL glass beaker covered with a glass cover containing 10 mL of 17.5% NaOH solution and maintained at 20 °C in a water bath. This mixture was agitated with caution with a rod glass until the holocellulose was soaked with the NaOH solution. 5 mL of 17.5% NaOH solution were then added every 5 min during 15 min and the mixture left for 30 min. The mixture was then diluted with 33 mL of

distilled water and kept for 1 h before filtration on a Büchner funnel. The crude α -cellulose residue was successively washed with 100 mL of 8.3% NaOH solution, 100 mL of distilled water, 15 mL of 10% acetic acid solution and finally 250 mL distilled water.

The residual α -cellulose was dried at 103 °C to a constant weight and the hemicellulose content calculated as follows:

$$\text{Hemicellulose(\%)} = \text{Holocellulose(\%)} - \alpha\text{-cellulose(\%)}$$

2.2.4. Klason lignin content

500 mg of sawdust were mixed with 72% H₂SO₄ (10 mL) for 4 h at room temperature. The mixture was then diluted with 240 mL of distilled water, heated under reflux for 4 h and filtered. The residue of Klason lignin thus obtained was washed with hot water and dried at 103 °C to a constant weight. The different filtrates were precariously transferred in a 1 L volumetric flask, rinsed with distilled water and completed to 1 L for further monosaccharide content determination.

2.3. Elemental composition

Wood was grounded to fine sawdust and passed through different sieves to obtain a powder of granulometry comprised between 0.2 and 0.5 mm. Sawdust was conditioned at 103 °C for 24 h and stored in closed bottle before analysis. Elemental analyses were performed on a Thermofinnigan Flash EA1112 micro-analyzer.

2.4. Monosaccharide content

Separation and quantification of neutral sugars were performed using a Dionex ICS-3000 system consisting of an SP gradient pump, an AS autosampler, an ED electrochemical detector with a gold working electrode, an Ag/AgCl reference electrode and Chromeleon version 6.8 (Dionex Corp., USA). A CarboPac PA1 (4 × 250 mm, Dionex) column with a guard column (4 × 50 mm, Dionex) was used as a stationary phase using isocratic conditions with 1 mM sodium hydroxide as the eluent. Eluents were prepared by dilution of a 46–48% NaOH solution (PA S/4930/05 Fisher Scientific) in ultrapure water. All eluents were degassed before use by flushing helium through for 20 min; subsequently they were kept under a constant helium pressure (eluent degassing module, Dionex). After each run, the column was washed for 10 min with 200 mM NaOH solution and reequilibrated for 15 min with the starting conditions. Samples of monosaccharides (cf. 2.2) were injected through a 25 μ L full loop and separations were performed 25 °C at a rate of 1 mL/min. The pulse sequence for pulsed amperometric detection consisted of a potential of +100 mV (0–200 ms), +100 mV integration (200–400 ms), –2000 mV (410–420 ms), +600 mV (430 ms), and –100 mV (440–500 ms).

2.5. Thermodesorption of volatile by-products

Different heat treated wood and the untreated wood were grounded to fine sawdust and passed through different sieves to obtain a powder of granulometry comprised between 0.2 and 0.5 mm. Sawdust was conditioned at 103 °C for 24 h and stored in closed bottle before analysis. Approximately 30–40 mg of dried sawdust was placed in a glass thermal desorption tube with ¼ inch outer diameter and 3.5 inches long from Supelco, Inc and end capped with glass wool. Thermal treatment was performed in a Turbomatrix 300 Thermal Desorber system from Perkin Elmer, USA. The conditions included a pre-purge for 1 min at room temperature using helium carrier gas at 1 mL min⁻¹ followed by heating at 230 °C for 15 min in the glass thermal desorption tube of

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