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Comparison of mechanical properties of heat treated beech wood cured under nitrogen or vacuum

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ABSTRACT

Heat treated wood has been subjected to increasing interest during the last decade. This non biocidal treatment is an attractive alternative with a low environmental impact to improve decay resistance of low natural durability wood species. Nowadays, several types of heat treatment processes exist. These treatments differ mainly by the nature of the inert atmosphere used to avoid combustion of wood: nitrogen, steam pressure, oil or more recently vacuum. We have shown in a previous study that utilization of vacuum to perform thermal treatment instead of nitrogen allows to reduce considerably degradation of wood polysaccharrides. Indeed, it appears that for a similar 12% mass loss generated by thermal degradation, thermodegradation performed under vacuum allowed to reduce degradation of sugar constitutive of hemicelluloses and formation of recondensation products within the wood structure. These results may be explain by the effect of vacuum allowing removal of volatile degradation products like organic acids, aldehydes and furans limiting therefore acidic degradation of polysaccharides and recondensation of volatile by-products. Decay durability tests, performed against different brown and white rots fungi, have shown no significant differences for vacuum and nitrogen heat treated samples, all presenting an improved decay resistance.

According to these results, the aim of this short paper is to report the effect of vacuum versus nitrogen on the mechanical properties of heat treated samples making the assumption that lower degradation of wood polysaccharides during treatment performed under vacuum may reduce the weakening of mechanical properties. For this purpose, modulus of rupture (MOR) and modulus of elasticity (MOE) in bending and Brinell hardness (HB) were determined. Results show that the mechanical properties were lower degraded in the case of wood samples treated under vacuum.

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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]. Previous studies have shown that the mass loss of wood due to thermodegradation reaction is a good indicator for the treatment intensity depending directly on temperature and duration of heat treatment [4]. It has been also demonstrated that the decay durability of heat treated wood can be

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predicted by mass loss [5–7]. According to our previous experiments, mass losses between 10 and 14% are generally required to reach full durability of treated wood towards classical brown rot and white rot fungi recommended in wood durability standards [6–9]. In parallel with improvement of wood decay durability, the mechanical properties were generally greatly weakened [10,11]. Degradation of hemicelluloses, which connects cellulose and lignin in the cell wall, causes deterioration in strength of wood. Deterioration in toughness, hardness, bending, compression, and tension strength due to heat treatment have been reported in the literature [12,13]. The degree of deterioration of the mechanical properties is a function of the heat treatment conditions and intensity [14]. The mechanical property loss was explained as the result of hemicelluloses degradation, increasing crystalline cellulose content and decreasing the flexibility supra-molecular arrangement of wood cell wall components [15].

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Recently, vacuum was proposed as a solution to assure inert atmosphere during wood thermal treatment [16–18]. In a previous study, we have shown that inert atmosphere used to perform wood thermomodification (vacuum versus nitrogen) impacted directly the nature of wood thermodegradation reactions leading to materials with different chemical compositions [19]. Heat treated wood under nitrogen shows higher Klason lignin and carbon content, lower hemicelluloses and neutral monosaccharides contents comparatively to heat treated wood under vacuum. These results were explained by the removal of volatile degradation by-products during treatment performed under vacuum limiting therefore acidic degradation of polysaccharides due to formation of acetic acid and recondensation of volatile degradation products within the wood structure [20,21].

Effect of heat treatment performed under vacuum was also investigated on decay durability and compared to treatment performed under nitrogen leading to similar mass losses due to thermodegradation reactions (ML = 12%). Even if chemical composition of both heat treated samples were not exactly the same, results indicated no significant differences according to the nature of the inert atmosphere used indicating limited effect of the level of polysaccharide's degradation on resistance to basidiomycetes, however effect of chemical composition on mechanical properties has not been investigated.

The aim of this study is to determine the effect vacuum used during treatment on mechanical properties of the material. For this purpose, beech wood samples have been treated under vacuum and nitrogen and their mechanical properties evaluated. All treatments were performed at 230 °C for 12% mass losses resulting from wood thermodegradation and MOR and MOE in bending and Brinell hardness (HB) were determined.

2. Material and methods

2.1. Wood samples and heat treatment protocols

Heat treatment was performed simultaneously on tree boards of beech (*Fagus sylvatica*) of $110 \times 650 \times 25 \text{ mm}$ (T \times L \times R) in a 0.2 cubic meter laboratory autoclave by conduction between two electric heated metallic plates meant 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 reached. Heating system is then stopped and wood samples allowed to cool down to room temperature under inert atmosphere.

2.2. Mechanical properties

In order to assess the effect of heat-treatment parameters on the mechanical properties, three point bending (MOE, MOR) and Brinell hardness were carried out for untreated samples and heat-treated samples under different conditions, and the results compared. INSTRON 4467 Universal Mechanical Test Machine was used for the measurements. Samples were conditioned in a room with 65% RH and 22 °C during the necessary time to stabilize the samples weights.

Three point static bending tests were carried out according to The EN 408 (2003) standard. The size of the samples was 0.20 m \times 0.01 m \times 0.01 m (L \times R \times T). The moving head speed and the span length were 1.8 mm s⁻¹ and 160 mm, respectively. The load deformation data obtained were analyzed to determine the modulus of elasticity (MOE) and the modulus of rupture (MOR). Tests were repeated twelve times (4 samples per heat treated boards) for each treatment condition.

Brinell hardness tests were performed in accordance to EN 408 (2003) standard. The force was applied by a sphere with a diameter of 10 mm. This force is applied in three steps. It was slowly increased by 0.2 kN s⁻¹ during 15 s. After this period, the force was maintained for 25 s and finally the applied force was decreased. Brinell hardness tests were repeated twelve times (4 tests per wood boards). Every test was separated by at least 30 mm from the edge of the boards and 25 mm between each test. Accuracy of the measurement of the ball penetration depth was ± 0.01 mm and that of the applied force was ± 0.005 kN.

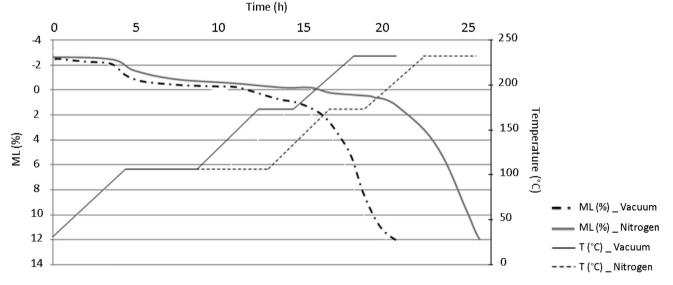


Fig. 1. Effect of inert atmosphere (nitrogen or vacuum) on thermal treatment of beech wood cured under similar conditions.

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