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ADVANTAGE OF VACUUM VERSUS NITROGEN TO ACHIEVE INERT ATMOSPHERE DURING SOFTWOOD THERMAL MODIFICATION

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Abstract:

Wood heat treatment is an attractive alternative to improve decay resistance of wood species with low natural durability. Durability and mechanical properties are strongly correlated to thermal degradation of wood cells wall components. Mass loss resulting from this degradation is a good indicator of treatment intensity and final treated wood properties. Several types of convective heating processes exist currently differing mainly by the nature of the inert atmosphere used during treatment: nitrogen, steam or oil. Conductive heat treatment using vacuum as inert atmosphere is an attractive new alternative to previous classical methods. Heat transfer by conduction has been reported to provide better treatment homogeneity than heat transfer using convection. The aim of this study is to investigate the effect of vacuum comparatively to nitrogen on the thermal degradation pathways and on the conferred properties to the material. It appears that utilization of vacuum permit a better control of thermal degradation reactions limiting the mass loss resulting from degradation of wood cell wall polymers. Chemical analysis indicates that wood heat treated under nitrogen present higher Klason lignin and carbon contents, lower hemicelluloses and neutral monosaccharides contents comparatively to wood heat treated under vacuum. At the same time, mechanical properties are less affected under vacuum, which constitute another advantage of this technology.

Key words: *chemical composition; mechanical properties; nitrogen; silver fir; thermal degradation; vacuum.*

INTRODUCTION

Wood thermal treatment by mild pyrolysis changes physical and chemical properties of the material (Viitaniemi *et al.* 1993). Thermal modification leads to reduced mechanical properties but dimensional stability and biological durability of wood are increased without adding biocides (Rowell *et al.* 2009, Ponsack *et al.* 2010, Yildiz *et al.* 2002). These new properties are the result of chemical modifications of wood cell wall polymers occurring during heat treatment (Tjeerdma *et al.* 2005, Yildiz *et al.* 2006). Reported in the

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literature effects of the high-temperature heating on wood mechanical properties vary, because of natural wood chemical and anatomical variability and the different methods of heating (Hillis *et al.* 1984).

Several thermal modification processes are currently used in Europe and all over the world: Thermowood®, OHT MenzHolz®, Plato®. These processes differ mainly by the nature of their inert atmosphere and the used curing conditions. Previous studies have shown that wood degradation and final end properties depend on heat treatment intensity directly linked to treatment temperature and duration (Yildiz *et al.* 2006). The mass loss of wood issued from the different degradation reactions is also reported as a good indicator of treatment intensity allowing prediction of decay durability of heat treated wood (Nguila *et al.* 2007a, Hillis *et al.* 1984, WElzbacher *et al.* 2007). Elemental analysis is another parameter of heat treated wood quality. Oxygen and carbon content are also correlated to Mass Loss generated by thermal degradation and Weight Loss due to fungal attacks (Nguila *et al.* 21009, Šušteršic *et al.* 2010, Chaouch *et al.* 2010). According to previous experiments, 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 (Hakkou *et al.* 2005, Mburu *et al.* 2007).

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 (Hill 2006). Indeed, it is obvious that inert atmosphere used during treatment may impact directly thermal degradation reactions and consequently the final properties of the material. More recently, some studies reported a new thermal degradation process based on the use of vacuum as inert atmosphere (Rep *et al.* 2004, Surini *et al.* 2012, Allegretti *et al.* 2012). 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 (Allegretti *et al.* 2012). Our previous study has shown that chemical composition of heat treated beech wood under vacuum is less modified than those heat treated under nitrogen (Candelier *et al.* 2013a). It seems that vacuum removes volatile degradation products limiting therefore acidic degradation of polysaccharides due to formation of acetic acid and recondensation of volatile degradation products within the wood structure. In addition, the utilization of vacuum during thermal treatment of wood allows reducing the drying time necessary to stabilize wood mass before thermal modification step limiting the overall treatment duration (Candelier *et al.* 2013b). This last study has also shown that mechanical properties of heat treated beech wood under vacuum are less affected than those heat treated under nitrogen. These results shown that heat treated under nitrogen wood presents lower MOR and MOE in bending and lower Brinell hardness comparatively to the heat treated under vacuum wood.

The aim of this study is to investigate the influence of vacuum or nitrogen atmosphere on the chemical and mechanical modifications occurring during heat treatment performed on softwood specie (silver fir) and also on wood thermal degradation kinetics. For this purpose, silver fir wood has been treated at 230°C to reach similar mass loss of approximately 12%, under vacuum or nitrogen, and the effect of the inert atmosphere on wood chemical and elemental composition as well as its mechanical properties was evaluated.

MATERIAL AND METHODS

Wood samples and heat treatment protocols

Heat treatment was performed simultaneously on two boards of silver fir (Abies pectinata Lam) of 110 x 650 x 25mm^3 (T x L x R) in a 0.2 cubic meter laboratory autoclave by conduction between two electrically heated metallic plates. The device is 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 hours and placed in the oven between two metallic plates. The oven is closed and placed under nitrogen or vacuum (200mbar). 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°Cmin^{-1} from 103°C to 170°C and the temperature maintained for 2h. The temperature was then increased by 0.2°Cmin^{-1} from 170°C to 230°C to perform thermal modification of wood until a mass loss of 12% was obtain. Heating system is then stopped and wood samples allowed to cool down to room temperature under inert atmosphere.

Chemical composition determination

Extractives content

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

Holocelluloses content

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

Hemicellulose and α-cellulose contents

The dried holocelluloses prepared as above was placed into a 250mL glass beaker covered with a glass cover containing 10mL 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 holocelluloses was soaked with the NaOH solution. 5mL of 17.5% NaOH solution were then added every 5 minutes during 15 minutes and the mixture left for 30 minutes. The mixture was them diluted with 33mL of distilled water and kept for 1 hour before filtration on a büchner funnel. The crude α-cellulose residue was successively washed with 100mL of 8.3% NaOH solution, 100mL of distilled water, 15mL of 10% acetic acid solution and finally 250mL distilled water.

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

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

Klason lignin content

500mg of sawdust were mixed with 72% H_2SO_4 (10mL) for 4 hours at room temperature. The mixture was then diluted with 240mL of distilled water, heated under reflux for 4 hours and filtered. The residue of Klason lignin thus obtained was washed with hot water and dried at 103°C to a constant weight.

Elemental composition

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

Monosaccharide content

Separation and quantification of neutral sugars were performed using a Dionex ICS-3000 system consisting on a 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 × 250mm, Dionex) column with a guard column (4 × 50mm, Dionex) was used as a stationary phase using isocratic conditions with 1mM sodium hydroxide as 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 20min; subsequently they were kept under a constant helium pressure (eluent degassing module, Dionex). After each run, the column was washed for 10min with 200mM NaOH solution and reequilibrated for 15min with the starting conditions. Samples were injected through a 25µL full loop and separations were performed 25°C at a rate of 1mL/min. The pulse sequence for pulsed amperometric detection consisted in a potential of +100mV (0–200ms), +100mV integration (200-400ms), −2000mV (410-420ms), +600mV (430ms), and -100mV (440-500ms).

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 are 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 stabilise the samples weights.

Three point static bending tests were carried out according to the EN 408 (2003) standard. The size of the samples was 0.20m x 0.01m x 0.01m (L x R x T). The moving head speed and the span length were respectively 1.8mm.s^{-1} and 160mm. The load deformation data obtained were analysed 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 0.2kN.s^{-1} during 15s. After this period, the force was maintained for 25s and finally the applied force was cooled down. Brinell hardness tests were repeated twelve times (4 tests per wood boards). Every test was separated by at least 30mm from the edge of the wooden sample and 25mm separated each test.

RESULTS AND DISCUSSION

Kinetics of thermal degradation

Silver fir mass losses recorded for similar treatment intensity under nitrogen or vacuum are presented in Fig. 1.

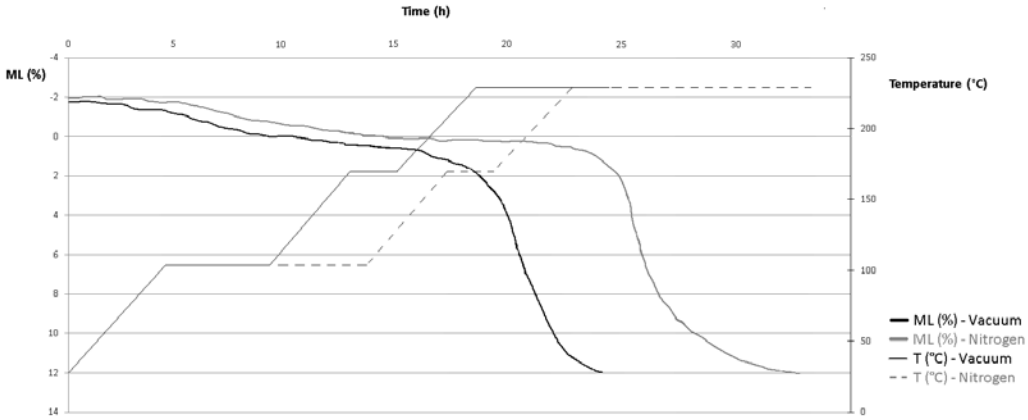

Fig. 1.
Effect of inert atmosphere (nitrogen or vacuum) on mass loss due to thermal treatment of silver fir wood.

Until 170°C, only very small mass losses were observed corresponding to vaporization of volatile extractives and desorption of wood fibres' bound water. Thermal degradation reactions begin for higher temperatures (close to 220°C) and are fully effective at 230°C. The main difference between treatment performed under nitrogen and vacuum is the reduction of duration necessary to reach a given mass loss under vacuum due to an improved drying of the sample. Indeed, the increase of treatment temperature after the 103°C drying phase is conditioned by the stability of the sample mass indicating that this latter one is the oven dried mass allowing calculation of mass loss. Such result was already observed for the beech wood that vacuum allows reducing the drying step duration (Candelier *et al.* 2013b). This study confirms also the vacuum effect on the drying on softwood specie (silver fir wood). At higher temperatures, mass losses for treatment performed under vacuum are slightly higher by comparison with these ones recorded for treatment performed under nitrogen. This observation is in accordance with previous results and it indicates that heat treated wood under vacuum seems to be more affected than those treated under nitrogen, for a same treatment intensity and whatever the nature of wood specie (hardwood or softwood).

Chemical modifications

Fig. 2 describes the content of the main components of silver fir wood before and after its thermal treatment performed either under nitrogen or under vacuum.

As both heat treatments performed on beech wood (Candelier *et al.* 2013a), similar results were found for treatment with same intensity (ML=12%) performed on silver fir. These results confirm the influence of vacuum on the chemical composition evolution of treated wood compare to a similar treatment carried out under nitrogen. It results that extractives content after thermal treatment under vacuum is slightly lower than this obtained after thermal treatment under nitrogen for a similar mass loss of 12% confirming the evaporation of low molecular weight compounds generated during wood thermal degradation. Lignin content is higher in the case of thermal treatment carried out under nitrogen comparatively to this performed under vacuum. Holocelluloses as well as hemicelluloses and α-cellulose contents decrease more significantly for treatment performed under nitrogen. These results may be easily explained by the effect of vacuum. Under vacuum, all volatile degradation products like acetic acid or furfural are removed progressively as soon as they are formed limiting the degradation of wood polysaccharides and the recondensation of degradation products through thermal reticulation and crosslinking reaction.

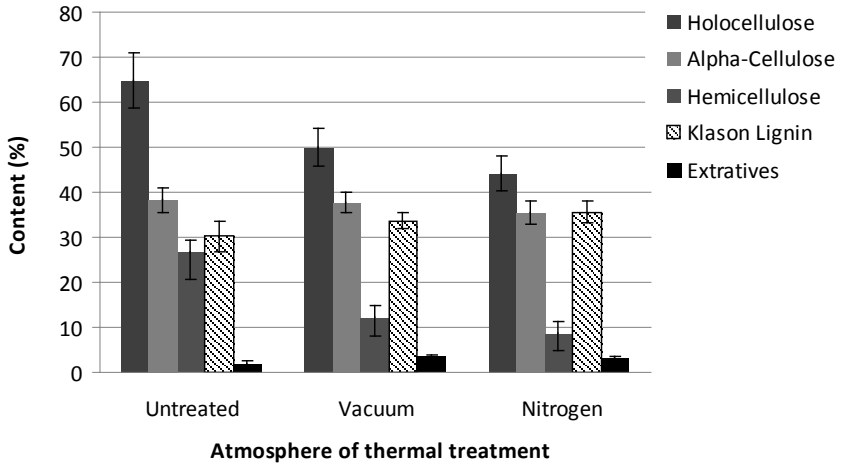

Fig. 2.
Chemical composition of untreated and heat treated silver fir wood under different inert atmospheres.

To confirm the behavior of wood polysaccharides during thermal treatment and check the influence of inert atmosphere, analysis of monosaccharides resulting from acidic hydrolysis of wood during Klason lignin determination was performed using ionic chromatography. Results are presented in Table 1. Untreated samples present classical neutral sugar composition according to literature data indicating mainly arabinoglucuronoxylan (5-10%) and small amounts of galactoglucomannan (20%) as the main components of softwood hemicelluloses (Sjöström 1981). After thermal treatment, the content of all sugars constitutive of hemicelluloses decreases confirming the high susceptibility of these latter ones to thermal degradation (Esteves *et al.* 1981). Percentages of all monosaccharides are lower for treatment carried out under nitrogen comparatively to treatment performed under vacuum indicating a higher level of degradation of wood's polysaccharides. Heat treatment carried out under vacuum allows limiting polysaccharides degradation both during a treatment on softwood as hardwood (Candelier *et al.* 2013a).

Table 1

Monosaccharide composition of untreated and heat treated silver fir wood according to the sort of thermal treatment

		monosaccharides				
Sample	**Mass loss, %**	**Arabinose**	**Galactose**	**Glucose**	**Xylose**	**Mannose**
Untreated	0	1,07	1,58	52,95	21,78	11,22
Vacuum	12	0,31	0,51	50,37	11,62	3,27
Nitrogen	12	0,29	0,47	49,23	11,54	3,14

As previously reported, thermal treatment leads to a raise of carbon content, while oxygen decreases leading to an important decrease of O/C ratio after thermal treatment (Nguila *et al.* 2009, Nguila *et al.* 2006, Nguila *et al.* 2007b). Fig. 3 shows that the increase of carbon content is more important in the case of treatment performed under nitrogen indicating higher degradation of wood polysaccharides and/or higher amounts of carbonization products within the wood structure. These results correspond to those found previously (Candelier *et al.* 2013a).

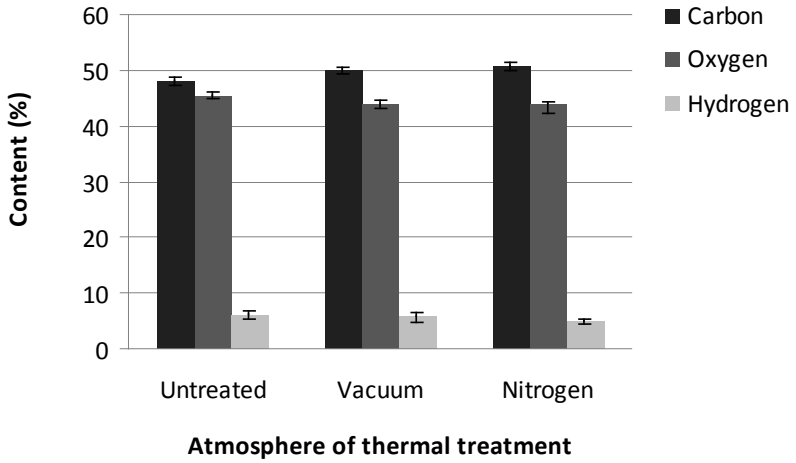

Fig. 3.
Elemental composition of untreated and heat treated silver fir wood under different inert atmospheres.

Considering that the main effect of vacuum is to remove progressively volatile wood degradation by-products limiting acidic hydrolysis of wood's polysaccharides and recondensation reactions, the mass loss of 12% recorded for treatment performed under vacuum corresponds effectively to real mass loss of 12%, while this recorded for treatment performed under nitrogen correspond probably to an higher mass loss due to the recondensation of degradation products which distorts the true mass loss value. These results indicate lower wood degradation under vacuum treatment.

Consequently, mass loss of 12% recorded for treatment performed under nitrogen is underestimated leading to higher degradation of wood polymers explaining the lower hemicelluloses content, the lower monosaccharides contents and the higher carbon content. The vacuum effect during softwood (silver fir) heat treatment on final chemical composition is similar than a heat treatment performed under vacuum on a hardwood specie (Candelier *et al.* 2013a).

Mechanical properties

Mechanical properties of thermally modified spruce, fir, and other softwoods under a wide range of conditions are available in literature (Shi *et al.* 2007, Finnish Thermowood association 2003). These mechanical properties are also reduced after a heat treatment performed under vacuum on Norway spruce and fir wood (Allegretti *et al.* 2012). The mechanical tests results of samples conditioned at a temperature of 20°C and 65% RH are shown on Fig. 4. All mechanical properties, Brinell hardness, MOE in bending and more particularly MOR in bending are less affected after vacuum thermal treatment comparatively to treatment performed under nitrogen. For both thermal treatment (nitrogen and vacuum), silver fir modulus of elasticity (MOE) decreased slightly after treatment. Similarly to previous studies, this decrease is lower in the case of vacuum heat treated wood confirming the lower degradation of wood's polysaccharides measured before (Candelier *et al.* 2013b). The decrease of the MOR in bending is more pronounced. MOR is more degraded in the case of treatment performed under nitrogen confirming the higher degradation of wood polymers during treatments performed under nitrogen. MOR decreases respectively of 50% and 42% after nitrogen and vacuum heat treatment comparatively to untreated samples. The Brinell hardness decreases of 40% and 28% for nitrogen and vacuum heat treated silver fir.

A statistical analysis of data of MOE, MOR' and HB'' (one-way analysis of variance) using Fisher test with the JMP 10.0.2 program (SAS Institute Inc., Cary, NC, USA) allows to class results from each mechanical properties in three categories from A to C as indicated in Fig. 4. Systems not connected by the same letter are largely different, at the 5% level. This analyze highlighted the fact that thermal modification atmosphere has a significant effect on the modulus of rupture of wood samples heat treated for a similar mass losses, while effects on modulus of elasticity and Brinell hardness are less obvious.

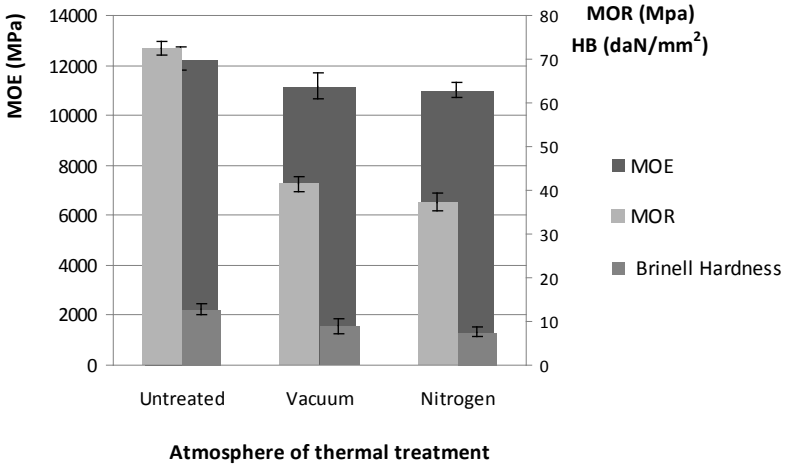

Fig. 4.
Evolution of mechanical properties after thermal treatment of silver fir wood correlated to inert atmosphere.

Indeed, according to ANOVA analysis, values obtained for MOE and Brinell hardness of samples heat treated under vacuum are not significantly different at 5% level from those of the untreated controls. Meanwhile, values obtained for MOE and Brinell hardness of samples heat treated under vacuum are significantly different at 5% level indicating the benefits of using vacuum to create the inert conditions for heat treatment.

CONCLUSION

The aim of this study was to confirm the effect of inert atmosphere, nitrogen versus vacuum, on the thermal degradation reactions occurring during softwood thermal treatment. For this purpose, chemical and mechanical analyses have been performed on untreated and treated silver fir wood. Results confirm that silver fir, as well as beech heat treated under vacuum present lower Klason lignin and carbon content, higher hemicelluloses and neutral monosaccharides contents comparatively to wood heat treated under nitrogen. These results may be explain by the effect of vacuum allowing removal of volatile degradation products limiting therefore acidic degradation of polysaccharides due to formation of acetic acid and recondensation of volatile degradation products within the wood structure. The influence of vacuum is supported by weakening of mechanical properties occurring during wood thermal treatment. Results show that wood heat treated under nitrogen presents lower MOR and MOE in bending and lower Brinell hardness comparatively to wood heat treated under vacuum. Among the three investigated mechanical properties, MOR was the most sensitive property to the heat treatment conditions. Wood samples heat treated under vacuum are consequently less degraded mechanically than heat treated under nitrogen samples, whatever the nature of wood specie, which constitute another advantage of this vacuum process technology. Finally, the utilization of vacuum during thermal treatment of wood allows also reducing the drying period necessary to stabilize wood mass before thermal modification step limiting the overall treatment duration.

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