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Giovanna Puccioni, *Accademia Italiana di Scienze Forestali*

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## IMPROVING TIMBER PROPERTIES OF FAST GROWING SPECIES BY HEAT TREATMENT

Giacomo Goli<sup>1</sup>, Lorenzo Barnini<sup>1</sup>, Linda Cocchi<sup>1</sup>, Giuseppina Di Giulio<sup>1</sup>, Bertrand Marcon<sup>1</sup>,  
Paola Mazzanti<sup>1</sup>, Giovanni Signorini<sup>1</sup>, Marco Fioravanti<sup>1</sup>

<sup>1</sup>GESAAF, Università di Firenze; giacomo.goli@unifi.it

Heat treatment is a useful tool to improve some properties of wood and give new market to fast growing species. The effect of heat treatment in saturated steam was tested on poplar wood and its effect on some physical and mechanical properties determined. It was shown that the mass loss is well dependent on the treatment intensity and that the colour variation presents a linear trend with the mass loss. Surprisingly the volume after treatment resulted increased. Weak heat treatments up to 4% of mass loss have an increasing effect on MOE. After 4% of mass loss MOE tends to decrease getting lower of untreated samples once 5% of mass loss is reached. According to FTIR analysis the main effect of the heat treatment can be identified in the degradation of the hemicelluloses and in particular of the glucoxylans. A chemical degradation of the hemicelluloses can be observed by FTIR since very low mass losses.

*Keywords:* heat treatment, saturated steam, poplar wood, mechanicals behaviour, colour variation.

*Parole chiave:* trattamento termico, vapore saturo, legno di pioppo, comportamento meccanico, variazione di colore.

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### 1. Introduction

Poplar wood is a material widely used in Italy as solid timber or as a raw material for wood-engineered products such as plywood or particleboards. According to recent studies (Brega, 2011), now days the yearly need of poplar wood in Italy is around  $3.9 \times 10^6$  m<sup>3</sup> whom one half are home grown. Poplar (*Populus nigra* L.) and in particular Euro-American clone I-214 is a fast growing specie very appreciate for applications where the lightness is an essential requirement. A limit of poplar wood, as for many fast growing species, is a lack of durability. In order to give new market to these products a research was planned in order to verify the effects of heat treatment on several poplar wood properties. Heat treatment (HT) in facts is a well-known process applied with very limited environmental impact (no preservatives are implied) able to strongly increase the durability of wood (Navi *et al.*, 2012). The treatment have non-negligible effects on other properties of wood such as colour, strength, stiffness, density, shrinkage and swelling. In particular, if compared to untreated wood, colour gets darker, strength is reduced, stiffness is increased for weak treatments and decreased for moderate and intensive treatments, density is reduced as well as shrinkage and swelling. The increased durability and dimensional stability resulting from the heat treatment can contribute to give new market to fast growing species such as poplar wood.

### 2. Material and method

In this work, the effect of HT in saturated moisture conditions is studied and the treatments was performed at different temperatures (100, 120, 140, 150 °C) for different treatment periods (1, 2, 3, 6 12 hours). The pressures applied during the treatments are those resulting from the saturated steam conditions that corresponds to: ~ 1 bar for 100°C, ~ 2 bar for 120°C, ~ 3.6 bar for 140°C, ~ 3.8 for 150°C. The complete experimental matrix is reported in Table 1.

The properties variations were determined as the difference between the treated and untreated state (20°C and 65% RH) and referred to the untreated state. The properties variations were studied on: colour, mass, MOE and moisture content. All these features, except than for moisture content, were measured as a variation on the same specimen. The measurement of the colour was performed according to the CIE L\*a\*b\* 1976 definitions and by a Konica-Minolta mod. CM700d colorimeter and the  $\Delta E_{ab}$  was determined according to Equation [1].

[1]

MOE was measured by a longitudinal tensile test on small samples (8x3x150 mm) up to a 30% of ultimate load. Qualitative chemical analyses of the samples was performed by FTIR after extraction in Soxhlet as from the method defined by (TAPPI, 2007).

Three different types (A, B, C - see Figure 1) of samples of poplar wood (*Populus x euroamericana* Guinier) were prepared from the same board after a first cut by cleavage. A - 20x3x130 mm - used for the determination of mass, density, colour, chemical composition (measured as paired data); B - 8x3x150 mm - used for the determination of MOE variations (measured as paired data); C - 8x3x150 mm - test for reference. Three samples per treatment were used to assess physical and mechanical properties with a total number of 30 samples, 9 color readings per treatment were used to assess the colour with a total number of 90 measurements.

### 3. Results

The mass loss was verified to be clearly dependent on the treatment intensity intended as the coupled effect of temperature and time as shown in Figure 2.

The reaction kinetics in saturated steam has shown to be very fast if compared to treatments performed in conventional oven such as in (Goli *et al.*, 2014). A mass loss of 8% (at 20°C and 65% RH equilibrium state) was attained after 3 hours of HT at 150°C in saturated steam while at 180°C in a conventional oven are necessary about 20 hours. The volume of the specimens have shown a non conventional behaviour if compared to thermal degradation performed in a oven. In fact, after the treatment was completed and the hygroscopic equilibrium was reached at standard environmental conditions (20°C and 65% RH), for the whole sample set the volume resulted increased from 3 to 5 % if compared to untreated state (Fig. 3). This is surprising because to a mass reduction should correspond a volume reduction. The increased volume could be explained by the difference between the internal pressure of the sample and the exterior pressure once the pressure is released from the autoclave.

This could produce a behaviour similar to the one described for the steam explosion of wood (Brownell *et al.*, 1986). The colour variation shows a linear trend with the mass loss highlighting how the treatment intensity (intended as the combined effect of treatment temperature and time) have a similar evolution on both colour and mass loss (as shown in Fig. 4). MOE has shown a tendency to increase up to 10% until a mass loss of 4% was attained. For larger mass losses MOE tends to reduce again getting to the same value of untreated samples for a 5% of mass loss. For higher

mass losses MOE gets again to lower values if compared to untreated samples (Fig. 5). From a chemical point of view the FTIR spectra show that a significant loss in hemicelluloses can be observed after each treatment while for the other wood components the modifications are very small. Figure 6 shows how from the reference sample (Fig. 6a) and the treatments performed at 140°C for 6 hours (Fig. 6b) and 150°C for 1 hour (Fig. 6c) a remarkable reduction of the glucoxy-lans at a wavelength of 1734.85 cm<sup>-1</sup> can be observed while the other wavelengths remain almost unchanged. Increasing the mass losses - 1.2% of mass loss for 6 hours of treatment at 100°C and 4.6% of mass loss for 3 hours of treatment at 140°C - results in a reduction of the hemicelluloses peak.

### 4. Conclusions

Heat treatment is an opportunity to give new market to fast growing species and low quality wood increasing drastically durability, hydrophobicity and dimensional stability. Saturated steam treatment of poplar wood have shown to be very effective in terms of treatment time and in terms of hemicelluloses degradation.

Mass loss, colour and MOE have shown to be well correlated the one the other and a larger experimental base could lead to use one of these value as a predictor of the others. At a first sight time-temperature equivalency could be profitably used to model the treatment kinetic. A well calibrated treatment could lead to an increase of stiffness of the treated material. The degradation can be mainly ascribed to hemicelluloses and in particular to glucoxy-lans. The treated material presents large density reductions because of the simultaneous effect of mass losses and of the increased volume making this material very suitable for light applications with high durability demand.

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Table 1. Complete experimental matrix.

| Treatment      | 1   | 2   | 3    | 4    | 5    | 6    | 7    | 8    | 9    | 10   |
|----------------|-----|-----|------|------|------|------|------|------|------|------|
| Temp. [°C]     | 100 | 100 | 120  | 120  | 120  | 140  | 140  | 140  | 150  | 150  |
| Time [h]       | 3   | 6   | 3    | 6    | 12   | 2    | 3    | 6    | 1    | 3    |
| Pressure [bar] | 1   | 1   | 1,99 | 1,99 | 1,99 | 3,61 | 3,61 | 3,61 | 4,76 | 4,76 |

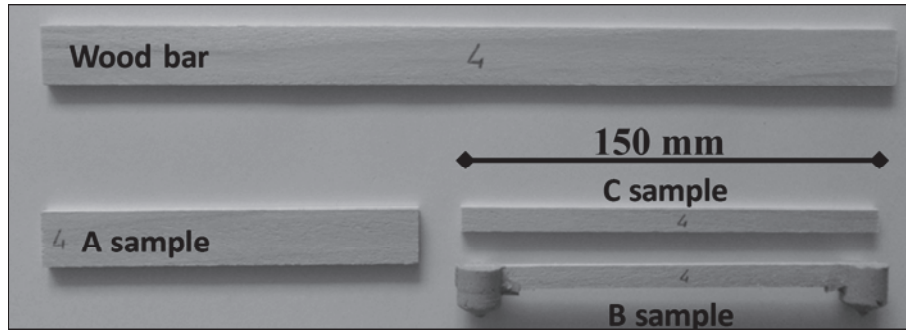


Figure 1. The samples used in the tests.

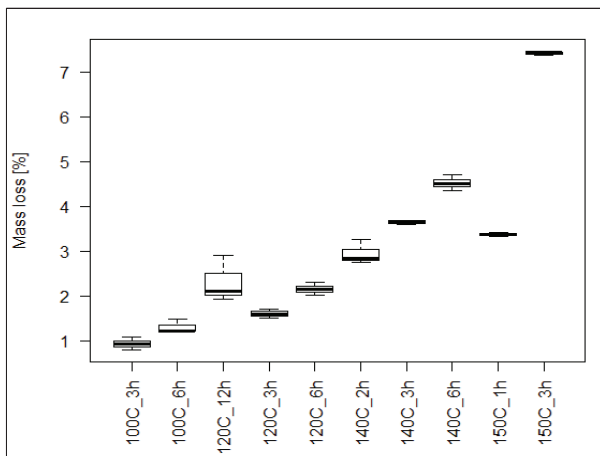


Figure 2. Mass loss at standard environmental conditions (20°C and 65% RH) versus treatment temperature and time.

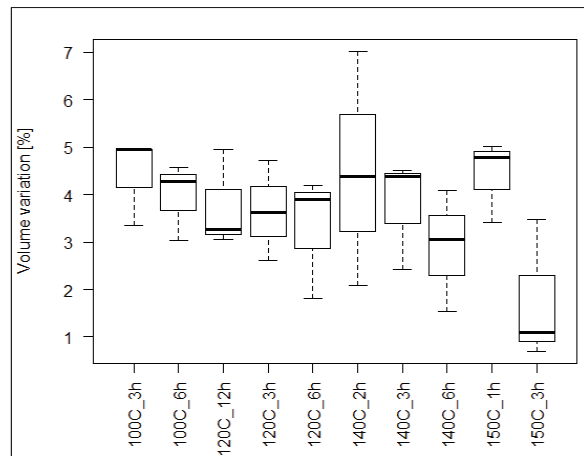


Figure 3. Volume variation vs type of treatment at 20°C and 65% RH compared to untreated state.

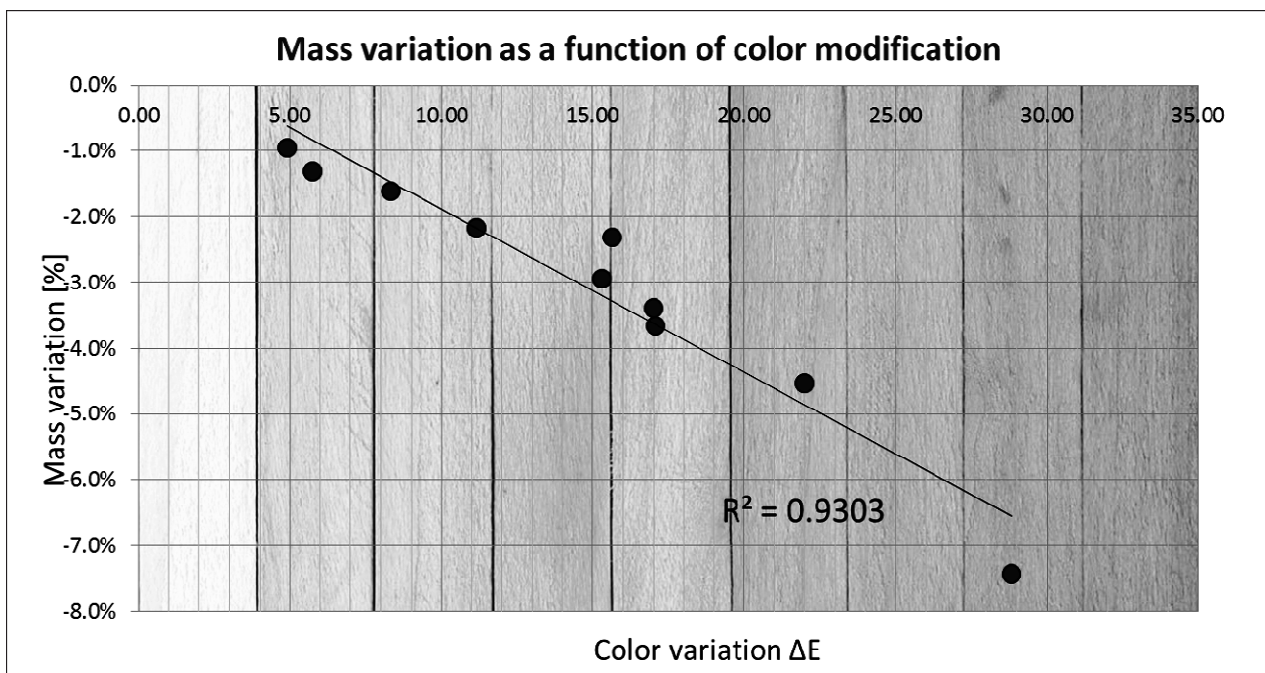


Figure 4. Colour variation vs. mass variation. The trend shows a linear behaviour.

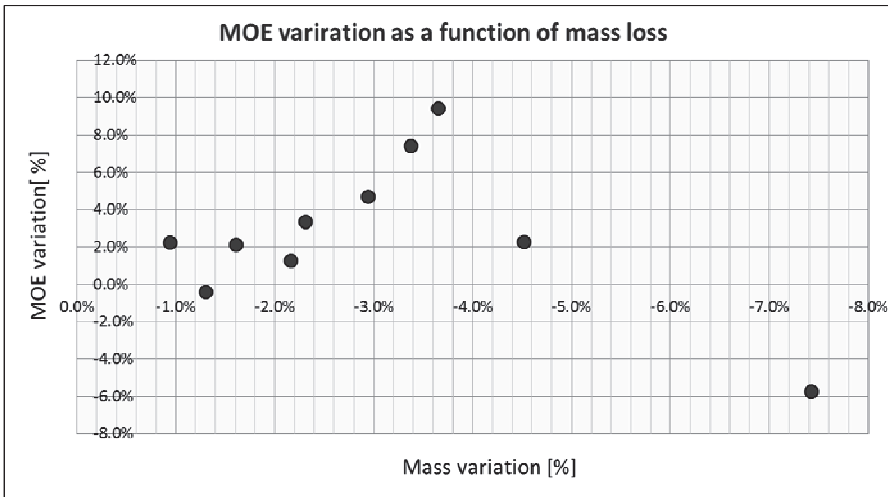


Figure 5. MOE vs. mass loss at 20°C and 65% RH.

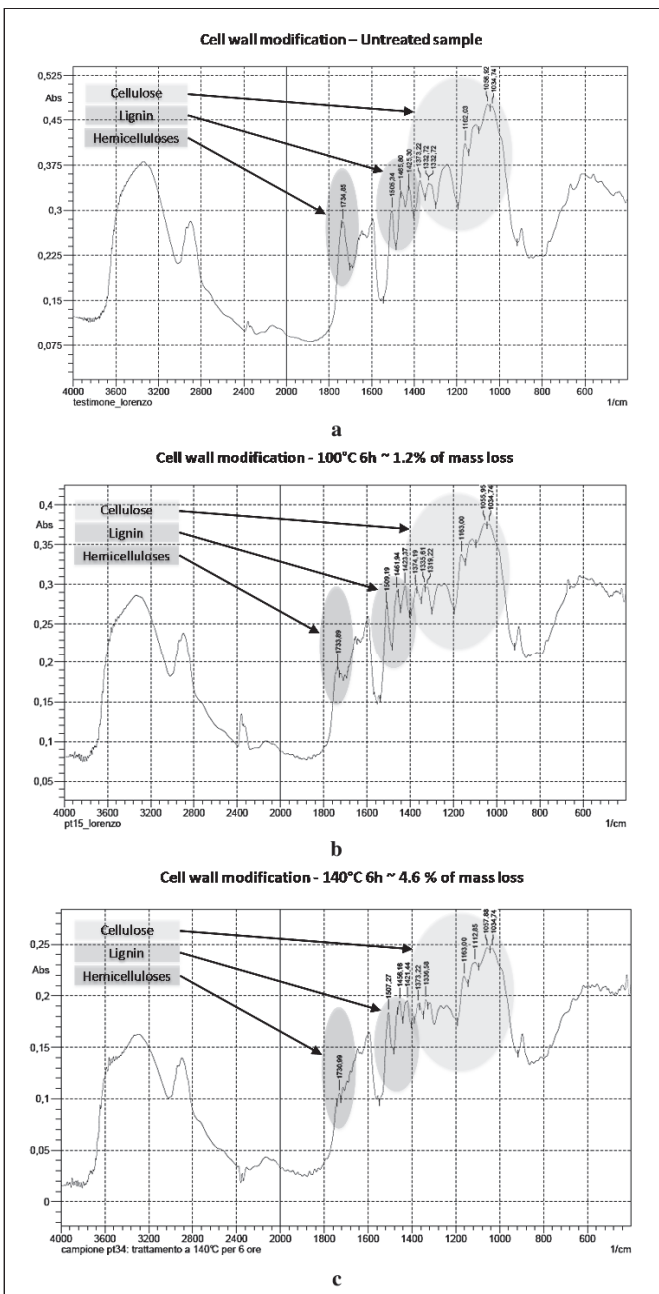


Figure 6. FTIR spectra of an untreated sample (a), of a sample treated at 100°C for 6 hours (b) and at 140°C for 6 hours (c). A remarkable degradation of the hemicelluloses can be observed as an effect of the heat treatment. The other wood constituents are not very affected by the heat treatments performed.



## RIASSUNTO

### Valorizzazione di specie a rapido accrescimento tramite trattamento termico

Il legno di pioppo è un materiale ampiamente utilizzato in Italia sia come legno massiccio che come materia prima per la produzione di derivati come pannelli compensati o pannelli di particelle. Secondo studi recenti [Brega 2011], il fabbisogno odierno di legname di pioppo in Italia si aggira attorno ai  $3.9 \times 10^6$  m<sup>3</sup> di cui circa la metà è prodotto in Italia. Il pioppo nero (*Populus nigra* L.) ed in particolare il suo clone euroamericano I-214 è una specie a rapido accrescimento molto apprezzata per gli impieghi dove la leggerezza è un requisito essenziale. Un limite importante del legno di pioppo, così come di molte specie a rapido accrescimento, è la bassa durabilità naturale. Nel tentativo di aprire nuovi mercati a questo legname si è messo a punto un programma di ricerca mirante a verificare l'effetto dei trattamenti termici su alcune proprietà del legno di pioppo. Il trattamento termico (TT) in effetti è un processo che notoriamente è in grado di conferire elevata durabilità naturale a specie di per se non durabili [Navi and Sandberg, 2012] con un limitato impatto ambientale visto che non vengono utilizzati preservanti di origine chimica. In questo lavoro si è verificato l'effetto del TT applicato in condizioni di vapore saturo a differenti temperature (100, 120, 140, 150 °C) e per differenti tempi (1, 2, 3, 6, 12 hours). Alcuni provini di legno di pioppo (*Populus x euroamericana* Guinier) sono stati preparati da una stessa tavola dopo una prima rottura eseguita per spacco al fine di garantire una precisa orientazione della fibratura. La variazione delle proprietà del legno è stata determinata come variazione tra lo stato precedente e successivo al trattamento dopo l'equilibramento a 20°C e 65% di umidità relativa ed espressa in funzione del valore iniziale. Le proprietà studiate sono: colore, massa, MOE e contenuto di umidità. Tutte le proprietà, ad eccezione del contenuto di umidità, sono misurate come dati appaiati. La misura del colore è stata eseguita secondo lo standard CIE L\*a\*b\* 1976 con un colorimetro Konica-Minolta mod. CM700d. Il MOE è stato misurato con un test di trazione longitudinale su campioni piccoli (8x3x150 mm) caricati al 30% del loro valore di rottura presunto. Le analisi chimiche qualitative sono state effettuate con tecnologia FTIR dopo un'estrazione con Soxhlet utilizzando il metodo descritto in (TAPPI, 2007). Le perdite di massa sono risultate chiaramente dipendenti dall'intensità del trattamento e la cinetica della reazione in ambiente saturo di vapore è

risultata molto più rapida di quella in stufa convenzionale a ventilazione forzata (Goli *et al.*, 2014). Una perdita di massa dell'8% (ad umidità normale) è stata ottenuta dopo 3 ore di TT a 150°C in vapore saturo mentre in forno convenzionale e 180°C sono state necessarie 20 ore per avere la stessa perdita di massa. La variazione dell'umidità di equilibrio a condizioni normali risulta avere lo stesso trend delle perdite di massa. Il volume, contrariamente alle aspettative, ha mostrato una tendenza a crescere nonostante le umidità di equilibrio in ambiente normale siano inferiori. Le variazioni di colore hanno presentato trend simili a quelli delle perdite di massa. Il MOE ha mostrato una tendenza ad aumentare fino all'10% per perdite di massa fino al 4%. Per perdite di massa maggiori il MOE tende poi a diminuire nuovamente riportandosi al valore dei provini non trattati attorno a perdite di massa del 5% ed inferiori per perdite di massa superiori. Da un punto di vista chimico i risultati hanno mostrato una riduzione significativa di glucoxilani inizia già dopo 6 ore di trattamento a 100°C. I glucoxilani inoltre scompaiono completamente dopo un trattamento di 12 ore a 120°C, 6 ore a 140°C e 1 ora a 150°C. Gli altri costituenti del legno non sembrano subire grandi conseguenze dai trattamenti termici applicati.

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