Physical and mechanical characteristics of poor-quality wood after heat treatment

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Poor-quality Corsican pine (Pinus nigra subsp. laricio (Poir.) Maire) and Douglas fir (Pseudotsuga menziesii (Mirb.) Franco) wood samples were heat treated with the aim of testing the improvement of wood quality that could increase their economic value. Wood properties were measured to assess quality in treated and non-treated materials, including density, hardness, anti-swelling efficiency (ASE), modulus of elasticity (MOE), modulus of rupture (MOR), and compression strength. The results showed higher dimensional stability in heat-treated wood, yet mechanical performance in compression and bending strength were only marginally affected by loss of density. Despite having a relatively low density after heat treatment, Corsican pine sapwood has potential in manufacturing higher-value products. In contrast, heat treatment applied to Douglas fir wood did not appear economically viable; insufficient differences were detected between the naturally desirable characteristics of this species and heat-treated samples.

Keywords: Thermowood, Density, Compressive Strength, Bending Strength, Sapwood, Wood Quality, Douglas Fir, Corsican Pine

Introduction

High-temperature heat treatment is considered a suitable method to improve wood characteristics. It reduces toxic chemical applications normally required to increase wood durability (Kandem et al. 2002, Welzbacher & Rapper 2007; Mohareb et al. 2010) and enhance dimensional stability, reducing the hygroscopic behavior of the material (Kortelainen et al. 2006). High-temperature treatment can be viewed as a type of wood modification, due to the possibility of controlling color changes (Bekhta & Niemz 2003, Johanson & Moren 2006, Esteves et al. 2007a, 2008) and mechanical performance (Bekhta & Niemz 2003, Welzbacher et al. 2007, Bal 2014). The process results in a new material in terms of durability, wood-water relationships and some mechanical properties, which exhibits performance dependent on the specific high-temperature treatment conditions (Militz 2002, 2008, Esteves & Pereira 2008), but the wood product does not present any environmental hazards in its disposal (Hillis 2006). The final product typically has the disadvantage of reduced mass and mechanical performance, although some difference in degree of change has been observed between softwoods and hardwoods depending on the treatment method (Kocaefe et al. 2010, Li et al. 2011).

Therefore, the overall extent of modification and the final result of the process is quite variable. In this study, we assessed heat treatment effects on Corsican pine and Douglas fir wood properties obtained from a conifer reforestation site in Italy, with particular consideration of different sapwood and heartwood behavior. This timber is considered poor quality and is currently used as biomass for energy or packaging.

Materials and methods

Study area

Forty Douglas fir (Pseudotsuga menziesii) and 40 Corsican Pine (Pinus nigra ssp. laricio) trees were sampled in an artificial mixed stand in Poggio Nibbio near Viterbo, Central Italy (latitude 42° 22′ N, longitude 12° 10′ W). Trees were arranged in rows with a cross-distance of 4 m; the stand was thinned to promote tree regeneration and introduction of indigenous hardwoods. The harvested wood is generally of poor quality due to the fact that the trees have grown quite fast, and not straight, producing large rings that contain a high proportion of earlywood. The sapwood is also quite large, particularly in Corsican pine.

From each of the eighty trees sampled, single boards 6 cm thick, 80 cm long, and 7 cm in width were produced and subsequently crosscut to obtain two half-boards of approximately 40 cm in length. From these two half boards, we obtained twin specimens measuring 2 × 2 cm in the transverse radial and tangential directions, and 40 cm longitudinally. The wood samples were largely juvenile, approximately 10 years in cambial age, and evidence of reaction wood was not observed in any samples analyzed. For each of the 2 × 2 × 40 cm sample pairs, one sample was heat treated, and the second sample was left untreated. Subsequently, two blocks measuring 2 × 2 × 3 cm in size were cut from the end of each sample to measure post-treatment/treated (t) and control sample/not treated (nt) physical properties and compression strength; the total sample size was 160 specimens. Only sapwood samples were used in Corsican pine (CP), as the heartwood was limited to a very narrow inner portion of the sample section. However, the heartwood occupied a much larger surface area and increased number of rings in Douglas fir; therefore it was possible to distinguish and discuss the results for sapwood (D-S) and heartwood (D-H) samples separately.

Heat treatment

Thermowood treatment was performed at the Margaritelli farm in France (http://www.margaritelli.com/france) according to the following steps:
• Preliminary heating stage at 110 °C.
• Drying phase. The wood was fully dried to prepare it for high-temperature treatment by a gradual warming over 14 h until a temperature of 140 °C was reached. An atmosphere of superheated steam, with low oxygen content, prevented the formation of cracks and alterations within the wood. At the end of this phase, wood moisture content was reduced to little more than 0%.
• High-temperature treatment phase. Application of 210 °C for 2 h.
• Cooling phase. Following the heat treatment, the cooling phase slowly reduced the wood temperature to 80-90 °C over the course of 15-20 h, allowing for moisture

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adsorption. The wood typically measured equilibrium moisture content between 3-4% at the end of this treatment.

Physical parameters
Untreated samples in fresh state were conditioned in the laboratory at 20 °C and 65% relative humidity (RH), reaching an equilibrium moisture content (EMC) of 12%. Half of the samples were then subjected to heat-treatment; after the treatment the heat-treated samples showed a moisture content between 8 and 11%. Density of samples was measured and their values were subsequently adjusted in treated samples to 12% (ρt) using the UNI-ISO-1361 formula for moisture content correction (eqn. 1):

\[ \rho_t = \rho_o \left( \frac{[W - 12]}{100} \right) \]

where \( W \) is the wood moisture measured as \( W = (m_w - m_d)/m_o \), \( m_w \) is the fresh weight, \( m_d \) is the dry weight (0% moisture content), and \( K \) is the coefficient of volumetric shrinkage, assumed to be \( K = 0.85 \). We adopted the above procedure to compare all the physical and mechanical properties of treated samples with the untreated ones at the same moisture content.

Following EMC measurements, all samples (both treated and untreated) were soaked in water until a constant weight was reached, which was considered the maximum water content, and sample linear size and volume were measured again. The samples were subsequently placed in a 103 °C oven until achieving a constant weight and dry state.

Size and weight of the samples were measured again, and moisture content of the fully soaked samples (both treated and untreated) was determined by a gravimetric system. The following parameters were examined by measuring the linear size of samples at oven-dry moisture content: total shrinkage (\( \beta_t \)), radial shrinkage (\( \beta_r \)), coefficient of anisotropy (\( P \)), which is the ratio between tangential and radial shrinkage; and basic density (\( \rho_b \)), which is the ratio between dry weight and maximum volume (over the fiber saturation point).

Dimensional change in specimens was evaluated using two methods: maximum shrinkage determination and swelling coefficient or anti-swelling efficiency (\( ASE \)). \( ASE \) is an approach commonly employed to evaluate dimensional stabilization processes (Militz 2002, Militz 2008), however differences in the application to dry and soaked specimens must be considered. Dimensional changes of oven-dried samples compared with maximum water content at room temperature results in the most notable effects.

We therefore applied the protocol of Santos (2000), and \( ASE \) was calculated by the formula (eqn. 2):

\[ ASE = \frac{100(\Psi_t - \Psi_o)}{\Psi_t} \]

where \( \Psi_t \) is the linear or volumetric swelling coefficient of untreated wood, and \( S \) is the linear or volumetric swelling coefficient of treated wood.

\begin{figure}
\centering
\includegraphics[width=\textwidth]{image}
\caption{Physical characteristics of treated (t) and untreated (nt) Corsican pine (CP), Douglas fir heartwood (D-H), and Douglas fir sapwood (D-S). (a) Tangential shrinkage (\( \beta_t \)) and radial shrinkage (\( \beta_r \)); (b) anisotropy coefficient; (c) density; and (d) basic density. (*) (**) (***) levels of significance between treated (t) and untreated (nt) specimens at \( P < 0.05 \), \( P < 0.01 \), and \( P < 0.001 \), respectively using the Bonferroni test. Vertical bars represent standard error.}
\end{figure}

Mechanical properties
Three samples of each species, taken from 40 specimens of each taxon (totaling 240 samples, 120 Corsican pine and 120 Douglas fir), were tested to measure main mechanical properties, and differences between treated and untreated wood.

All test samples were prepared by cutting pieces from the remaining wood strips; and all mechanical tests were performed using a Zwick-Roell universal testing machine. Three mechanical tests (Romagnoli & Spina 2013) were performed to evaluate the following:

- Compression strength in the longitudinal direction, using specimens 2 × 2 × 3 cm in accordance with UNI-ISO-3787 (1985).
- Modulus of elasticity (MOE), using a three-point bending test with a preload of 50 N and a machine head speed of 8 mm/min. The distance between the support points was 28 cm, and the load was applied in a tangential direction at a point 14 cm from the supports.
- Modulus of rupture (MOR) during bending of 2 × 2 × 32 cm specimens, as per UNI-ISO-13061.3.
- Hardness (Brinell hardness, HB) according to UNI-EN-1534 (2011), which was carried out using a preload of 50 N and a test speed of 15 mm/min. All samples were tested tangentially and measured 30 × 20 × 20 mm in size.

The treated sample moisture content (~8%) was below the reference condition, and thus the measured value was adjusted to 12% moisture by adopting the formula provided by the UNI-ISO-3787 (1985) standards (eqn. 3):

\[ P_{\text{max}} = 1 + 0.02(MC_i - 12) \]

where \( P_{\text{max}} \) is the maximum load and \( MC_i \) is the moisture content of treated specimen.

Statistical analysis
Pairwise differences in physical and mechanical properties of wood samples were tested by two-sample Kolmogorov-Smirnov test (a = 0.05). Pearson’s correlation coefficients were calculated to assess the relationships between density and physical and mechanical properties of treated and non-treated wood. The relationships between density and other wood properties were further investigated by linear regression analysis.

Results and discussion
The non-treated wood of the Corsican pine and Douglas fir obtained from the Poggio Nibbio site had specific densities (Fig. 1c) that fall within the lower threshold for the two species (467 kg m⁻³ for Douglas fir and 517 kg m⁻³ for Corsican pine - Nardi Berti 2006), and are even low compared with
other sites in Italy (Fioravanti 1999, Todaro & Macchioni 2011).

Results showed the mechanical properties of non-treated Corsican pine wood (45 MPA for compression strength - Fig. 2a-d, Tab. 1) were comparable to previously published values (Nardi Betti 2006, Fioravanti 1999, Laicenbruch et al. 2010), except for MOE which was lower. Douglas fir sapwood showed lower density values compared to heartwood (Fig. 1c), but the mechanical properties revealed little difference between the two wood portions (Fig. 2a-d, Tab. 1). However, sapwood shrinkage (Fig. 1a) was higher than heartwood, which was similarly reported by Metsä-Kortelainen (2011).

The most important heat treatment outcome was an increased dimensional stability. In addition, a significant (P < 0.001) decrease in shrinkage values (approximately 42% in Douglas fir and 40% in Corsican pine) were observed, as compared with non-treated wood (Fig. 1a).

Wood modification significantly (P < 0.05) reduced the anisotropy coefficient in Corsican pine (<14% - Fig. 1b). Results showed a 12% change in Douglas fir heartwood, though not statistically significant. Militz (2002) reported similar results for stone pine.

Heat treatment effects were reflected by an ASE value of 44% (Fig. 3) for Douglas fir and Corsican Pine, which exceeded values reported for hardwoods, including eucalyptus (24% - Santos 2000) and silver oak (<40% - Srinivas & Pandey 2012), and some tropical species (9.2-14.8% - Korkut 2012). These values were considerably lower compared to Chinese fir (Cunninghamia lanceolata; >60% - Cao et al. 2012). Moreover, the ASE value in Douglas fir was comparable to the volume swelling obtained by Li et al. (2011).

Results showed increased ASE values in the tangential direction for Corsican pine and Douglas fir heartwood samples, consistent with P. banksiana (Poncsak et al. 2011), P. sylvestris and P. radiata (Militz 2002). However, despite substantial stability improvement in the tangential direction, the anisotropy coefficient (Fig. 1b) remained quite high in heat-treated specimens, congruent with results observed by Esteves et al. (2007a, 2007b, 2008). This minor effect of the treatment in the radial direction might be due to the higher lignin content, which is less sensitive to heat (González-Peña et al. 2009).

Heat treatment did not significantly affect specific density in Douglas fir and Corsican pine. A marked reduction in specific density of heat-modified wood was previously detected in hardwoods (Welzbacher & Rapper 2007, Korkut 2012) and conifers, including P. elliottii (Severo et al. 2012), P. sylvestris and Picea abies (Boonstra et al. 2007). A lower reduction in treated wood specific density, comparable to our experimental results, was observed in P. sylvestris (Zaman et al. 2000).

Following heat treatments, we observed a general decrease in mechanical performance in Douglas fir sapwood and heartwood. This result is consistent with most conifers treated at approximately 200 °C (Bengtsson et al. 2002, Esteves & Pereira 2008, Bakar et al. 2013) or higher (280 °C - Stamm et al. 1946, Esteves & Pereira 2008). We also observed a reduction in Brinell hardness previously detected in spruce, beech, and red oak (Bakar et al. 2013) treated at 190 °C, and P. brutia treated at temperatures exceeding 180 °C.
Fig. 4 - Load-deformation graph for Corsican pine showing the relationship between MOR (a, c) and axial compression strength (b, d) in treated (t) and untreated specimens (nt).

Fig. 5 - Load-deformation graph for Douglas fir sapwood showing the relationship between MOR (a, c) and axial compression strength (b, d) in treated (t) and untreated specimens (nt).
(Ates et al. 2009). As for Corsican pine, a behavior similar to Douglas fir heartwood was observed, though differences between treated and untreated samples were less evident. The Douglas fir examined in this study showed differences in hardness, an 8% reduction in sapwood, and a 28% significant decrease (P < 0.05) in heartwood.

MOR value results showed a decrease of approximately 28% following heat treatment for both species, suggesting MOR was the most sensitive property to heat treatment. Several studies similarly reported decreased MOR in softwood and hardwood species (Reppelin & Guyonnet 2003, Leijten 2004, Esteves & Pereira 2008, Ates et al. 2009, Srinivas & Pandey 2012). Bal (2014) demonstrated the modification in bending strength (MOR) was due to mass loss following heat treatment, and although it was not possible to measure the mass loss in this experiment, some general comments can be made based on the knowledge that MOR typically exhibits a positive relationship to wood density (Zobel & Van Buijtenen 1989, Nocetti et al. 2012, Romagnoli et al. 2014). In the present study, the density reduction following heat treatment was not as high as expected, particularly when compared to the observed change in strength. However, the ratio between the mechanical properties and untreated wood density would be expected to change following heat treatment due to chemical degradation of woody components, such as hemicelluloses and cellulose, which play a major role in determining wood properties in relationship to density (Esteves & Pereira 2008). Furthermore, Corsican pine sapwood (516.8 kg m⁻³) had a higher density than Douglas fir (457.3 kg m⁻³), therefore a major decrease in Corsican pine sapwood mechanical properties after heat treatment should have been observed due to higher thermal conductivity of denser wood (Bal 2013, 2014). The differences detected might be explained by the volatile resin amounts in the two species’ sapwood.

Lower mechanical performance in compression strength is usually found in heat treated hardwoods and softwoods (Ates et al. 2009, Korkut 2012). In our study, we observed a significant reduction (P < 0.05) in axial compression strength in Douglas fir after heat treatment in both heartwood and sapwood. Corsican pine however, showed significantly higher axial compression strength (4%, P < 0.05) in the treated samples. This is reflected in the Corsican pine bending MOE values, which were also higher in treated samples (12%) and were consistent with other studies (Santos 2000, Boonstra et al. 2007, Bal 2014).

Results showed the reduction in mechanical properties due to heat treatment was in some cases lower than that observed in Pinus banksiana (Kocafe et al. 2010) and in P. nigra (Gunduz et al. 2008, Bal 2014). In our study, the bending MOE was not notably affected by heat treatment in Corsican pine or Douglas fir, particularly when compared with results reported in hardwoods, including aspen (Tremblay & Barbeaut 2009) and birch (Shi et al. 2007).

Special attention must be given to the load-deflection curves (Fig. 4, Fig. 5) when assessing MOR and axial compression strength. Heat-treated Corsican pine exhibited abrupt failures in MOR values when it reached a deformation of approximately 4 mm, and a considerably larger deflection at maximum load for untreated samples. The curve shape typically reflects the behavior of heat-treated wood, which was reported in other wood species (Arnold 2010) and ascribed to transverse fracture surfaces of tension-stressed fibers, or transverse ruptures in tracheid cell walls (Arnold 2010). Unsal & Ayrilmis (2005) showed compression strength was reduced by heat treatment, even under relatively low temperature conditions. Many Corsican pine control samples under compression exhibited a residual deformation up to 6 mm (Fig. 4b) after reaching maximum load, whereas the mechanical behavior demonstrated by the load-deformation graph differed in Douglas fir (Fig. 5b). Specifically, the MOR value following the maximum load shows a residual deformation lower than that observed in control (nt) samples (Fig. 5a, Fig. 5c), with the same result ob-

Fig. 6 - Linear regression between density and the measured physical properties in treated (t) and untreated (nt) specimens for Corsican pine (CP) and Douglas fir (D).

Fig. 7 - Linear regression between wood density and mechanical properties in treated (t) and untreated (nt) samples for Douglas fir. The Douglas fir sapwood and heartwood specimens were grouped together. (MOR): bending strength; (MOE): bending modulus of elasticity.
served in compression strength.

Sapwood and heartwood results before and after treatment requires special consideration. For example, heat treatment appeared to mitigate the differences between the two tissues (heartwood and sapwood) compared to the physical and mechanical properties of non-treated wood material, with the exception of the compression strength (Cao et al. 2012). Heat treatment also affected heartwood specific density and volumetric shrinkage; however, bending MOE, compression strength, and hardness was primarily impacted in sapwood. It was notable that bending strength and MOE decrease in sapwood was observed, but a significant reduction was not detected. This result indicated it is possible to obtain an improvement in dimensional stability in Douglas fir sapwood, with a far less substantial loss of mechanical performance than would otherwise be expected.

Few studies have evaluated the different behaviors of sapwood and heartwood after thermowood treatment. A recent paper examined EMC in Turkey oak (Todaro et al. 2012), however the outcome of this and other studies were not congruent with our results. In fact, Burgos & Rolleri (2012) observed increased shrinkage in heat-treated Douglas fir wood, with decreased extractives, although the result might be comparable, as Douglas fir sapwood contains fewer extractives than heartwood. A separate examination of radial and tangential directions in the present study indicated ASE was very similar to shrinkage values, with the exception of heartwood vs. sapwood radial shrinkage. Sapwood substances might be chemically modified into new polyphenols, which could affect the expected physical and mechanical behavior of sapwood (Alen et al. 2002). A linear regression analysis was performed between density and shrinkage for Corsican pine and Douglas fir, and results showed that both species exhibited very low R² values (Fig. 6, Fig. 7). It can be hypothesized that the absence of a relationship between the variables could be due to juvenile wood in the conifer tissue, which weakened the mechanical properties as compared with mature wood. Increased shrinkage can be explained by orientation of the microfibril angle; and decreased wood density did not make as great a contribution (Zobel & Sprague 1998, Larson et al. 2001, Bal 2014). Following treatment, density did not exhibit an increased relationship to physical properties (Fig. 6), and its relationship to mechanical properties was reduced (Fig. 7). Increased understanding can be inferred from the correlation coefficient between density and the physical and mechanical properties (Tab. 2), where the correlation was often significant in Corsican pine. However, Douglas fir showed an absence of significant results, with the exception of sapwood density-compression strength and density-anisotropy coefficients. In addition, a remarkable result showed the correlation coefficient between density, MOR, and radial shrinkage was increased by heat treatment.

The most marked result, however, was the change in the correlation trend between density and radial shrinkage after heat treatment. The hornification process might serve to explain this result, which in spruce promoted irreversible hydrogen bonding between wood elements during drying that accompanied heat treatment (Borrega & Kärenlampi 2010a). Hornification is a process term often applied to wood pulp when polymeric structures in lignocellulosic materials stiffen during drying (Fernandes Diniz et al. 2004). Hornification in heat-treated wood results in a mass reduction without decreasing mechanical properties, which could promote silvicultural options should be explored. Treatment had a decreased effect on wood density and subsequent physical and mechanical properties, which might promote silviculture with high growth rates. In fact, even with a wider ring width and increased amount of earlywood, and therefore lower density, the reduction does not noticeably compromise the physical and mechanical characteristics of the treated wood. Douglas fir results indicated heat treatment was not less beneficial in terms of mechanical performance. Furthermore, this species has naturally good wood durability, and the increased heartwood presence was reported to be associated with high growth rates. These

**Table 2 - Pearson’s correlation analysis between density and physical-mechanical properties.** (MOR): bending strength; (ρc): axial compressive strength; (HB): Brinell hardness; (MOE): bending modulus of elasticity; (βr): volumetric shrinkage; (βt): radial shrinkage; (βp): tangential shrinkage; (P): coefficient of anisotropy; (CP): Corsican pine; (D-S): Douglas fir sapwood; (D-H): Douglas fir heartwood, (nt): untreated; (t): treated. (*) (**) (***) levels of significance between treated (t) and untreated (nt) specimens at P < 0.05, P < 0.01, and P < 0.001, respectively using the Bonferroni’s test.

| Parameter | MOR (MPa) | σ12 (MPa) | HB (MPa) | MOE (MPa) | βr max (%) | βt max (%) | βp max (%) | ψ |
|-----------|-----------|-----------|----------|-----------|------------|------------|------------|---|
| CP nt (n=40) | 0.385 | 0.767*** | 0.135 | 0.468* | 0.610*** | 0.554*** | 0.487* | -0.381 |
| CP t (n=40) | 0.636*** | 0.695*** | 0.127 | 0.358*** | 0.630*** | 0.726*** | 0.441 | -0.392 |
| D-S nt (n=12) | 0.231 | 0.643*** | 0.353 | 0.416 | -0.143 | 0.400 | -0.540 | -0.885*** |
| D-S t (n=12) | 0.274 | 0.603** | 0.264 | 0.460 | -0.347 | 0.358 | -0.649 | -0.681 |
| D-H nt (n=28) | 0.152 | 0.418** | 0.209 | 0.228 | 0.190 | 0.331 | 0.032 | -0.344 |
| D-H t (n=28) | 0.245 | 0.284 | 0.365 | 0.150 | 0.236 | 0.336 | 0.143 | -0.390 |

**Conclusion**

Corsican pine heat treatment significantly reduced the anisotropy coefficient and shrinkage, whereas in Douglas fir sapwood specific density was most impacted by heat treatment. Overall, _P. nigra_ subs. _laricio_ results suggested this material has more potential as a valuable wood product than biomass for energy or to manufacture pellets, which indicates silvicultural options should be explored. Treatment had a decreased effect on wood density and subsequent physical and mechanical properties, which could promote silviculture with high growth rates. In fact, even with a wider ring width and increased amount of earlywood, and therefore lower density, the reduction does not noticeably compromise the physical and mechanical characteristics of the treated wood. Douglas fir results indicated heat treatment was not less beneficial in terms of mechanical performance. Furthermore, this species has naturally good wood durability, and the increased heartwood presence was reported to be associated with high growth rates. These
characteristics therefore discourage further investment into P. menziesii heat processing, as treatment is only required if the need is evident for more dimensional stabilization in specific wood products.

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