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DENSIFICATION AND HEAT TREATMENT OF MARITIME PINE WOOD

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ABSTRACT

Pine (*Pinus pinaster* Ait.) wood samples were subjected to a combined treatment by densification and heat treatment. Samples were densified before and after heat treatment. The heat treatment was made inside an oven at 190ºC during 2 to 6 h and wood densification was made in a hot press at around 48 bar pressure and temperatures between 160ºC and 200ºC for 30 min. Compression-set, compression-set recovery after three cycles of water soaking followed by oven drying, density, hardness, bending strength and stiffness and durability against subterranean termites were determined after the treatment. Results show that densification increases density, hardness, bending strength, stiffness and durability against termites. Heat treatment applied after the densification is more effective in reducing compression-set recovery than if applied before.

KEYWORDS: Densification, heat treatment, *Pinus pinaster*, compression set-recovery, termite resistanceintroduction

Compressed wood exist for over a century with several patents emitted in the USA since 1900 while in Europe the first patent was emitted in Germany in 1923 (Kutnar et al. 2011). This process, called "Lignostone", used high temperatures (140°C) and densification was done by hot-press (Kollmann 1951). Another product of laminated compressed wood was marketed in Germany under the name "Lignifol" (Morsing and Hoffmeyer 2000). In 1937, in the USA (Seborg et al. 1945) a similar product which was created called "Staypack".

In accordance to Morsing and Hoffmeyer (2000) the densification process can be divided into four steps. The first being softening or plasticization of the cell wall followed by compression perpendicular to the grain in the softened state, setting by cooling and drying in the deformed state and spring-back or fixation of the deformed state. The spring-back when wood is re-moisture is one of the main problems of compressed wood. These authors proposed three mechanisms to avoid spring-back. Prevent the wood from being re-softened by changing the hygroscopicity of the cell, form covalent crosslinks between the wood components in the deformed state or release the elastic stresses and strains created during compression. Li et al. (2012) studied mechanically induced residual stresses in densified softwoods and simulated the stress-releasing process by means of a mathematical model. These authors stated that the mechanically induced residual stresses increased with increasing compression ratio and about 50% of maximum total residual stress in densified fir and pine could be released in the first several minutes after soaking in hot water. Several authors have been testing the reduction of hygroscopicity of the cell through heat treatment (Inoue et al. 1993, 2008), pre-steamed wood at temperatures from 120°C to 220 °C for 5—20 min before compression in radial direction and concluded that the set recovery decreased with increasing pre-steaming temperature and time. These authors also showed that it was possible to reduce springback by both dry and steam heating though dry heating required 20 hours at 180°C to avoid recovery and 5-20 min are enough if steam is used (Inoue et al. 1993, 2008). Ito et al (1998a; b) obtained similar results with Harigiri (*Kalopanax pictus* (Thunb.) Nakai), concluding that 200°C and 4 min or 180°C, 8 min of steaming are enough to prevent spring-back. These authors believe that the partial hydrolysation of the para-crystalline region of cellulose and subsequent steam rearrangements of hydrolysed constituents into crystalline regions are the responsible for keeping the transformed shape intact. In accordance to Morsing and Hoffmeyer 2000) it is possible to eliminate springback by four different methods: heat-treatment in a hot-press at 200°C for 1 hour by compressing water saturated wood; steam-treatment in autoclave at 190°C for 15 minutes (13 atm); heat-treatment in an oven at 190°C for 20 hours or hygrothermal treatment in a closed hot-press at 190°C for 10 minutes. Nevertheless Boonstra and Blomberg (2007) stated that there was a limited effect on the recovery of the dimensions when densified *Radiata pine* (*Pinus radiata* D. Don) was exposed to moisture. Similar results were presented by Welzbacher et al.(2008) who densified Norway spruce by the OHT-process and concluded that compression-set recovery of densified and oil-heat treated spruce was almost completely eliminated by an OHT at temperatures above 200°C. The permanent fixation of deformation is in accordance to Dwianto et al. (1999) due to the chain scission of hemicelluloses accompanied by a slight cleavage of lignin. At the same time heat treatment reduces spring-back it will also improve wood stability and durability as stated before (Esteves and Pereira 2009).

The objective of compressing wood is to increase strength and stiffness which is attributed to increased density. Kulticova (1999) compressed both mature and juvenile southern pine (*Pinus taeda* L.) and yellow-poplar (*Liriodendron tulipifera* L.) and concluded that the ultimate tensile stress and the modulus of elasticity after all densification treatments increased. O'Connor (2007) compressed 7-year old Eastern cotton wood (*Populusdeltoides* Bartram ex Marshall) and sweetgum (*Liquidambar styraciflua* L.) and stated that density, MOE, and MOR increased up to

178%, 254%, and 156% for cottonwood and 168%, 213%, and 182%for sweetgum, respectively. Both species exhibited increases in MOE and MOR which were approximately proportional to increases in wood density. However in accordance to Blomberg et al. (2005) mechanical strength is lower than it would be expected from the increased density, mainly for strength perpendicular to grain. This might be due to wood degradation upon compression. An increase in densification temperature can decrease strength properties as stated by Ulker et al. (2012), who explained the decrease by the increasing chemical degradation. In accordance to these authors the most suitable temperature is 120ºC in order to achieve a higher bending (42%), shear (20%), and compression strength (47%), and 140ºC for a higher radial (242%) and tangential hardness (268%) in densified Scots pine (*Pinus sylvestris* L.). An increase in the Brinell hardness after densification was also found (Pelit et al. 2015) for Scots pine and Eastern beech wood, nevertheless these authors stated that Brinell hardness reduced after the heat treatment. Different results were presented by Rautkari et al. (2013) who stated that the hydrothermal post-treatment did not reduce the Brinell hardness of control and surface densified specimens. In accordance to Blomberg (2006) hardness, bending and axial compression strength are the most improved mechanical properties. The compressibility of wood is increased with increasing pre-steaming temperature and time as stated by Inoue et al. (2008) who also mentioned that the set recovery of the pre-steamed wood decreased as pressing temperature and time increased.

Results presented before (Müller et al. 2003) show that the effect of compression depends on the characteristics of wood particularly at the transition from elastic to plastic deformation and along the stress plateau. These differences were explained as resulting from the differences in anatomy and cell wall microstructure. In accordance to these authors, radial compression of spruce (ductile plastic deformation) is limited by the buckling load of only a few cells closely behind the ring border while for oak (brittle failure) is determined by the buckling of the earlywood vessels and vasicentric tissue, and beech (elastomeric yielding) by the densification of the vessels at high plastic deformations. The results presented by Tu et al. (2014)revealed through microscopic observation that the deformations in densified wood resulted from the viscous buckling of cell walls without fracture and that the volume of the void areas in the specimens decreased uniformly.

One of the advantages of compressed wood might be the increased durability. Schwarze and Spycher (2005) studied colonisation and wood degradation by three brown-rot fungi, *Coniophora puteana* (*Schumach*.) P. Karst., *Gloeophyllum trabeum* (Pers.) Murrill and *Poria placenta* (Fr.) Cooke, in Norway spruce (*Picea abies* (L.) Karsten) and concluded that the weight loss induced by all three fungi was lowest in THM-densified wood post-treated at 180°C and that hyphal growth on THM-densified wood was sparse and confined to the cell lumina of earlywood tracheids. In controls and TH-treated wood hyphal growth was abundant. The main reason indicated for the lower hyphal growth was the occlusion of cell lumina. Similar results were presented before with poplar wood (Khademi Bami and Mohebby 2011).

However, Skyba et al. (2008 stated that THM-densified beech and Norway spruce wood are still highly susceptible to soft rot and consequently unsuitable for class 4, in ground contact or in fresh water and (Kutnar et al. 2011) determined that viscoelastic thermal compression of hybrid poplar did not change decay resistance to fungi like *Pleurotus ostreatus* (Jacq.) P. Kumm. and *Trametes versicolor* (L.) Lloyd. In relation to the durability against termites studies are scarce. Lower mass losses were reported for some variables of thermally pressed wood in no-choice feeding tests with Eastern subterranean termites (*Reticulitermes flavipes* Kollar) (Unsal et al. 2009) while Choowang (2013) tested the effect of thermally compressing oil palm against subterranean termites (*Coptotermes gestroi* Wasmann) in a 4-week no-choice test and concluded that the surface damage to the samples treated at 220°C showed improved resistance to subterranean termites based on visual observation.

The effect of thermo-hygro-mechanical (THM) densification temperature on the surface color, roughness, wettability, and chemical composition of trembling aspen (*Populus tremuloides* Michx.) and hybrid poplar (*Populus maximowiczii* A. Henry x *P. balsamifera* L.) veneers was investigated (Diouf et al. 2011). Veneer color darkened with increasing THM densification temperature. Surface roughness decreased between 160°C and 200°C. Wettability decreased after THM densification, but no significant difference was found between treated specimens. ATR-FTIR and XPS results confirmed that THM densification caused major chemical changes in veneer surfaces, and more pronounced at temperatures higher than 160°C. Ratkauri et al.(2010) stated that FT-IR indicated no significant chemical changes occurred during the densification process.

MATERIAL AND METHODS

Treatment

Pine wood (*Pinus pinaster* Ait.) from the Viseu region in Portugal was used in the tests. Sapwood samples with approximately 145 x 145 x 32 mm (longitudinal x tangential x radial) were cut from a central board and placed in a controlled environment at 20°C and 65% relative moisture content for two weeks. The samples were subjected to a combined treatment by densification and heat treatment. Samples with initial equilibrium moisture content around 12% were densified before and after heat treatment in accordance to (Tab. 1). The heat treatment was made inside an oven at 190°C during 2 to 6 h and wood densification was done in the radial direction and made in a hot press at around 48 bar pressure and temperatures between 160°C and 200°C for 30 min.

Tab. 1: Alternative treatments.

Sample	First treatment	Second treatment
D160	Densification at 160°C, 180°C or 200°C	No treatment
D180		
D200		
DT1902	Densification at 160°C, 180°C or 200°C	Heat treatment at 190°C for 2h, 4h and 6h
DT1904		
DT1906		
T1902	Heat treatment at 190°C for 2h, 4h and 6h	No treatment
T1904		
T1906		
TD1902	Heat treatment at 190°C for 2h, 4h and 6h	Densification at 160°C, 180°C or 200°C
TD1904		
TD1906		

Density and compression-set

After each measurement was taken at the middle and in both ends of the sample and an average was determined. Apparent density was determined accordingly. Compression-set was determined by Eq. 1.

$$C_r\,(\%) = \frac{L_0 - L_{afc}}{L_0} \times 100\% \tag{1}$$

where: L_0 - oven dry thickness before,
L_{afc} - after compression.

In order to determine the compression-set recovery when wood is re-moistured, samples with approximately 20 mm in tangential and longitudinal directions and with variable radial dimensions in accordance to the compression obtained were cut from. All samples were subjected to three cycles of water soaking during 1 week followed by oven dry during 24 h. After each cycle mass and dimensions were measured and compression-set recovery was determined in relation to the final thickness after the densification treatment as:

$$C_r(\%) = \frac{L_{cycle} - L_{afc}}{L_0 - L_{afc}} \times 100 \tag{2}$$

where: L_0 - oven dry thickness before compression,
L_{afc} - after compression,
L_{cycle} - after each cycle (Inoue et al. 2008).

Determination of physical and mechanical properties

Janka hardness was determined on the surface of the treated boards and determined by ISO 3350 measuring the force required to embed a 11.28mm steel ball on the radial surface of wood to half the ball's diameter. The tests were made in a Servosis universal test machine.

Bending strength and stiffness were determined by a three point bending test in a Servosis Universal test machine. Samples with approximately 5 mm thickness in tangential direction were cut from the original samples. The approximate dimensions of the samples were 145 mm longitudinal, 5 mm tangential and the radial dimension varying between 17-32 mm depending on the compression. Test were made with a 100 mm span and a constant velocity of 100 kgf·min^{-1}

MOE and bending strength were determined according to the following Eqs. 3 and 4:

$$\text{MOE}(\text{N}\cdot\text{mm}^{-2}) = \frac{\Delta F \times L^3}{\Delta x \times 4 \times b \times h^3} \qquad (\text{N}\cdot\text{mm}^{-2}) \tag{3}$$

where: $\frac{\Delta F}{\Delta x}$ - the slope of the elastic zone (N·mm^{-1}),
L - the arm length (mm),
h - height (mm),
b t - width all expressed (mm).

$$\text{Bending strength (MPa)} = \frac{3F_{máx} \times L}{2 \times b \times h^2} \qquad (\text{MPa}) \tag{4}$$

where F_{max} - load on rupture (N),
L - arm length (mm),
h - height (mm),
b - width (mm).

Durability

Durability against subterranean termites was determined by a no-choice termite resistance test using an adaptation of the EN 117 2012).

Colonies of 150 workers of *Reticulitermes grassei* Clément, collected from broken trees and stubs in a forest of *Maritime pine* situated about 25 km east of Lisbon, Portugal, were established in 200 ml glass jars with moistened sand (Fontainebleau sand and water; 4:1 v/v) as substrate.

Three replicates (30 x 10 x 10 mm)per treatment were then placed in contact with the termites and the test run for four weeks in a conditioned room with 24ºC ± 2ºC and 80% ± 5% relative humidity of the air. Maritime pine test specimens with the same dimensions were also included as virulence controls. The initial moisture content of the blocks was measured in sets of three additional replicates per treatment and these values were used to determine the theoretical initial dry mass (IDM) of the exposed specimens (in all tests conducted). At the end of the trial the final moisture content was recorded and the mass loss (percentage) was obtained using the expression: % mass loss = (FDM-IDM)/IDM x 100, where FDM is the oven dry mass of the block at the end of the test (blocks were cooled in a desiccated or before weighing).

In order to detect significant ($p<0.05$) variation caused by different wood treatments, the data obtained was submitted to analysis of variance (ANOVA), these analyses were done with RStudio (RStudio Team 2015) v 0.99.467 and R-3.1.2. Regression analysis was used on mass loss and wood density data with Microsoft excel® (2010).

The survival (%) of the termites was recorded and all wood blocks were graded in terms of termite attack using the scale: 0 = no damage; 1 = attempted attack; 2 = slight damage; 3 = superficial and inner damage; 4 = heavy inner damage.

RESULTS AND DISCUSSION

Density and compression-set

The compression-set for untreated wood densified at 160-200ºC ranged from 45.4% to 46.9%. Although compression-set increases with the pressing temperature the differences are not significant (Tab. 2). In relation to samples that were heat treated before compression, the compression-set was around 13%. This means that to attain a similar compression on samples heat treated prior to compression a higher pressing pressure would be necessary. This smaller compression-set is possible due to the formation of several crosslinks in lignin during the heat treatment making wood less suitable for softening. Although the decrease in the dimensions of the samples that were only heat treated is presented here as compression-set for comparison purposes, no compression was done. The decrease in dimensions is only due to the heat treatment. The compression-set depends on wood species, pressure and pressing temperature. Different compression-sets have been reported before, for instance Heger et al. (2004) reported a 66% compression-set by a two steps process: heating with saturated steam until 140°C during 10 minutes followed by densification under a maximum load of about 22 kN. Boonstra and Blomberg (2007) reported a 47-56% compression of *Radiata pine* boards by a combined heat treatment and densification process while (Welzbacher et al. 2008) reported a compression-set of between 39.3% and 47.8% for Norway spruce treated in three steps: heating up, compression, and cooling/conditioning. These authors also reported that the higher the temperature and duration of compression the higher the compression-set obtained.

As a result of compression, density increases. The final density was around 1040 $kg.m^{-3}$ for maritime pine densified wood corresponding to a 70% increase in relation to untreated wood. Similar results were reported by Welzbacher et al. (2008) that achieved a 44-80% increase in spruce density. Boonstra and Blomberg (2007) reported a higher increase of 82 to 123% after densification of *Radiata pine* boards. The density of the samples that were heat treated after the compression decreased with the heat treatment, reaching a final density of around 980 kg.m-3 traducing a 62% increase in relation to untreated wood. Similar results were reported by Pelit et al.(2015) with densified Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* L.) that presented, respectively, a 4% and 5% decrease in wood density after the heat treatment. This reduction is attributed to the mass loss due to heat treatment. For samples that were heat treated prior to compression the maximum density obtained was around 630 kg.m-3 which mean that the final density presents just a 3% increase in relation to initial wood. The density of the samples that were only heat treated decreased about 6% (Tab. 2). The density increase depends on the species. When testing at the same conditions Blomberg and Persson (2007) obtained different density increase for Scots pine (78%) and for birch (*Betula pendula*) (55%).

Tab. 2: Compression-set and density after the 1st and 2nd treatments.

Sample	Thickness (mm)			Compression-set (%)	Density (kg·m^{-3})		
	Initial	After 1st treatment	After 2nd treatment		Initial	After 1st treatment	After 2nd treatment
D160	32.3	17.6		45.4	615	1048	
D180	32.3	18.0		45.9	614	1031	
D200	32.3	17.2		46.9	612	1041	
DT1902	32.5	17.6	17.4	46.5	607	1057	988
DT1904	32.3	17.8	17.6	45,7	608	1026	973
DT1906	32.4	17.3	17.4	46,3	616	1036	1002
T1902	32.4	31.7		2.2	615	577	
T1904	32.3	31.6		2.4	607	569	
T1906	32.5	31.6		2.8	607	562	
TD1902	32.3	31.7	28.2	12.7	608	566	627
TD1904	32 .5	31.7	28.3	12.8	602	562	633
TD1906	32.5	31-6	28.3	13.0	609	563	619
Untreated	32.3				610	610	610

Compression-set recovery

Fig. 1 presents the compression-set recovery (Cr) of samples after three water soaking cycles followed by oven drying. The dimensions were determined after the drying step of each cycle. These tests showed that the Cr of densified wood without heat treatment was around 80% (in relation to final thickness after compression) regardless of the pressing temperature. This means that only 20% of the compression remained permanent. Almost all of the compression-set recovery was obtained after the first wetting cycle. Even though after the third cycle the Cr is slightly higher, the samples have a lot of checks due to the wetting/drying cycles which increases the radial dimension. Welzbacher et al. (2008) reported smaller Cr for densified wood without any post-treatment. Spruce samples densified at 160°C, 180°C and 200°C for 30 min reached a Cr of 43%, 53% and 34% respectively. Although the conditions and the compression-set (around 40%) are similar, a different species is used.

The Cr of heat treated wood before densification depends on the time of treatment. With a higher treatment the Cr is smaller. After the second cycle the recovery was about 70% for wood treated at 190 °C during 2h while for 4h treatment the recovery reduced to 57% and for 6h to about 50%. Nevertheless the Cr recovery is still very high. In the third cycle the Cr increased for all the treatments although there was already some damage in the samples due to the wetting/drying cycles.

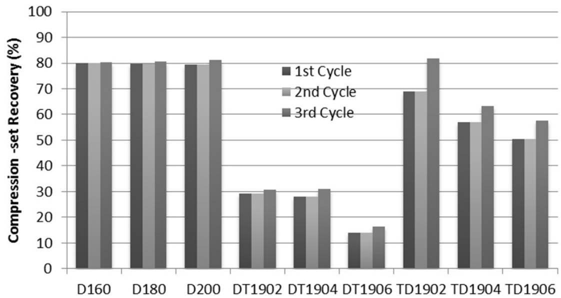

Fig. 1: Compression-set recovery after three wetting cycles for wood: densified (D), heat treated after densification (DT) and heat treated before densification (TD).

The best treatment is obtained when the densification is followed by the heat treatment. The compression-set recovery was less than 30% for the first two cycles and a little higher in the third cycle for all of the samples. For wood treated during 6 hours the Cr was less than 15%. So the higher the treatment temperature, the lower is the recovery. Similar results were presented (Welzbacher et al. 2008) for densified spruce wood heat treated at 220°C for 2h where Compression-set recovery after the first cycle varied from 3.5%-21.5% depending on the temperature and duration of densification. (Fang et al. 2011) reported an almost 0% recovery for densified Rotary-peeled aspen (*Populus tremuloides*) veneers post-treated by OHT at 180°C for 3h. In order to achieve a smaller Cr a higher temperature or time of treatment would be necessary. Probably the use of steam rather than heat would allow a Cr reduction at low temperatures as reported before (Inoue et al. 1993).

Heat treatment before densification seems to be less effective than after densification to enable compression-set recovery. Nevertheless we have to take into account that the compression-set for heat treated wood is smaller. It would be necessary to use a higher pressure to attain a compression-set similar to untreated wood. Tests are underway to try to obtain a similar compression-set on heat treated wood before compression in order to compare it with heat treated wood after compression.

Physical and mechanical properties

Janka hardness

Hardness is one of the most important properties for flooring and its increase is one of the biggest advantages of densified wood. Fig. 2 presents the hardness (Janka) of treated samples in relation to untreated. These tests showed that hardness of samples that were only heat treated and the samples densified after heat treatment have a hardness similar to untreated wood. Similar results were presented by Gong et al. (2010) that heat treated uncompressed and compressed aspen (*Populus tremuloides*) at 190°C, 200°C and 210°C and found that hardness of uncompressed heat treated wood was similar to untreated wood with a maximum decrease of 5% for wood treated at 210°C.

In relation to densified samples without heat treatment, hardness increased significantly and this increase was higher at higher densification temperatures. The increase varied between 50% to around 220%. Ulker et al. (2012) reported a similar increase in hardness (242%) for densified Scots pine at 140°C. Inoue et al. (1990) densified sugi (*Cryptomeria japonica* (L. f.) D. Don), hinoki (*Chamaecyparis obtuse* (Siebold & Zucc.) Siebold & Zucc. ex Endl.) and Western hemlock

(*Tsuga heterophylla* (Raf.) Sarg.) with a 45% compression and found an increase in hardness of 120–150%. Blomberg et al. (2005) densified Scots pine, Norway spruce, silver birch (*Betula pendula* Roth), black alder (*Alnus glutinosa* (L.) Gaertn.), European aspen (*Populus tremula* L.), European beech (*Fagus sylvatica* L.), English oak (*Quercus robur* L.) and European ash (*Fraxinus excelsior* L.). The highest hardness increase was found for spruce with a 293% increase and the lowest for oak with 154% increase in relation to initial value.

The hardness of the samples that were heat treated at 190°C after densification increased for 2 and 4h treatment, decreasing afterwards. The highest hardness value was obtained for densified wood post treated at 190°C for 4 h with an increase of around 220% in relation to initial value. In accordance to Rautkari et al.(2013) densification increased the Brinell hardness of Scots pine (*Pinus sylvestris*) wood more than 90 % and the post-hydrothermal treatment did not reduce Brinell hardness of control and densified samples. Different results were reported (Gong et al. 2010) that compressed Aspen (*Populus tremuloides*) and treated it with steam at 190°C, 200°C and 210°C and found a reduction of hardness of almost 37% after the treatment.

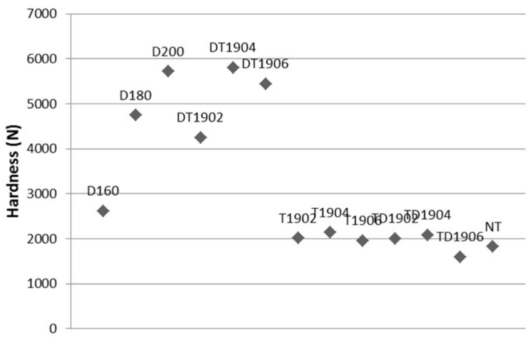

Fig. 2: MOE, Bending strength and Janka hardness changes in relation to untreated wood for: densified (D), heat treated (T), heat treated after densification (DT) and heat treated.

Bending strength

Fig. 3 presents bending strength of treated and untreated samples. These tests showed that densified samples have a higher bending strength with an increase of more than 60%. The samples that were heat treated after the compression present a smaller bending strength. Nevertheless the final bending strength is still 33% higher than for untreated wood. This means that overall the treatment increased bending strength which might be important for some utilization as for example for stairs where bending strength plays an important role. Similar results were reported (Kutnar et al. 2008) with low density hybrid poplar (Populus deltoides × Populus trichocarpa) treated by the VTC method. These authors found that the MOR of treated wood increased by 32, 66 and 102% for wood with a compression-set of 63, 98, and 132% in comparison with undensified wood. (Blomberg et al. 2005) densified eight different species and reported an increase in bending strength for all treated woods. Different results were reported (Ulker et al. 2012). These authors obtained anot significant difference between the bending strength of undensified (69.12 N·mm^{-2}) and densified (64.93 N·mm^{-2}) Scots pine compressed at a temperature of 160°C.

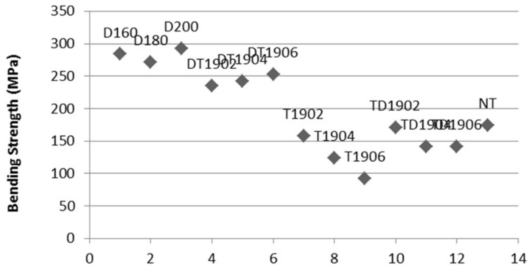

Fig. 3: Bending Strength for wood: untreated (NT), densified (D), heat treated (T), heat treated after densification (DT) and heat treated before densification (TD).

Bending stiffness (MOE) is presented in Fig. 4 as percentage difference from untreated wood (ΔMOE). Densified wood presented an increase in MOE from about 40% to 80%. The samples with a higher MOE corresponded to the samples pressed at higher temperature. The post-heat treatment did not seem to decrease MOE significantly. The samples that were only heat treated showed a small decrease in MOE reaching about a 20% decrease in relation to untreated wood. The samples that were compressed after the heat treatment exhibited a higher MOE than untreated wood for less intense heat treatment but decreasing with the intensity of the treatment. Similar results were presented by Kutnar et al.(2008) that reported a MOE increased by 37% in comparison with undensified wood for VTC wood with 63%degree of densification, increasingto 84 and 129% for 98 and 132% degrees of densification, respectively. (Gong et al. 2010) found a 13% increase in MOE of compressed Aspen (*Populus tremuloides*) that decreased with the post-heat treatment to reach a final MOE lower than untreated wood.

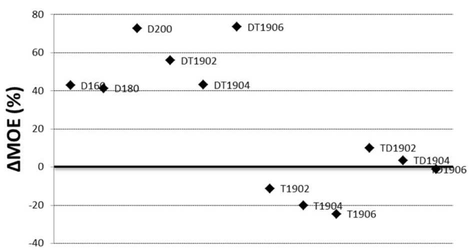

Fig. 4: MOE changes in relation to untreated wood for: densified (D), heat treated (T), heat treated after densification (DT) and heat treated before densification (TD).

Durability

The survival of the termites and the grade of the attack attributed at the end of the test were consistently high and did not show relevant differences between the different treatments (Tab. 3). The same happened with the survival rate that was approximately the same for untreated and treated wood ranging from 85.6-95.8%. The final moisture content was around 50% for most of the samples.

Tab. 3: Average results (n=3) for termite durability. Final moisture content, survival of termites and attack grade.

Sample	Final Moisture Content (%)	Survival (%)	Grade of attack
D160	49.4 (7.2)	89.3 (2.9)	4
D180	59.2 (18.8)	85.6 (5.0)	4
D200	49.6 (15.4)	95.8 (1.7)	4
DT1902	50.5 (10.1)	88.0 (7.1)	4
DT1904	53.4 (8.7)	90.4 (3.5)	3.7 (0.5)
DT1906	63.5 (5.9)	86.9 (6.3)	4
TD1902	52.4 (21.5)	92.7 (3.0)	4
TD1904	58.3 (15.0)	88.4 (4.6)	4
TD1906	43.1 (10.9)	88.7 (0.5)	4
T1902	76.0 (10.5)	88.0 (6.9)	4
T1904	51.5 (16.5)	86.9 (4.8)	4
T1906	43.5 (11.0)	89.1 (2.3)	4
Untreated	46.5 (20.8)	85.8 (1.7)	4

(Standard deviation in brackets)

Mass loss due to subterranean termite attack (*R. grassei*) is presented in Fig 5. For mass loss, the type of treatment was considered significant ($F=47.6$; $p=1.78e-13$). The results showed that D and DT wood are both significantly different from NT ($F=10.6; p<0.01$ and $F=8.9; p<0.01$, respectively), T ($F=10.5; p<0.01$ and $F=8.8; p<0.01$, respectively) and TD ($F=9.9; p<0.01$ and $F=8.2; p<0.01$, respectively). Results show that heat treatment alone does not significantly decrease mass loss. The samples that were compressed after heat treatment (TD) presented a smaller mass loss than untreated wood, although the difference was not significant. Wood that was heat treated after densification presented a mass loss lower than 8% which is almost half of the mass loss in untreated wood. The best results however were achieved with densified wood without heat treatment with a mass loss generally under 6%. These results seem to imply that the main factor affecting termite attack is density as previously observed for natural durability of different species (Arango et al. 2006)

Fig. 6 presents therefore the relation between mass loss due to termite attack and wood density. The results ($R^2=0.937$) show that there is a close relation between mass loss due to termite attack and wood density. Similar results were presented by

(Unsal et al. 2009) with thermally pressed wood in no-choice feeding tests with Eastern subterranean termites (*Reticulitermes flavipes*) that reported lower mass losses for thermally pressed wood. Choowang, (2013) also tested the effect of thermally compressing oil palm against subterranean termites (*Coptotermes gestroi*) in a 4-week no-choice test and concluded that the surface damage to the samples treated at 220 °C showed improved resistance to subterranean termites based on visual observation.

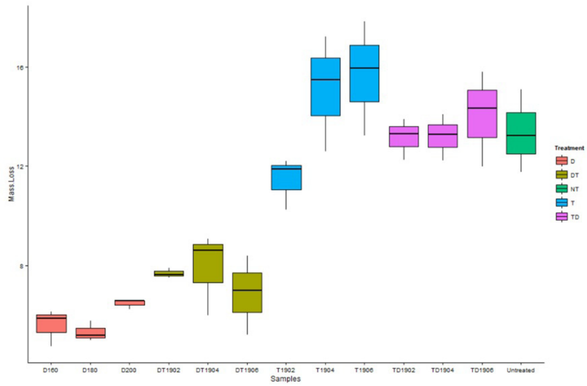

Fig. 5: Mass loss (%) due to termite attack on wood: untreated, densified (D), heat treated (T), heat treated after densification (DT) and heat treated before densification (TD).

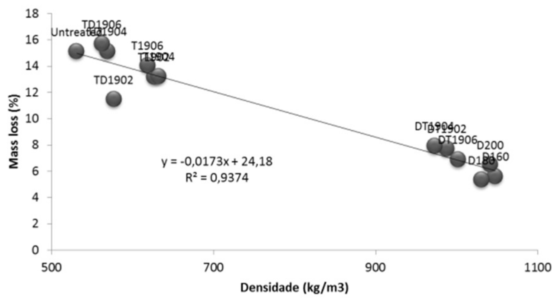

Fig. 6: Relation between mass loss due to termite attack and wood density.

CONCLUSIONS

Results show that densification increases density, hardness, bending strength and stiffness, and termite durability. Heat treatment applied after the densification is more effective in reducing compression-set recovery than if applied before. Pine wood from *Pinus pinaster* species densified and heat treated afterwards shows a high potential to replace more expensive hardwoods in the manufacture of flooring or stairs.

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REFERENCES

1. Arango, R. A., Green, F., Hintz, K., Lebow, P. K., Miller, R. B., 2006: Natural durability of tropical and native woods against termite damage by Reticulitermes flavipes (Kollar), International Biodeterioration & Biodegradation, 57(3): 146–150.
2. Blomberg, J., 2006: Mechanical and physical properties of semi-isostatically densified wood. Luleå University of Technology 62 pp.
3. Blomberg, J., Persson, B., 2007: Swelling pressure of semi-isostatically densified wood under different mechanical restraints, Wood Science and Technology 41(5),: 401–415.
4. Blomberg, J., Persson, B., Blomberg, A., 2005: Effects of semi-isostatic densification of wood on the variation in strength properties with density, Wood Science and Technology 39(5): 339–350.
5. Boonstra, M. J., Blomberg, J., 2007: Semi-isostatic densification of heat-treated *Radiata pine*, Wood Science and Technology 41(7): 607–617.
6. Choowang, R. 2013: Effects of hot pressing on resistance of compressed oil palm wood to Subterranean termite (Coptotermes gestroi Wasmann) attack, BioResources 9(1): 656−661.
7. Diouf, P. N., Stevanovic, T., Cloutier, A., Fang, C.-H., Blanchet, P., Koubaa, A., Mariotti, N., 2011: Effects of thermo-hygro-mechanical densification on the surface characteristics of trembling aspen and hybrid poplar wood veneers, Applied Surface Science 257(8): 3558–3564.
8. Dwianto, W., Morooka, T., Norimoto, M., Kitajima, T., 1999: Stress relaxation of sugi (*Cryptomeria japonica* D. Don) wood in radial compression under high temperature steam Holzforschung 53(5): 541–546.
9. Esteves, B. M., Pereira, H., 2009: Wood modification by heat treatment: A review, BioResources (4): 340−404.
10. EN117. 2012: Wood preservatives. Determination of toxic values against Reticulitermes species (European termites) (Laboratory method).
11. Fang, C.H., Cloutier, A., Blanchet, P., Koubaa, A., Mariotti, N., 2011: Densification of wood veneers combined with oil-heat treatment. Part I: Dimensional stability, BioResources, 6(1): 373–385.
12. Gong, M., Lamason, C., Li, L., 2010: Interactive effect of surface densification and post-heat-treatment on aspen wood, Journal of Materials Processing Technology 210(2): 293–296.
13. Heger, F., Groux, M., Girardet, F., Welzbacher, C., Rapp, A. O., Navi, P., 2004: Mechanical and durability performance of THM-densified wood, in: Final Workshop COST Action E22 Environmental optimisation of wood protection, Lisbon, Pp 22–23.
14. Inoue, M., Norimoto, M., Otsuka, Y., Yamada, T., 1990: Surface compression of coniferous wood lumber I. A new technique to compress the surface layer, Mokuzai Gakkaishi 36(11): 969–975.
15. Inoue, M., Norimoto, M., Tanahashi, M., Rowell, R. M., 1993: Steam or heat fixation of compressed wood, Wood and Fiber Science 25(3): 224–235.
16. Inoue, M., Sekino, N., Morooka, T., Rowell, R. M., Norimoto, M., 2008: Fixation of compressive deformation in wood by pre-steaming, Journal of Tropical Forest Science 20(4): 273–281.
17. Ito, Y., Tanahashi, M., Shigematsu, M., Shinoda, Y., 1998a: Compressive-molding of wood by high-pressure steam-treatment: Part 2. Mechanism of permanent fixation, Holzforschung 52(2): 217–221.

18. Ito, Y., Tanahashi, M., Shigematsu, M., Shinoda, Y., aOhta, C., 1998b: Compressive-molding of wood by high-pressure steam-treatment: Part 1. Development of compressively molded squares from thinnings, Holzforschung 52(2): 211–216.
19. Khademi Bami, L., Mohebby, B., 2011: Bioresistance of poplar wood compressed by combined hydro-thermo-mechanical wood modification (CHTM): Soft rot and brown-rot, International Biodeterioration & Biodegradation 65(6): 866–870.
20. Kollmann, F., 1951: Technologie des Holzes und der Holzwerkstffe, Springer-Verlag, Berlin 1054 pp.
21. Kulticova, E. V., 1999: Structure and properties relationship of densified wood, M. Sc. Thesis, Polytechnic Institute and State University, Blacksburg, VA, USA.
22. Kutnar, A., Humar, M., Kamke, F. A., Sernek, M., 2011: Fungal decay of viscoelastic thermal compressed (VTC) wood, European Journal of Wood and Wood Products 69(2): 325–328.
23. Kutnar, A., Kamke, F. A., Sernek, M. 2008: The mechanical properties of densified VTC wood relevant for structural composites, Holz als Roh-und Werkstoff 66(6): 439–446.
24. Li, Y. F., Gong, D. W., Dong, X. Y., Liu, Z. B., 2012: Mechanical properties of wood-polymer composite prepared by in-situ polymerization of methylmethacrylate and styrene, Applied Mechanics and Materials 166: 2938–2941.
25. Morsing, N., Hoffmeyer, P. 2000: Densification of Wood.: The influence of hygrothermal treatment on compression of beech perpendicular to gain, Technical University of Denmark, Danmarks Tekniske Universitet, Administration, Office for Study Programmes and student affairs afdelingen for uddannelse og studerende.
26. Müller, U., Gindl, W., Teischinger, A., 2003: Effects of cell anatomy on the plastic and elastic behaviour of different wood species loaded perpendicular to grain, Iawa Journal 24(2): 117–128.
27. O'Connor, J. P., 2007: Improving wood strength and stiffness through viscoelastic thermal compression, Oregon State University, USA.
28. Pelit, H., Sönmez, A., Budakçı, M., 2015: Effects of thermomechanical densification and heat treatment on density and Brinell hardness of Scots pine (*Pinus sylvestris* L.) and Eastern beech (*Fagus orientalis* L.), BioResources 10(2): 3097–3111.
29. Rautkari, L., Laine, K., Kutnar, A., Medved, S., Hughes, M., 2013: Hardness and density profile of surface densified and thermally modified Scots pine in relation to degree of densification, Journal of Materials Science 48(6): 2370–2375.
30. Rautkari, L., Properzi, M., Pichelin, F., Hughes, M., 2010: Properties and set-recovery of surface densified Norway spruce and European beech, Wood Science and Technology 44(4): 679–691.
31. RStudio Team 2015: RStudio: Integrated development for R. RStudio, Inc., Boston, MA.
32. Schwarze, F. W., Spycher, M., 2005: Resistance of thermo-hygro-mechanically densified wood to colonisation and degradation by brown-rot fungi, Holzforschung 59(3): 358–363.
33. Seborg, R., Millet, M., Stamm, A. J., 1945: Heat stabilized compressed wood (Staypak), Mechanical Engineering 67: 25–31.
34. Skyba, O., Niemz, P., Schwarze, F. W., 2008: Degradation of thermo-hygro-mechanically (THM)-densified wood by soft-rot fungi, Holzforschung 62(3): 277–283.
35. Tu, D., Su, X., Zhang, T., Fan, W., Zhou, Q., 2014: Thermo-mechanical densification of Populus tomentosa var. tomentosa with low moisture content, BioResources 9(3): 3846–3856.

36. Ulker, O., Imirzi, O., Burdurlu, E., 2012: The effect of densification temperature on some physical and mechanical properties of Scots pine (*Pinus sylvestris* L.), BioResources 7(4): 5581–5592.
37. Unsal, O., Kartal, S. N., Candan, Z., Arango, R. A., Clausen, C. A., Green, F., 2009: Decay and termite resistance, water absorption and swelling of thermally compressed wood panels, International Biodeterioration & Biodegradation 63(5): 548–552.
38. Welzbacher, C. R., Wehsener, J., Rapp, A. O., Haller, P., 2008: Thermo-mechanical densification combined with thermal modification of Norway spruce (*Picea abies* 39. 39. Karst) in industrial scale–dimensional stability and durability aspects, Holz als Roh-und Werkstoff 66(1): 39–49.

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