Effect of Vacuum and Pressure Treatments on the Mechanical Properties and Moisture Balance of Wood from *Pinus sylvestris*

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Several works have analyzed the alteration in the mechanical strength of wood after the application of protective substances. However, it remains unclear whether the significant differences obtained are caused by the protective substances, the pressure and vacuum conditions used in the treatment, or the simultaneous effect of both. In this study, 123 wood samples from *Pinus sylvestris* were tested for bending, and 85 samples were tested for compression strength parallel to the grain. These samples were randomly distributed in three groups (treated with water pressure, water vacuum, and control samples simply submerged in water). The results indicated that there was no difference in mechanical properties between the treated and untreated samples. In contrast, significant differences were detected in equilibrium moisture after a prolonged drying process.

Keywords: Mechanical properties; Moisture; Pinus sylvestris; Pressure treatment; Stiffness; Strength; Treated wood; Vacuum

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INTRODUCTION

Currently, a large proportion of the timber used in structures is treated to protect it from deterioration by fungi and xylophagous insects. These treatments are particularly important for timber located outdoors or in contact with the ground, where its useful life may be drastically curtailed due to decay. The problems caused by this reduced lifespan include the need to acquire new structural elements and their associated re-installation costs. Several authors have analyzed the effect of introducing different protective substances to the wood interior. In some cases there are significant losses in mechanical properties, as in the modulus of elasticity (MOE) in southern pine treated with copper xyligen (Barnes *et al.* 2009) and the modulus of rupture (MOR) in *Pinus sylvestris* treated with environmentally friendly borates (Simsek *et al.* 2010). These results coincide with findings showing reduced MOR in wood treated with waterborne preservatives (Forest Products Laboratory 2010).

In contrast to these studies, other works indicate that the MOR or MOE are not altered by these treatments, or are significantly improved (Yildiz *et al.* 2004; Barnes *et al.* 2005, 2008, 2009; Barnes and Lindsey 2009; Villasante *et al.* 2013; Barnes *et al.* 2014). These results concur with those of the Forest Products Laboratory (2010) for oil-type preservatives and for the MOE in wood treated with waterborne preservatives. This disparity reflects that all studies use different protective substances, each of which can

alter mechanical strengths in a different way.

The impact of short duration pre-steaming and treatment was analyzed for hardwoods (Barnes *et al*. 2014). In these experiments, no significant differences were observed in the mechanical properties (bending and parallel compression). Similarly, the pressure or vacuum conditions during the treatment can also cause alterations in the mechanical properties. However, their impact is obscured because it occurs in tandem with those associated with the protective substance. Thus, some researchers use untreated wood for their test samples, while others have opted for wood treated (under pressure or vacuum) only with water (Yildiz *et al.* 2004; Barnes *et al.* 2005, 2008, 2009; Barnes and Linsay 2009; Simsek *et al.* 2010; Villasante *et al.* 2013). However, the effect of pressure or vacuum has not yet been accurately determined, as these works only use one of the two types of test sample. The aim of this work was to determine the effect of vacuum and pressure conditions on the wood's properties and the equilibrium moisture content.

EXPERIMENTAL

Obtaining the Samples

The study was conducted on ten sawn pieces of *Pinus sylvestris* L. wood with a length of 1.2 m (100 mm × 200 mm) from the municipal district of Plan in Spain (42°34'N, 0°21'E). Cuts were made with a bandsaw and circular saw perpendicular and parallel to the growth rings. The strips obtained were thickened to achieve a square section with an edge of 20 mm, according to UNE 56-537-79 (1979), and subsequently cut crosswise in lengths of 280 mm to adapt them to the dimensions of the treatment chamber. All samples with defects that might affect the strength of the wood (knots, cracks, deviation of the fiber, fungal attacks, *etc*.) were eliminated. In areas where this was not possible, lengths of 60 mm were extracted (also free from defects) to conduct the mechanical testing for compression strength parallel to grain (UNE 56-535-77 1977).

Bending Tests

The 123 samples, measuring 280 mm in length for the bending test, were randomly distributed in three groups, each of which was subjected to a different type of treatment. The two most frequent situations in the current industrial processes were selected. One type of treatment was the application of vacuum, similar to the double vacuum system used with organic preservatives. The second type of treatment consisted of applying pressure similar to the methods used industrially with waterborne preservatives. In the second case it was decided not to apply the prior or final vacuum phase in order to be able to separate with certainty the effects of positive and negative pressure.

The first group of 41 samples were exposed to an initial vacuum of 20 kPa for 10 min, following which the vessel was filled with water and maintained at atmospheric pressure for 15 min. The water was finally removed, and a vacuum of 87 kPa was applied for 25 min. The second group of 41 samples was treated in the autoclave with water at 1177 kPa of pressure for 90 min. The third group, comprising the 41 control samples, was submerged in water for 1 h at atmospheric pressure.

The treated samples were stored in a conditioning chamber for three weeks at 20 °C and relative moisture of 65%. The bending strength was subsequently calculated in a Microtest PB2-F50 testing machine (Madrid, Spain) with a central load and a distance of

240 mm between supports (UNE 56-537-79). The crosswise measurements of the samples were taken immediately prior to the test using a micrometer with a precision of 0.01 mm. The samples were placed with the rings in the vertical position. Once the test was complete, they were weighed (with a precision of 0.01 g), and subsequently dried at 103 °C to obtain their moisture. Given the differences in moisture obtained, the 4% factor recommended by Hoffmeyer (1995) was applied to correct the bending strength of defect-free wood (Eq. 1). The value for the bending modulus of elasticity was 1.5%, as determined by Eq. 2 (Hoffmeyer 1995). The values were corrected at reference moisture of 12%.

$$MOR_{12} = \frac{MOR_m}{1-0.04 \cdot (m-12)} \quad (1)$$

$$MOE_{12} = \frac{MOE_m}{1-0.015 \cdot (m-12)} \quad (2)$$

In Eqs. 1 and 2, m is moisture content (%), MOR_{12} is the bending compression strength at 12% moisture, MOR_m is the bending compression strength at m moisture, MOE_{12} is the bending modulus of elasticity at 12% moisture, and MOE_m is the bending modulus of elasticity at m moisture.

Compression Strength and Equilibrium Moisture Samples

The 85 samples, with a length of 60 mm for the test of compression strength parallel to grain, were also distributed randomly in three groups. The same treatments as the bending samples were applied (29 with pressure, 29 with vacuum, and 27 control samples), after which the samples were stabilized for two months at laboratory moisture, and subsequently stored in a conditioning chamber. The moisture analysis was done with the compression samples, as their shorter length means they reach the state of equilibrium faster. To achieve the correct homogeneity of moistures, the 85 samples were submitted to a first phase (1 month duration) in which temperature conditions of 20 °C ± 1 °C were established by combining a range of relative moistures above and below 65%. The samples were then once again stored in the laboratory for one month. Finally, they were replaced in the conditioning chamber at a temperature of 20 °C ± 1 °C and a relative moisture of 65% ± 5% for 15 days. After this stabilization process lasting four and a half months, it was verified that the samples had a constant weight in readings taken 48 h apart.

The crosswise dimensions of the samples were measured using a micrometer (accuracy of 0.01 mm) seconds before they were placed in the Matest Srl H11 testing machine (Treviolo, Italy) for compression strength parallel to the grain (UNE 56-535-77 1977). The samples were loaded on the press plates at a speed of 25 $N \cdot mm^{-2} \cdot minute^{-1}$ until their rupture. The remains were weighed with an accuracy of 0.01 g and dried at 103 °C to a constant weight to calculate their moisture. Equation 3 shows the 5% factor that was applied to correct the compression strength parallel to grain in defect-free wood (Hoffmeyer 1995). A correction factor of 1% was used in the case of the compression modulus of elasticity according to Eq. 4 (EN 384 2010).

$$C_{12} = \frac{C_m}{1-0.05 \cdot (m-12)} \quad (3)$$

$$MOEC_{12} = \frac{MOEC_m}{1-0.01 \cdot (m-12)} \quad (4)$$

In Eqs. 3 and 4, m is moisture content, C_{12} the compression strength parallel to grain at 12% moisture, C_m the compression strength at m moisture, $MOEC_{12}$ the compression modulus of elasticity parallel to grain for 12% moisture, and $MOEC_m$ the compression modulus of elasticity parallel to grain for m moisture, respectively.

Statistical Analysis

Before analyzing the mechanical properties of the samples, an analysis of biases was conducted to verify that the groups had been formed correctly. The variables analyzed were specific weight at 12% moisture, radial measurement, and tangential measurement. The verification was made by previously applying the Shapiro-Wilk test (normality check) and Bartlett's test (homoscedasticity check). If the results indicated that the requirements of normality and homoscedasticity were met, the bias was verified with the analysis of variance. Otherwise, the non-parametric Kruskal-Wallis test was used.

The differences in the mechanical properties between the different treatment groups were verified following the same procedure, analysis of variance or the Kruskal-Wallis test, based on the compliance with the requisites of normality and homoscedasticity. Statistical analyses were performed with the R package (R 2012). The level of significance was established at 0.05.

RESULTS AND DISCUSSION

After testing for possible errors in the distribution of the samples and groups, the results did not indicate any significant differences between the groups of woods treated with pressure or vacuum and the control samples (Table 1). The analysis of bias confirmed the random distribution of the samples in groups.

The mean values of the mechanical strength after different treatments are shown in Table 2. Table 3 contains the results of the statistical tests, which can be consulted to determine the existence of significant differences. The effect of the treatment on mechanical strength was not significant in any of the cases. In contrast, in spite of the long period of moisture stabilization, the equilibrium moisture content showed clearly significant values of less than 0.01.

The values for the mechanical strength of the untreated samples matched those obtained by Gutierrez and Plaza (1967) for *Pinus sylvestris* wood from Spain. These authors indicated average values of 40.6 $N \cdot mm^{-2}$ and 105.7 $N \cdot mm^{-2}$ for compression strength parallel to grain and bending strength, respectively, in 166 defect-free samples. These values do not differ substantially from those obtained in the present study (43.8 $N \cdot mm^{-2}$ and 99.5 $N \cdot mm^{-2}$, respectively) and are within the 95% confidence intervals that were established with the data of Gutierrez and Plaza (1967): 37.7 $N \cdot mm^{-2}$ at 43.6 $N \cdot mm^{-2}$ in the case of compression strength parallel to grain and 96.9 $N \cdot mm^{-2}$ at 114.4 $N \cdot mm^{-2}$ in the case of bending.

Table 1. Analysis of Bias in the Distribution of Samples

Variable	Type of sample	Shapiro-Wilk			Bartlett	ANOVA	Kruskal-Wallis
		Pressure	Vacuum	Control			
Density (oven-dry)	B	n.s. (0.336)	n.s. (0.144)	n.s. (0.333)	n.s. (0.847)	n.s. (0.439)	---
Radial dimension (oven-dry)	B	n.s. (0.185)	n.s. (0.302)	n.s. (0.179)	n.s. (0.366)	n.s. (0.589)	---
Tangential dimension (oven-dry)	B	0.010	0.018	0.005	n.s. (0.632)	---	n.s. (0.149)
Number of growth rings	B	n.s. (0.070)	n.s. (0.311)	n.s. (0.231)	n.s. (0.492)	n.s. (0.391)	---
Density (oven-dry)	C	n.s. (0.390)	0.018	n.s. (0.076)	n.s. (0.227)	---	n.s. (0.873)
Radial dimension (oven-dry)	C	n.s. (0.961)	0.037	n.s. (0.095)	n.s. (0.988)	---	n.s. (0.929)
Tangential dimension (oven-dry)	C	n.s. (0.054)	n.s. (0.687)	n.s. (0.093)	n.s. (0.917)	n.s. (0.901)	---
Number of growth rings	C	n.s. (0.727)	n.s. (0.076)	n.s. (0.268)	n.s. (0.980)	n.s. (0.918)	---

Values indicate *p*-values of each test (between brackets if the result is not significant). B = Bending; C = Compression; n.s. = not significant. Test results indicating the presence of bias are shown in ANOVA or Kruskal-Wallis columns.

No significant differences were detected in the mechanical strengths between vacuum-treated wood samples and control samples. These results confirm those found in Blair *et al.* (1967), who reported that the effect of vacuum on compression and bending strengths was negligible.

Several studies have established the effect of drying wood with vacuum. Ouertani *et al.* (2015) did not detect any significant changes in the MOE and MOR in vacuum-dried *Pinus banksiana* compared with convective drying processes; these results are similar to those of the present study. In contrast, Altun *et al.* (2011) found increases in bending and compression strength in infrared-dried *Pinus sylvestris* wood in vacuum conditions compared with wood dried in a conventional kiln. In this case, the results do not coincide with those of the present work due to the fact that their experiments were done by drying the wood at high temperatures, thus producing a combined effect of temperature and vacuum.

Table 2. Mean Values, Standard Deviations, and Confidence Intervals for the Mechanical Properties and Equilibrium Moisture Content

	Mean	SD	95% Confidence Interval
MOR_{12} ($N \cdot mm^{-2}$)			
Pressure	97.7[a]	6.99	95.53 – 99.94
Vacuum	96.6[a]	7.02	94.34 – 98.78
Control	99.5[a]	7.79	97.02 – 101.94
MOE_{12} ($N \cdot mm^{-2}$)			
Pressure	8774[a]	720	8547 – 9001
Vacuum	8592[a]	871	8317 – 8867
Control	8862[a]	830	8600 – 9124
C_{12} ($N \cdot mm^{-2}$)			
Pressure	43.9[a]	3.55	42.58 – 45.29
Vacuum	44.9[a]	1.83	44.21 – 45.60
Control	43.8[a]	3.34	42.50 – 45.14
$MOEC_{12}$ ($N \cdot mm^{-2}$)			
Pressure	3884[a]	704	3616 – 4152
Vacuum	4115[a]	484	3932 – 4299
Control	3925[a]	657	3666 – 4185
Equilibrium moisture content (%)			
Pressure	13.4[a]	0.362	13.30 – 13.57
Vacuum	13.4[a]	0.337	13.31 – 13.56
Control	13.1[b]	0.253	13.04 – 13.25

[a. b] Different letters show significant difference using Tukey's mean separation test ($p = 0.05$). MOR_{12} = bending compression strength at 12% moisture; MOE_{12} = bending modulus of elasticity at 12% moisture; C_{12} = compression strength parallel to grain at 12% moisture; $MOEC_{12}$ = compression modulus of elasticity parallel to grain for 12% moisture

Table 3. Effect of Pressure or Vacuum Treatments on the Mechanical Properties and Equilibrium Moisture Content

Variable	Shapiro-Wilk			Bartlett	ANOVA	Kruskal-Wallis
	Pressure	Vacuum	Control			
MOR_{12}	n.s. (0.512)	n.s. (0.223)	n.s. (0.967)	n.s. (0.739)	n.s. (0.192)	---
MOE_{12}	n.s. (0.272)	n.s. (0.517)	n.s. (0.083)	n.s. (0.472)	n.s. (0.309)	---
C_{12}	n.s. (0.924)	n.s. (0.455)	n.s. (0.602)	0.0013**	---	n.s. (0.277)
$MOEC_{12}$	n.s. (0.434)	n.s. (0.786)	n.s. (0.945)	n.s. (0.130)	n.s. (0.330)	---
Equilibrium moisture content	0.035*	n.s. (0.202)	n.s. (0.080)	n.s. (0.195)	---	0.0029**

MOR_{12} = bending compression strength at 12% moisture; MOE_{12} = bending modulus of elasticity at 12% moisture; C_{12} = compression strength parallel to grain at 12% moisture; $MOEC_{12}$ = compression modulus of elasticity parallel to grain for 12% moisture.

n.s. = not significant; * = 0.01 < *p*-value ≤ 0.05; ** = 0.001 < *p*-value < 0.01. Test results indicating the influence of the treatment are shown in bold.

The results presented here concur with several other works that use wood treated with water as test samples, in which no significant differences were found in the MOR and MOE in wood treated with different products applied with vacuum and pressure (Barnes *et al.* 2005, 2008). The results also agree with some similar studies that used untreated wood as test samples, *e.g.*, of Yildiz *et al.* (2004) for treatments with Tanalith, Simsek *et al.* (2010) for the MOR in wood from *Fagus orientalis*, and Suirezs (2005).

Other studies detected significant differences between untreated, vacuum, and pressure-treated woods (Barnes and Lindsey 2009; Villasante *et al.* 2013; Barnes *et al.* 2014). By ruling out the effect of changes in pressure on the mechanical strength of the wood in these cases, the present study confirms that alterations are caused by the chemical substances applied to the wood.

The present work has detected significant differences in the equilibrium moisture between vacuum or pressure-treated wood in water, and wood simply submerged in water. This difference was not very great (the average moisture in treated samples was 0.3% higher than in untreated samples), but was clearly significant ($p = 0.0029$). No other studies have reported this effect, possibly due to its limited impact. One surprising finding was that the effect of pressure was similar in treatments with negative pressure (vacuum of 20 kPa) and positive pressure (1177 kPa); no significant differences were detected between them. This result can be explained by the alterations caused by pressure and vacuum on the aspiration of bordered pits, as these anatomical elements control the movement of fluids in conifer wood (Petty 1970). Another explanation could be related to the alteration in the extractives during the treatment processes. However, the work of Choong and Achmadi (1991) indicates that below 70% relative humidity, the removal of extractives does not alter the equilibrium moisture in the wood. As the moisture analysis in the present study was done at 65% relative humidity, the change in the equilibrium moisture cannot be justified by the quantity of extractives present in the samples.

CONCLUSIONS

1. The application of vacuum (87 kPa for 20 min) or pressure (1177 kPa for 90 min) in wood protective treatments for *Pinus sylvestris* does not alter its MOR, MOE, compression strength, or compression modulus of elasticity parallel to the grain.
2. The application of both vacuum and pressure causes significant increases in the equilibrium moisture of wood.

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