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Mechanical properties of wood from *Pinus sylvestris* L. treated with Light Organic Solvent Preservative and with waterbone Copper Azole

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Abstract

Aim of study: To determine the effect on wood from *Pinus sylvestris* of treatment with preservatives on mechanical properties and to establish the relation between the penetration and compression strenght.

Area of study: Spain.

Material and methods: 40 samples of defect-free wood from *Pinus sylvestris* L. were treated with Light Organic Solvent Preservative (Vacsol Azure WR 2601) and 50 with waterborne Copper Azole (Tanalith E 3492). 40 control samples were not treated (water or preservative). Mechanical resistance to static bending, modulus of elasticity and compression strength parallel to the grain were compared with untreated wood. Regression analysis between the penetration and compression strength parallel was done with the samples treated with waterborne preservative.

Main results: The results indicate that the treated wood (with either product) presents a statistically significant increase in mechanical resistance in all three mechanical characteristics. The results obtained differ from earlier studies carried out by other authors.

There was no correlation between parallel compression strength and the degree of impregnation of the wood with waterborne Copper Azole. The most probable explanation for these results concerns changes in pressure during treatment.

The use of untreated control samples instead of samples treated only with water is more likely to produce significant results in the mechanical resistance studies.

Research highlights: Treated wood presents a statistically significant increase in MOE, modulus of rupture to static bending and parallel compression strength.

There was no correlation between parallel compression strength and the degree of impregnation with waterborne preservative.

Key words: Light Organic Solvent Preservative; MOE; parallel compression; static bending; waterborne Copper Azole; wood technology.

Introduction

Currently the most widely used method of protection for wood against biodegradation consists of applying chemical substances. When using treated wood in structures, it is essential to be aware of the alterations that its mechanical characteristics can undergo as a result of this treatment.

In most of the studies published, the influence of the treatment on mechanical strength was not significant.

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Abbreviations: C_m (parallel compression to the grain at moisture *m*); C_{12} (parallel compression to the grain at 12% moisture); C-A (waterborne Copper Azole); LOSP (Light Organic Solvent Preservative); MOR (modulus of rupture to static bending at test moisture); MOR_{12} (modulus of rupture to static bending at 12% moisture).

Treatments included alkaline copper quat (Barnes *et al.,* 1993), waterborne copper naphthenate (Barnes *et al.,* 2005), micronized copper quat and copper azole (Barnes *et al.,* 2008) and copper xyligen in the study of MOR (Barnes *et al.,* 2009). Winandy (1995) indicates that the mechanical strength of wood treated with oil-type preservatives is not altered.

A significant relation is found in very few studies, and most point to losses in strength in the treated wood, as in the case of Winandy (1995) with waterborne preservatives. Barnes and Lindsey (2009) have studied the effects of treatment on the bending properties of southern pine using a product composed of propiconazole, tebuconazole and imidacloprid (PTI). They observe improvements in mechanical behavior compared to the control samples. Losses in mechanical strength are also reported in Barnes *et al.* (2009), who detected slight reductions in the MOE in wood treated with copper xyligen; and in Yildiz *et al*. (2004), who used different waterborne preservatives on wood from *Pinus sylvestris*. Furthermore, Simsek *et al*. (2010) also tested reduction in mechanical resistance for wood impregnated with environmentally-friendly borates.

Forest Products Laboratory (2010) indicates that water-based protectors may yield losses in mechanical strength in treated wood, an effect which is not observed in wood treated with organic-based products due to the fact that they do not react with the cell wall. It also reports that both the MOE and the parallel compression of treated wood are not affected, or else present only slight increases, unlike bending strength, which decreases between 0% and 20%. Another interesting aspect reported here is that wood of higher quality yields greater losses in mechanical strength.

The relation between mechanical strength and protection of wood is reported in the recent European regulation EN 15228:2009, whose annex A contains a list of protective substances used in Europe which do not produce modifications in the strength or rigidity of structural wood.

Most studies on the effect of chemical products on the mechanical strength of wood have established comparisons using samples treated only with water. In the present study we have opted to use samples of artificially dried wood which were not treated with water (Barnes and Lindsey, 2009), as commonly occurs in the usual commercial process for untreated wood.

The objectives of this study were: 1) to determine the effect on wood from *Pinus sylvestris* of treatment with Light Organic Solvent Preservative (Vacsol Azure WR 2601) and waterborne Copper Azole (Tanalith E 3492) on static bending, modulus of elasticity and compression strength parallel to the grain; and 2) to establish the relation between the penetration of the product into the wood and compression strength parallel to the grain.

Materials and methods

The samples were obtained in Segovia (Spain), from pure stands of *Pinus sylvestris* L. (one of the most commonly used species in Europe for construction). The logs were sawn into boards and dried at maximum temperatures of 65°C. They were subsequently planed and thinned until dimensions of 20 mm × 20 mm × 370 mm (radial, tangential and longitudinal measurements respectively). Samples which showed evidence of knots, cracks or processing defects were also eliminated. Additionally, samples whose transversal surface showed angles of over 15° between the transversal edges and the rings were eliminated (according to Nicholas *et al.,* 2009).

Before of treatment, all the samples were cut to a length of 280 mm to obtain the samples for subsequent bending tests. The remaining length of each sample was used to obtain compressive strength parallel to the grain samples (with a section of 20 mm × 20 mm and a length of 60 mm). The 280 mm and 60 mm samples were treated simultaneously in the impregnation phase.

The Light Organic Solvent Preservative (LOSP) used was Vacsol Azure WR 2601, composed of the active substances tolylfluanid (1.55%), tebuconazole (0.776%), propiconazole (0.776%) and permethrin (0.776%). The remaining components are light naphtha and excipients.

The waterborne Copper Azole (C-A) used was Tanalith E 3492, composed of copper carbonate (20.5%), 2-aminoethanol (18.5%), boric acid (4.5%), propiconazole (0.56%) and tebuconazole (0.42%). The remaining components are solvents and excipients. The product was applied after dissolving in water at a proportion of 6% as recommended by the manufacturer.

Three groups of samples were selected at random from the defect-free samples:

— 40 samples to be impregnated with LOSP.

— 50 samples to be impregnated with C-A.

— 40 control samples, which were not impregnated.

The 40 samples treated with LOSP were impregnated by means of an initial vacuum of 20 kPa for 10

minutes, after which time the protective product was added and maintained under atmospheric pressure for 10 minutes. The process was concluded with a final vacuum of 87 kPa for 25 minutes.

The 50 samples in the C-A batch were treated by means of an initial vacuum of 20 kPa for ten minutes, after which time the protective product was added and maintained at atmospheric pressure for 15 minutes. The process was concluded with a final vacuum of 87 kPa for 25 minutes.

A representative group of the specimens treated (12 samples per treatment) was weighed individually before and after impregnation in order to monitor the amount of product absorbed. The weight retention per volume of the samples was obtained from these values and from the impregnated volumes.

The following parameters were measured in each sample:

- Length (precision ±0.5 mm).
- Radial dimension (precision ±0.01 mm).
- Tangential dimension (precision ±0.01 mm).
- Number of growth rings at the squared end.
- Percentage of heartwood, in surface % (precision ±5%).
- Percentage of deviation of the grain from the axis of the sample (precision ±1%).
- Weight at the time of the mechanical tests (precision ±0.01 g).
- Density at the time of the mechanical tests (precision ±0.001 $g \cdot cm^{-3}$).

The samples for the bending test were air-dried for two months and subsequently examined to determine the modulus of rupture to static bending at the test moisture (MOR_m), and the modulus of elasticity (MOE), following the protocol established by Standard UNE 56537:1979. The estimated value of the MOR was calculated at 12% moisture (MOR_{12}) based on the information contained in Forest Products Laboratory (2010) according to the following formula [1]:

$$MOR_{12} = MOR_m \times [1 + 0.045 \times (\%moisture - 12)] \quad [1]$$

The samples for the compression parallel tests were air-dried for five months. The subsequent process was the same as for the bend tests. The samples were tested following the protocol established in Standard UNE 56535:1977 to obtain the parallel compressive strength at the test moisture (C_m), and using the formula included in the aforementioned Standard [2] to correct parallel compressive strength to 12% moisture content (C_{12}).

$$C_{12} = C_m \times [1 + 0.04 \times (\%moisture - 12)] \quad [2]$$

Moisture content was calculated by oven-drying the samples at 103°C immediately after the test.

The penetration of the treatment was calculated by applying reagents and evaluating the percentage of cross section area treated (with an approximation of 1%). The reagent used to detect the penetration of the LOSP was a solution of aniline at 15% in glacial acetic acid. In the case of the C-A, 0.05 g of chromazurol S and 0.5 g of sodium acetate were dissolved in 99 ml of distilled water.

The impregnation was complete in woods treated with LOSP. In woods treated with C-A, the cross section areas were digitalized with a scanner with a resolution of 600 points per inch, and the proportion of green in the image was enhanced in order to highlight the presence of copper. The areas were calculated using the Geographical Information System Software gvSIG 1.10 (GVSIG Association, 2011).

In the first stage, various characteristics of the samples (deviation of the grain, number of rings, percentage of heartwood, radial dimension and tangential dimension) were analyzed in order to verify that there was no bias in the treatments (as indicated by Barnes and Lindsey, 2009 for specific gravity). The tests selected were the Analysis of variance and the non-parametric Kruskal-Wallis test. This last test was applied in the case of samples which did not meet the requirements of normality (Shapiro-Wilk test) and homoscedasticity (Bartlett test). In the case of significant p-values in the Analysis of variance, the means were separated using Tukey's comparison. The level of significance was set at 5%.

In the second stage, the same statistical tests were applied to determine whether the factor resulting from the treatment bore a relation to the results obtained for mechanical properties.

Finally, given the significant increase in mechanical properties of the treated woods compared to the control specimens, it was decided to examine the relation between this increase and the amount of protector applied to each sample by means of Regression analysis. This was only done for the samples tested for compression strength, due to the fact that in the bend test the strain is a complex combination of tensile and compressive strengths on a impregnated irregular-serrated cross section.

Results

Table 1 shows the results of the statistical analysis for the detection of bias in the randomization of the

Table 1. Results of the statistical analysis for the detection of bias in the random selection of samples

Block variable	Shapiro-Wilk test (*p*-value)	Bartlett test (*p*-value)	Analysis of variance (*p*-value)	Kruskal-Wallis test (*p*-value)
Grain slope	1.34 × 10-11[(1)] 1.05 × 10-11[(2)] 5.88 × 10-11[(3)]	0.025	—	**0.630**
Number of rings	0.016[(1)] 0.207[(2)] 9.18 × 10-4[(3)]	7.03 × 10-6	—	**0.445**
% heartwood	4.36 × 10-9[(1)] 2.75 × 10-9[(2)] 3.77 × 10-8[(3)]	0.013	—	**0.186**
Radial dimension	0.910[(1)] 0.393[(2)] 0.222[(3)]	0.042	—	**0.997**
Tangential dimension	0.041[(1)] 0.147[(2)] 0.729[(3)]	0.277	**0.632**	—

[(1)] LOSP, n = 40. [(2)] C-A, n = 50. [(3)] Control samples, n = 40. n = number of specimens. Grain slope, number of rings, % heartwood and radial dimension were analyzed with the Kruskal-Wallis test due to the heteroscedasticity detected in the Bartlett test.

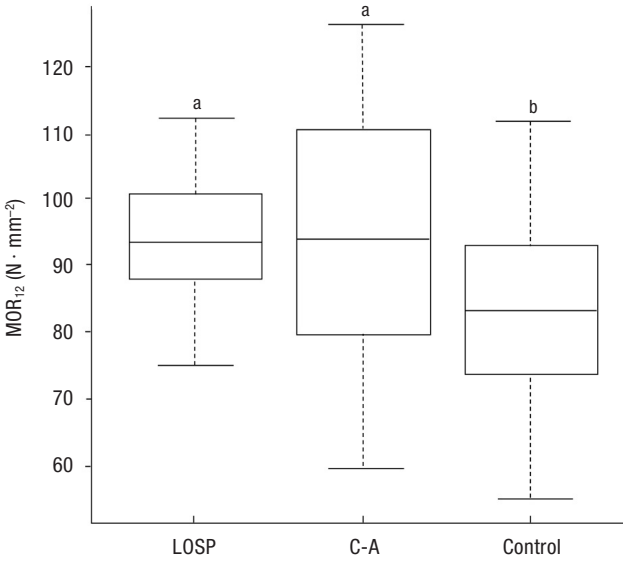

Figure 1. Box Plot graph of estimated bending strength with 12% moisture, MOR12, for each type of treatment. Treatments without a common letter (a, b) are significantly different at $p = 0.05$.

variables in the block. It can be seen that in no case are there any significant differences in the Analysis of variance or in the nonparametric Kruskal-Wallis test. It can therefore be assumed that the samples were correctly chosen. The average retentions obtained in the samples were 15 kg · m^{-3} in the case of C-A and 106 kg · m^{-3} for LOSP.

Static bending strength was analyzed by means of the Kruskal-Wallis test due to the presence of the marked heteroscedasticity in the distribution (the p-values of Bartlett's test of the MOR_m and the MOR_{12} were 0.0026 and 6.1×10^{-4} respectively). Although this test is more conservative than the Analysis of variance, the p-values obtained from Kruskal-Wallis clearly indicate the influence of the treatment on bend strength, and are below 0.01 (4.05×10^{-4} in the case of MOR_m and 0.0055 in MOR_{12}). The box graph corresponding to the MOR_{12} is shown in Fig. 1. The basic values of the distribution are shown in Table 2. The graph and the distribution of the MOR_m are very similar, due to the negligible differences in moisture content between the pieces after the conditioning period (95% confidence interval is between 12.18% to 12.80%).Both Fig. 1 and Table 2 show the increase in the MOR_{12} in the treated woods (both with LOPS and with C-A) compared to the control specimens. The increase in the values of the means and the medians is greater than 13%. This increase compared to the control specimens is practically the same for both treatments.

In the case of the modulus of elasticity to static bending (MOE), the Analysis of variance was applied due to the fact that the sample correctly meets the conditions of normality and homoscedasticity. The *p*-value was highly significant. Fig. 2 and Table 2 show the results obtained for the MOE depending on the treatment applied. Tukey's comparison indicates there are three differentiated non-overlapping groups, the group of samples of C-A above the rest, the group of LOSP in an intermediate situation, and the control group below the others (Fig. 2). Table 2 shows that the woods treated with LOSP have a mean MOE which is 14.3% greater than that of the control specimens; and in the case of woods treated with C-A, this figure is 24.8% greater.

Compression strength parallel to the grain was analyzed using the Analysis of variance, as the data exceeded the Shapiro-Wilk normality test and Bartlett's variance test. In both the case of the value at the test moisture (C_m) and at the estimated 12% moisture value (C_{12}), the p-values were highly significant: 7.2×10^{-10} in C_m and 2.5×10^{-11} in C_{12}. We can confirm that there is a clear relationship between the treatment and compression strength parallel to the grain. The type of

Table 2. Results of the statistical analysis of the MOR_{12}, MOE and C_{12} for each type of treatment

Mechanical property	Tratment	Average (N mm^{-2})	SD (N mm^{-2})	n	Shapiro-Wilk test (*p*-value)	Bartlett test (*p*-value)	Kruskal-Wallis test or ANOVA (*p*-value)
MOR_{12}	LOSP	94.1	9.99	40	0.133	6.1 × 10-4	0.0055 (kw)
	C-A	94.3	17.57	50	0.057		
	Control	83.1	17.97	40	0.153		
MOE	LOSP	7,647	1,095	40	0.292	0.104	1.7×10^{-6} (an)
	C-A	8,345	1,525	50	0.292		
	Control	6,688	1,339	40	0.445		
C_{12}	LOSP	47.8	6.03	40	0.297	0.086	2.5×10^{-11} (an)
	C-A	52.3	7.65	50	0.159		
	Control	40.1	8.63	40	0.243		

SD: standard deviation. n = number of specimens. kw: Kruskal-Wallis test. an: ANOVA.

influence is shown in Fig. 2 and Table 2. This figure shows three different non-overlapping situations. On the one hand, the treated woods are more resistant than the control specimens, and on the other hand, the woods impregnated with C-A present significantly higher results than those treated with LOSP. Table 2 shows a 19.2% increase in the average values of C_{12} compared to the control samples in the case of LOSP, and a 30.4% increase in the case of C-A.

The Regression analysis between the penetration and C_{12} was done only with the samples treated with C-A, given that the samples treated with LOSP had total impregnation. The result showed that the relation is practically negligible, as can be seen in Fig. 3. The pattern of dots does not allow an acceptable fit with any type of curve or straight line. The low value of R^2 (0.015 in the linear case, similar to other non-linear regressions tested), indicates that the percentage of transversal surface area impregnated is not related to the increase in parallel compression strength. Only 1.5% of the behavior of C_{12} could be explained by means of the penetration of the product. Fig. 3 also includes the corresponding Chi-plot, which clearly shows that most cases are located in the central band, demonstrating the independence of the two variables. Therefore, if the greater compression strength of the treated woods does not depend on the degree of impregnation, the increased strength could be due to the changes in pressure experienced during treatment, which do affect the whole of the test batch.

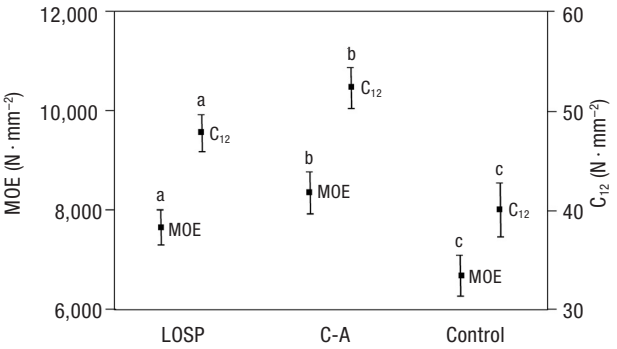

Figure 2. Confidence intervals to 95% for the MOE and C_{12}. Treatments without a common letter (a, b, c) are significantly different at $p = 0.05$.

Discussion

Results agree with the findings of Barnes and Lindsay (2009), and coincide in that the values of the modulus of rupture in wood treated with organic products PTI and with CA-B are greater, although statistically significant results were not achieved. The author himself invited further discussion as to whether these differences were merely statistical or the result of the treatments. The results obtained for bending (MOR and MOE in *Pinus sylvestris*) differ from Yildiz *et al*. (2004), who did not observe any significant differences between the control samples and the woods treated with C-A Tanalith E-3491. The most likely explanation is that the greater number of samples per variable makes it easier to observe the differences (40, as opposed to 10 in the works of Yildiz *et al.,* 2004). Similarly, these results do not concur with the findings of Simsek *et al*. (2010) for *Pinus sylvestris*. The variation in the results could be explained by the different chemical substance —environmentally-friendly borates— used for treatment. Indeed, these authors found

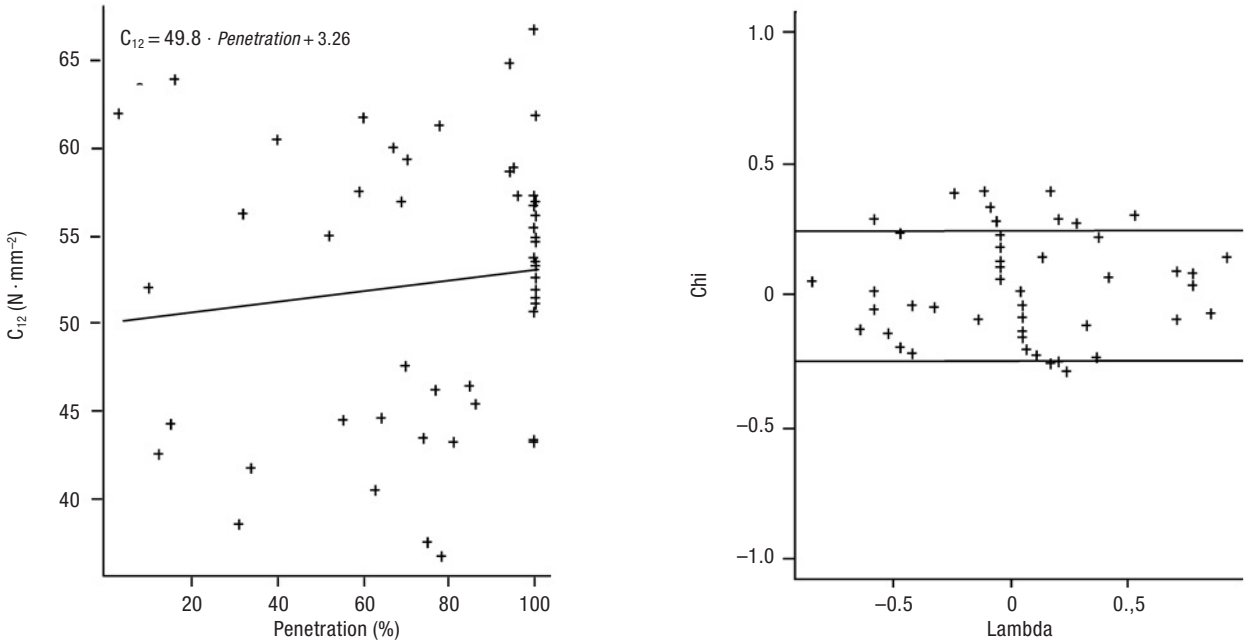

Figure 3. Scatter plot and Chi-plot showing the relation between the amount of protector (percentage of transversal surface impregnated compared to total transversal surface) and estimated parallel compression strength with 12% moisture (C_{12}).

a greater variation in mechanical properties as the concentration increased.

It should also be noted that some of the results do not concur with the findings reported in Forest Products Laboratory 2010, as instead of detecting similar values for MOE and parallel compression in conjunction with the decreases in bending strength, all cases point to significant and considerable increases. Similarly, the values of the water-based protector are higher than those obtained in the organic-based product, contrasting with the indications of the Forest Products Laboratory, 2010. It is essential to take into account that the studies on the mechanical properties of treated woods use two different types of control samples as a reference: untreated wood and wood treated with water. The use of wood treated only with water is widespread, and the results it offers are often not significant (Barnes *et al.,* 2005; Barnes *et al.,* 2008; Barnes *et al.,* 2009). In contrast, the studies by Barnes and Lindsay (2009) using untreated control samples as a reference did detect significant increases in the MOR and MOE, coinciding with the results obtained in the present study.

The decrease in mechanical resistance in wood treated with waterborne preservatives is commonly attributed to the chemical reactions between the wood and the protective products (Forest Products Laboratory, 2010; Winandy, 1995; Yildiz *et al.,* 2004; Simsek *et al.,* 2010). The results obtained in the present work contradict this explanation. No relationship was detected between the penetration of the product and the resistance to parallel compression. The alteration in the wood explained by the changes in pressure and vacuum during the treatment is a more convincing explanation. The significant decrease in mechanical resistance (MOE studied in Barnes *et al.,* 2009) may be partly due to the application of vacuum and pressure treatments. The use of VAC-VAC treatment (without pressure) in the present study could be the reason for the significant increase in mechanical resistance.

Conclusions

VAC-VAC treatment of wood with Light Organic Solvent Preservative or with waterborne Copper Azole significantly increases its resistance to static bending, its MOE and its compression strength parallel to the grain.

The negligible relationship between waterborne preservative penetration and parallel compression strength ($R^2 = 0.015$) indicates that this is not due to the presence of protective products to the cell wall, but could be explained by changes in vacuum pressure during treatment. The significant decrease in mechanical properties observed in previous studies may be partly caused by the use of pressure treatments.

It is also important to take into account the type of control sample. The use of wood treated only with water and submitted to a process of vacuum and pressure identical to the treated samples is more likely to produce non-significant results.

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