Resinous Wood of *Pinus pinaster* Ait.: Physico-mechanical Properties

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Pinus pinaster Aiton is the pine with the largest natural area of distribution in Spain and the species that sustains the country's resin industry, with an annual average production of 3.2 to 3.5 kg per tree. After trees have been tapped, their wood has a high resin content and is of little use because of machining difficulties. For the first time, resinous wood of this species was characterized to compare its physico-mechanical properties with those of non-resinous wood. Significant differences were found in all the properties studied except modulus of elasticity. The resin produced by tapping decreased swelling, probably by reducing accessibility to the –OH groups and decreasing the available spaces during the capillary condensation phase. Similarly, tapping caused an increase in wood density and therefore in hardness, at the same time improving the mechanical properties.

Keywords: Resinous wood; Pinus pinaster; Physico-mechanical properties; Tapping

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INTRODUCTION

Pinus pinaster Aiton is a conifer of the western Mediterranean area and the Atlantic zone of southwest Europe that forms forests in France, Spain, Portugal, Italy, Morocco, Algeria, and Tunisia, occupying an area of 4.2 million hectares (Sanz *et al.* 2006) (Fig. 1).

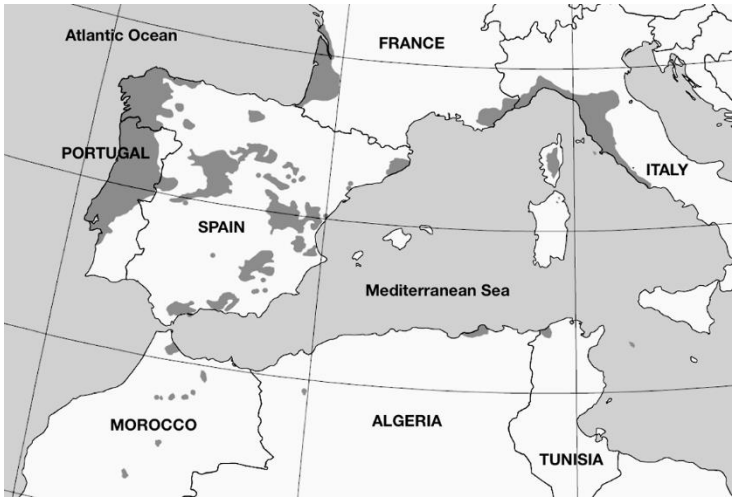

Fig. 1. Distribution map of *Pinus pinaster* Ait. (EUFORGEN, Alía and Martín 2009)

In Spain, it is the pine with the largest natural area of distribution and the most frequently used species in reforestation. It is distributed in more than 30 provinces in mainland Spain, from sea level to 1,500 meters. The species occupies large areas in Galicia and the Central, Iberian, and Baetic mountain systems, and it is also widely distributed in the northern submeseta. Currently, it occupies approximately 1.4 million hectares (SECF 2010), 750,000 of which are considered natural forest (Alía and Martín 2009). Like other Spanish pines, *P. pinaster* wood has been harvested for various uses. This species sustains the resin industry in Spain, with an average annual production of 3.2 to 3.5 kg per tree per tapping season (March to November) (Pinillos *et al.* 2009). The resin sector has recovered in recent years, increasing from a production of 1,821 tons in 2010 with a value of 1.1 million euros to 6,968 tons with a value of 7.3 million euros in 2012. Production is mostly centered in the province of Segovia (5,097 tons per year), well ahead of Valladolid (457 tons per year), Cuenca (401 tons per year), León (395 tons per year), Soria (295 tons per year), Ávila (275 tons per year), and Salamanca (48 tons per year) (MAGRAMA 2012).

The process for obtaining resin, which is based on inflicting light wounds (notches) on the tree throughout the year, lasts five years on a single face of the trunk. When tapping has been completed on a face, a new face is started next to the previous one, and the process continues for five more years. In total, five tapping faces are made in a 25-year period.

This process devalues the wood from the first log, where the tapping is performed. The wood in this area is highly resinous, making machining difficult. However, the increased resin content in the wood as a result of tapping causes a major change in the physical properties of the wood, such as an increase in density, as well as changes in the chemical properties (Ruel *et al*. 1998; Phillips and Croteau 1999), including improving the tree's natural protection from xylophagous agents (Berryman 1972; Croteau *et al*. 1987; Franceschi *et al*. 2005; Knebel *et al*. 2008; Kim *et al*. 2010; Rodríguez-García *et al.* 2014). Although studies have been conducted on the physico-mechanical characteristics of *Pinus pinaster* from Spanish forests (Gutierrez and Plaza 1976; Martínez 1992), none have addressed the first resinous logs or compared the results to non-resinous wood. The increased extractives in the wood after tapping can be a positive modification of the physical and mechanical properties of resinous wood.

The accessory substances of the cell wall can modify the mechanical behavior of wood in two ways: by acting as an inert mass in relation to the cell wall matrix structure, or by affecting hygroscopicity and therefore swelling (Hernández 2007). Some studies have associated wood extractives and their influence with fracture parameters (Petterson and Bodig 1983). In addition, resin incrustation in the cell lumen may act as a transmitter of efforts from one tracheid to another, helping the wood achieve a higher mechanical response, as occurs with synthetic polymers artificially included inside the cell lumen (Li 2011). The objective of this study was to obtain the physico-mechanical characteristics of the resinous wood of *Pinus pinaster* for the first time, using small, defect-free specimens, and compare the results with non-resinous wood of the same species.

EXPERIMENTAL

Collection and Preparation of the Materials

Ten trees were collected in the municipality of Navas de Oro in the Province of Segovia, Spain, five with resinous wood and five with non-resinous wood. All were more than 90 years of age.

The trunks were radially sawn at a height between 1.5 and 3 m to obtain boards 40 mm thick, which were air-dried to 18% moisture content. After discarding the first 30 growth rings to remove juvenile wood, square section strips measuring 35 × 35 mm were obtained from the boards and conditioned in a chamber at 20 ± 2 ºC and 65 ± 5% relative humidity. The final defect-free test pieces, with a cross-section of 20 × 20 mm, were prepared following the UNE 56528 standard (AENOR 1978a).

Physico-mechanical Evaluation

To determine impact bending strength, the instrumented Charpy method was applied using a CEAST Dart drop-weight tester (Norwood, MA, USA) and the DAS4000 data acquisition program (Fig. 2(a)). The tests for static bending strength, compressive strength parallel to the grain, hardness, tensile strength perpendicular to the grain, and splitting were conducted in a Microtest brand universal testing machine (Madrid, Spain) with load cells of 5,000 and 50,000 N and class 1 (Fig. 2(b) and (c)).

The moisture content of the wood was calculated after each test, following the standard UNE-EN 13183-1 (AENOR 2002, 2003, 2004). The equipment used to determine moisture content, density, and test piece dimensions comprised a COBOS brand CB-600 balance (Barcelona, Spain) with a range of 0 to 600 g and 0.01-g scale division, a Memmert brand D06836 oven (Schwabach, Germany) with forced air circulation capable of maintaining a temperature of 103 ± 2 °C, and a Mitutoyo brand Digimatic digital caliper (Aurora, IL, USA) with a range of 0 to 300 mm and 0.01-mm scale division.

All equipment was calibrated, and the uncertainties complied with the general technical competence requirements for testing laboratories in the standard UNE-EN ISO/IEC 17025 (AENOR 2005), and the testing standard requirements.

Charpy impact strength

A hammer with a mass of 11,000 g and a drop height of 1,000 mm was used. The test piece dimensions were 20 × 20 × 300 mm (T × R × L). A striking tup with a 1.5-mm radius was chosen. This parameter has a strong influence on the energy transmitted (Tanaka *et al.* 1995). The distance between the supports was 240 mm. In each test piece, a 45° notch was created to a depth of 2 mm, with a 0.25-mm radius along the base, following the specifications of the standard Charpy test (ASTM D256-05 2005). For the velocity, the recommendations of Kalthoff (1996) were taken into consideration. The response was fitted by reducing the force of impact until the right graph was achieved. The Charpy testing method was chosen because the support system at the two ends of the test piece removes the effect caused by clamping in the Izod method (McCowan *et al.* 2000).

Deflection during testing was determined using Eq. 1,

$$s(t) = \int_0^t v_o \, dt - \left(\frac{1}{m}\right) \int_0^t F(t) dt \quad (1)$$

where s is the test piece deformation at the point of impact (m), v_0 is the initial pendulum velocity (m/s), m is the pendulum weight (kg), F is the load (N), and t is the time interval from the initial moment when the load is applied to the test piece (s).

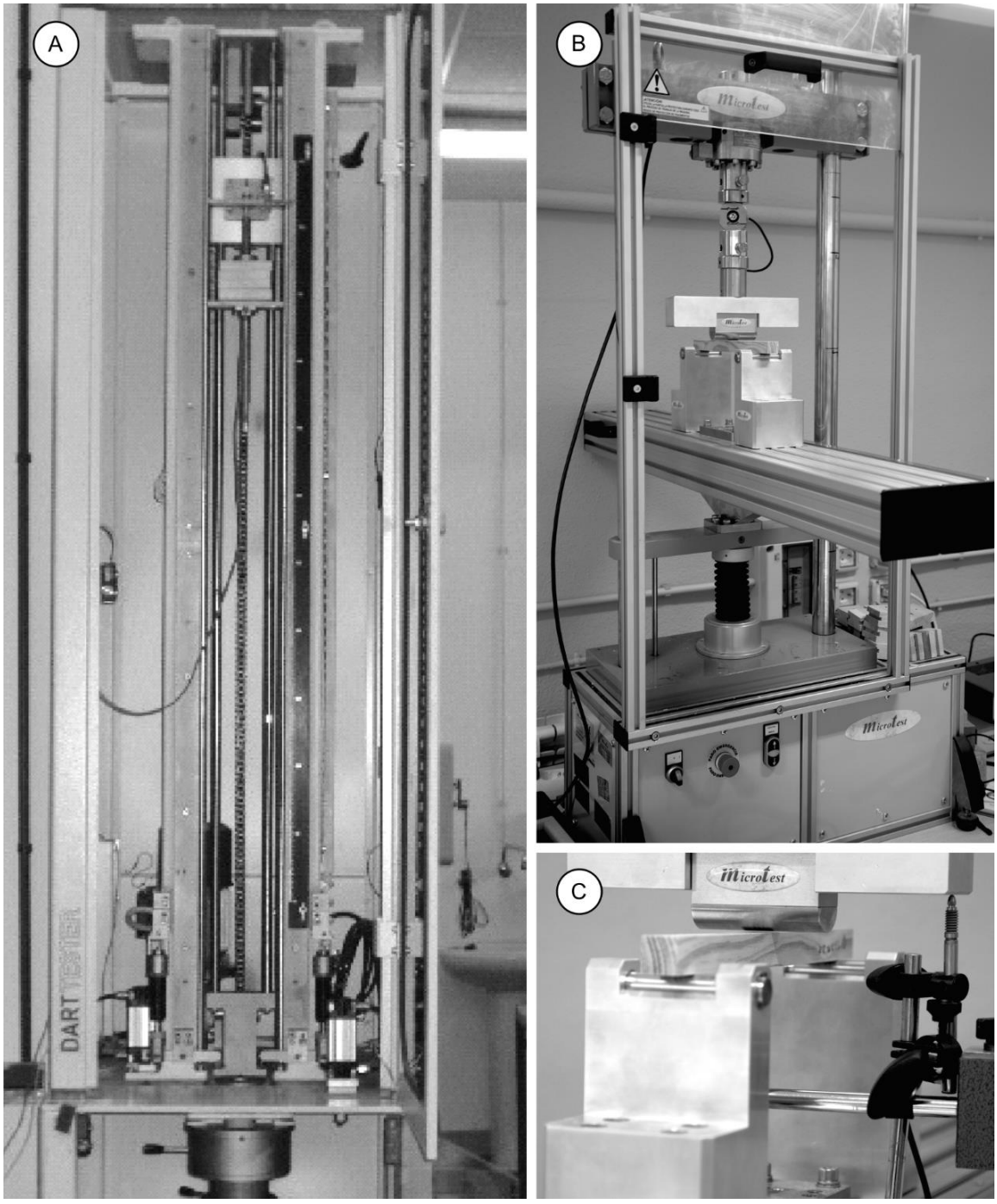

Fig. 2. Testing machines. (A) Dart tester drop-weight machine; (B) universal testing machine; and (C) Linear Variable Differential Transformer (LVDT) for measuring deformations during the static bending test

The energy consumed until a specific deformation occurred was calculated using Eq. 2:,

$$W_{(s)} = \int_0^s F(s)ds \quad (2)$$

Static bending strength

Static bending strength was determined following the standard UNE 56537 (AENOR 1979). Test piece dimensions were 20 × 20 × 300 mm. The modulus of rupture was calculated using Eq. 3,

$$MOR = \frac{3PL}{2bh^2} \quad (3)$$

where *MOR* is the modulus of rupture (N/mm^2), *P* is the breaking load (N), *L* is the distance between supports (mm), *b* is the test piece width (mm), and *h* is the test piece height (mm).

The modulus of elasticity was calculated using Eq. 4,

$$MOE = \frac{L^3(P_2 - P_1)}{4bt^3(a_2 - a_1)} \quad (4)$$

where *MOE* is the modulus of elasticity (N/mm^2), $P_2 - P_1$ is the load increase in the linear section of the load-deformation curve (N) (P_1 corresponds to 10% of the maximum load value and P_2 corresponds to 40%), *L* is the distance between supports (mm), *b* is the test piece width (mm), *h* is the test piece height (mm), and $a_2 - a_1$ is the increase in deformation in the half of the length of the test piece corresponding to $P_2 - P_1$.

Tensile strength perpendicular to the grain

The tensile strength perpendicular to the grain was determined following the standard UNE 56538 (AENOR 1978b) using Eq. 5 on two series of test pieces, one in the radial direction and the other in the tangential direction. Test piece dimensions were 20 × 20 × 70 mm.

$$\sigma_T = \frac{P}{F} \quad (5)$$

σ_T is the tensile strength perpendicular to the grain (N/mm^2), *P* is the breaking load (N), and *F* is the cross-sectional area of the test piece (mm^2).

Splitting strength

Splitting strength was determined following the standard UNE 56539 (AENOR 1978c) using Eq. 6. Test piece dimensions were 20 × 20 × 70 mm.

$$\sigma_S = \frac{P}{b} \quad (6)$$

where σ_S is the splitting strength (N/mm), *P* is the breaking load (N), and *b* is the test piece width (mm).

Compressive strength parallel to the grain

The standard used for compressive strength parallel to the grain was UNE 56535 (AENOR 1977d). Test piece dimensions were 20 × 20 × 60 mm. The strength value was calculated using Eq. 7,

$$\sigma_C = \frac{P}{F} \quad (7)$$

where σ_C is the compressive strength (N/mm^2), P is the breaking load (N), and F is the cross-sectional area of the test piece (mm^2).

Hardness

The hardness test was conducted following the standard UNE 56534 (AENOR 1977c). Test piece dimensions were 20 × 20 × 60 mm. The hardness and the indentation mark were calculated using Eqs. 8 and 9,

$$N = \frac{1}{f} \quad (8)$$

$$f = 15 - 0.5\sqrt{900 - a^2} \quad (9)$$

where N is the hardness (mm^{-1}), f is the deformation (mm), and a is the indentation mark width (mm).

Swelling

The swelling test was conducted following the standard UNE 56533 (AENOR 1977b). Test piece dimensions were 20 × 20 × 60 mm. Volumetric shrinkage was calculated using Eq. 10,

$$C_V = \frac{V_S - V_0}{V_0} x100 \quad (10)$$

where C_V is the total volumetric shrinkage in percentage, V_S is the saturated volume (cm^3), and V_0 is the anhydrous volume (cm^3).

Density

Density was obtained following the standard UNE 56531 (AENOR 1977a), using Eq. 11. Test piece dimensions were 20 × 20 × 25 mm.

$$\rho = \frac{W}{V} \quad (11)$$

where ρ is the density (g/cm^3), W is the test piece weight (g), and V is the test piece volume (cm^3).

Statistics

To study the normality of the data, standardized skewness and kurtosis statistics were used. If any statistic was not within the range of acceptance for a normal distribution, the data were transformed using the logarithmic function, and normality was obtained for all transformed data.

Significant differences between wood types were determined by performing a least significant difference (LSD) test using the ANOVA test data. Statistical calculations were performed in Centurion XV software (Statgraphics, Warrenton, VA, USA) with a confidence level of 95%.

RESULTS AND DISCUSSION

The results obtained in this study showed significant differences between the properties of the resinous and non-resinous wood analyzed, except in MOE (Table 1). Tapping was found to cause changes in the chemical composition of the cell wall, and this affected the physical and mechanical properties of the wood.

The higher density of resinous wood compared with non-resinous wood is a result of the tree's defense processes. The repeated wounding of the tree during the 25 years of tapping causes the permanent activation of its defense mechanisms. While wounding generates both axial and radial traumatic resin canals, it also increases resin production through the metabolic route from the ray parenchyma cells to the axial tracheid lumen through the cross-field pits. The combination of these two processes results in the artificial resinification of the wood, increasing the wood density (Esteban *et al.* 2005).

Table 1. Physical and Mechanical Properties of Resinous and Non-resinous *Pinus pinaster* Wood

Property	Resinous *P. pinaster* $\bar{x} \pm \sigma$ (range)	Non-resinous *P. pinaster* $\bar{x} \pm \sigma$ (range)
Density (g/cm^3)	0.618 ± 0.096 [a] (0.482–0.912)	0.461 ± 0.084 [b] (0.339–0.686)
Swelling (%)	11.01 ± 1.80 [a] (6.18–15.81)	13.55 ± 1.83[b] (9.23–19.73)
Hardness (mm^{-1})	2.50 ± 0.63[a] (1.43–4.63)	2.11 ± 1.12[b] (0.84–6.18)
Charpy impact strength (J)	1.84 ± 0.33[a] (1.32–2.56)	1.56 ± 0.18[b] (1.22–1.98)
Static bending strength, MOR (N/mm^2)	66.48 ± 11.30[a] (44.71–90.27)	58.3 9 ± 10.28[b] (21.44–82.59)
Static bending strength, MOE (N/mm^2)	6121.13 ± 1166.98[a] (4115.82–8146.68)	5970.45 ± 1062.79[a] (3426.81–9144.41)
Tensile strength perpendicular to the grain (tangential) (N/mm^2)	1.83 ± 0.34[a] (0.95–2.69)	1.63 ± 0.30[b] (0.69–2.53)
Tensile strength perpendicular to the grain (radial) (N/mm^2)	2.13 ± 0.34[a] (1.42–2.92)	1.86 ± 0.32[b] (0.89–2.86)
Splitting strength (N/mm)	15.42 ± 2.72[a] (8.79–22.04)	13.69 ± 2.11[b] (8.37–22.76)
Compressive strength parallel to the grain (N/mm^2)	40.68 ± 5.62[a] (27.12–53.96)	34.49 ± 4.72[b] (19.08–46.31)

Note: Different superscript letters indicate statistically different values ($p < 0.05$).

The difference in swelling behavior between the two types of wood can be explained by increased impregnation of the cell wall by resins during resinification. This results in a decrease in the cell wall fiber saturation point caused by the resin occupying the intermicellar spaces (Trendelenburg 1939), which probably causes a decrease in accessibility to the –OH groups during monolayer and multilayer sorption and a decrease in the spaces available for formation of the meniscus during the capillary condensation phase.

The hardness values in the resinous wood were also higher than in the non-resinous wood, confirming the strong correlation with density (de Palacios *et al*. 2008). Dumail *et al.* (1998) supported this conclusion by showing that density is a good predictor of hardness.

All the mechanical properties are strongly correlated with wood density (Kollmann and Côté 1968; Perneståll *et al.* 1995; Haygreen and Bowyer 1996; Hernández 2007). Luxford (1931) showed that extractives strengthen the wood structure and therefore the mechanical properties, whereas Arganbright (1971) reported that extractives do not influence the MOR of *Sequoia sempervirens* Endl. Badran and El-Osta (1977) and El-Osta *et al.* (1981) similarly reported that an increase in extractives had no effect on the mechanical properties, but according to other authors, these properties decreased (Arganbright 1971).

Despite these discrepancies, it is logical that as a result of a higher cellular metabolism and a subsequently high resin discharge, the resulting increase in density would lead to an increase in the mechanical properties of the resinous wood.

However, in addition to the influence of density there is the contribution of resin incrustation in the cell lumen, which may be similar to the reinforcement produced by vinyl monomers in wood composite polymers, *e.g.* Baysal *et al*. (2007), which considerably improve mechanical properties.

In this study, the higher response of the MOR can be explained by the increase in the density of the wood. In contrast, MOE is one of the few wood properties that has little relation to density (Anon 1980), and some authors (Arganbright 1971) have reported that MOE decreases when the extractives in the wood increase. No significant differences were found in this study.

With regard to the Charpy impact response, de Palacios *et al.* (2008) determined that there was a strong relationship between the impact response in wood and the density of the wood, as the response increases with increasing density. Their findings concur with the results obtained in this study, as the resinous wood had higher density and a better impact response.

The results for resistance to compressive strength parallel to the grain in the resinous wood compared with the non-resinous wood concurred with those obtained by Hernández (2007) for hardwoods, indicating that density is associated with increased accessory substances of the cell wall and that these substances positively affect the compressive properties of wood. However, these results differ from those obtained by Badran and El-Osta (1977), who confirmed the lack of correlation between extractives and compressive strength parallel to the grain, although they attributed this conclusion to the high variability in their results. In a later study, El-Osta *et al*. (1981) similarly found no relationship between extractives and compressive strength parallel to the grain in *Tectona grandis* L.f.

The discrepancies among the studies are probably caused by the different location of the extractives in the wood structure. Whereas some extractives remain in the interior of

the cell wall, others can be discharged into the cell lumen, considerably increasing the density of the wood.

The values of tensile strength perpendicular to the grain (tangential and radial) and splitting strength were also significantly different in the two types of wood. The higher values in the resinous wood are related to its higher density, as demonstrated by other authors (Kollmann and Côté 1968).

The resinous wood of *Pinus pinaster*, the result of tapping, has better physical and mechanical characteristics than its non-resinous wood. If the machining and blunting problems caused by the high resin percentage are solved, this type of wood could have new applications.

CONCLUSIONS

1. Resinous wood exhibited less swelling than non-resinous wood because of the resin occupying the intermicellar spaces and probably as a result of the decrease in the accessibility of the –OH groups.
2. Resin incrustation in the cell lumen and the subsequent increase in wood density resulted in the improved physico-mechanical characteristics of resinous wood, except in terms of MOE, where the differences were not significant.
3. The discrepancies among studies that have attempted to relate the physical and mechanical characteristics of wood to extractives content are probably caused by the presence or absence of extractives in the cell lumen.

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