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Original article

Density and dynamic modulus of three mexican woods impregnated with boron. Evaluation with transverse vibrations

Densidad y módulo dinámico de tres maderas mexicanas impregnadas con boro. Evaluación con vibraciones transversales

Densidade e módulo dinâmico de três madeiras mexicanas impregnadas com boro Avaliação com vibrações transversais

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ABSTRACT

The objective of this work was to evaluate the effects of boron salts on the physical-mechanical properties of wood. For this, forty specimens of *Spathodea campanulata*, *Fraxinus americana* and *Albizia plurijuga* were treated using the hot-cold bath method in three groups with concentrations of 1 %, 2 %, 3 %, plus a control group. Before and after the treatment, the densities of the wood, the moisture content, its retentions were calculated and its dynamic modules were determined with transverse vibrations. Retention values increased proportionally to the concentration of boron salts used, but decreased as the densities of each species varied. The treatment in small specimens did not alter the densities of the wood. In contrast, the treatment did modify the dynamic modules determined with transverse vibrations. The paradigm in wood sciences that proposes the characterization of the mechanical behavior of each particular species was confirmed. The experiences were carried out in the Wood Mechanics Laboratory of the Faculty of Engineering in Wood Technology of the Michoacana University of San Nicolás de Hidalgo, in Morelia, Mexico.

Keywords: *Albizia plurijuga*, boron salt retention, *Fraxinus americana*, hot-cold bath, *Spathodea campanulata*.

RESUMEN

El objetivo de este trabajo fue evaluar los efectos de las sales de boro en las densidades y en los módulos dinámicos de la madera. Para ello se trataron cuarenta probetas de *Spathodea campanulata*, *Fraxinus americana* y *Albizia plurijuga* mediante el método de baño caliente-frío en tres grupos con concentraciones de 1 %, 2 %, 3 %, más un grupo de control. Antes y después del tratamiento, se calcularon las densidades de la madera, el contenido de humedad, sus retenciones y se determinaron sus módulos dinámicos con vibraciones transversales. Los valores de las retenciones aumentaron proporcionalmente a la concentración de sales de boro utilizada, pero disminuyeron a medida que las densidades de cada especie variaron. El tratamiento en probetas de pequeñas dimensiones no alteró las densidades de la madera. En cambio, el tratamiento sí modificó los módulos dinámicos determinados con vibraciones transversales. Se confirmó el paradigma en ciencias de la madera que propone la caracterización del comportamiento mecánico de cada especie en particular. Las experiencias se realizaron en el laboratorio de Mecánica de la Madera de la Facultad de ingeniería en Tecnología de la Madera de la Universidad Michoacana de San Nicolás de Hidalgo, en Morelia, México.

Palabras clave: *Albizia plurijuga*, baño caliente-frío, *Fraxinus americana*, retención de sales de boro, *Spathodea campanulata*.

RESUMO

O objetivo deste trabalho foi avaliar os efeitos dos sais de boro sobre as densidades e módulos dinâmicos da madeira. Para este fim, quarenta espécimes de Spathodea campanulata, Fraxinus americana e Albizia plurijuga foram tratados pelo método de banho quente-frio em três grupos com concentrações de 1 %, 2 %, 3 %, mais um grupo de controlo. Antes e depois do tratamento, foram calculadas as densidades da madeira, o teor de humidade, as suas retenções e os seus módulos dinâmicos com vibrações transversais. Os valores das retenções aumentaram proporcionalmente à concentração de sais de boro utilizados, mas diminuíram à medida que as densidades de cada espécie variaram. O tratamento em pequenos exemplares não alterou as densidades da madeira. No entanto, o tratamento modificou os módulos dinâmicos determinados com vibrações transversais. Foi confirmado o paradigma nas ciências da madeira que propõe a caracterização do comportamento mecânico de cada espécie em particular. As experiências foram realizadas no Laboratório de Mecânica da Madeira da Faculdade de Engenharia Tecnológica da Madeira da Universidade Michoacana de San Nicolás de Hidalgo, em Morelia, México.

Palavras-chave: Albizia plurijuga; banho quente-frio; Fraxinus americana; retenção de sais de boro; Spathodea campanulata.

INTRODUCTION

The daily use and practical application of wood required its protection against occasioned deterioration by biological and/or physical agents (Cruz, 2010; Simsek *et al*., 2010). The treatment with boron salts are an efficient practice to protect wood of fungi and insects attack, as well as to diminish deterioration, occasioned by its fire exposition (Tondi *et al*., 2012; Keskin and Mutlu, 2017). This strategy is considered a tendency inside sustainable chemical technology (Simsek and Baysal, 2015; Pizzi, 2016). However, it is necessary to know the effects of preserved substances in physical and mechanical properties of wood.

Lahtela *et al*., (2014) report that, generally, preserved substances do not modify the wood elastic properties. However, the authors remark the empirical evidence existing in regard to contradictory results. The differences could be attributed mainly to the studied species, type and preserved concentration used, as well as temperature, pressure and treatment time.

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Percin *et al*., (2015) inform that elastic module of *Quercus petraea* woods increases or decreases according to type of boron salts used, in arrangement with the intensity and duration of thermic treatment used. On the other hand, Simsek and Baysal (2015) refer, for *Fagus orientalis* and *Pinus sylvestris*, the increase of wood density and at the same time the decrease of elastic module according to the species in relation to the quantity of boron salt retention. This parameter varies for each wood. These results confirm the Kartal *et al*., (2008) observations, *Cryptomeria japonica* wood and Adanur *et al*., (2017), for *Fagus orientalis* wood, which consider that all results are particular to each investigation.

Sotomayor and Villaseñor (2016a) and Sotomayor and Ávila (2019), Sotomayor Castellanos (2016) determine boron salt retention in concentrations of 1 %, 2 % y 3 %, with hot-cold bath method in different woods. Their results suggest the capacity of studied woods to retain boron salt depends on, mainly, the species and it decreases in relation to its density.

About densities and dynamic modules, Sotomayor and Correa (2016), use effort waves to analyze the boron salts effect with a concentration of 3 %, in *Guazuma ulmifolia*, *Spathodea campanulata* and *Abies religiosa*. The authors conclude that these woods could be impregnated with boron sales with hot-cold bath method without reduction of their physical and mechanic properties. The authors propose that it's necessary to characterize the physic-mechanic behavior of wood with an approach of experimentation of case by case of particular species.

Besides, Sotomayor and Villaseñor (2016b), use transverse vibrations to verify the boron sales effects with a concentration of 3 %, in the density and dynamic module of species *G. ulmifolia*, *S. campanulata* and *A. religiosa*. Sotomayor *et al*., (2018), do the same with the species and referred concentration, but this time with static flexion tests. The authors conclude that each species behaves in different way when it is impregnated with boron sales by hot-cold bath process.

In the referred works it is measured parameters such as concentrations and boron sales retentions, as well as its effects on density and elastic module, the last one determined mainly in static conditions. However, the results don´t show a clear tendency respected to increase or decrease of density and elastic module. The differences among previous investigations state that the results are related to variables and test conditions, particularly to the species and retention of reagents. That´s why these authors propose to characterize the mechanic behavior of wood with approach of experimentation of case by case of a particular species.

The strategy of referred works guides the experimental approach of the present investigation, so it is necessary to count with data statistically representative in order to propose impregnated processes in industrial levels (Gérardin, 2016). The objective of this investigation is to determine in which way the treatment modify density and dynamic module. It is started from hypothesis that impregnated treatment doesn´t modify those parameters. To verify this proposal, it was impregnated testing small clear piece of wood (test specimen) of *S. campanulata*, *F. americana* and *A. plurijuga* with boron sales through hot-cold bath method, with concentration of 1 %, 2 % and 3 %.

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MATERIALS AND METHODS

It was collected sawed timber of *S. campanulata, F. americana* and *A. plurijuga* in transformation enterprises of forest products Michoacán state, México. Timber did not have structural anomaly, neither growing defects and with it was prepared 40 testing small clear pieces of wood (test specimen) with transverse section of 0,02 m of width, 0,02 m of thickness and 0,4 m of length according to norm ISO 3129: 2012 (International Organization for Standardization, 2012). These dimensions corresponded to radial, tangent and length directions of woody plain. Before and after the impregnated treatment, test specimens were kept in an arrangement cabin with a temperature of 20 °C (± 1 °C) and a relative humidity of air of 65 % (± 2 %), until its weight was constant. The moisture content of the wood was for all samples 10 % on average, so it was considered that the moisture content would not play a role in the treatment studied. In order to avoid excessive liquid flow at the ends of the specimens, their sides were covered with vinyl paint.

Before the hot-cold bath treatment, densities were determined and vibration tests were performed, the results of which were identified as "pre-treatment" (PT). Once the treatment was completed, the samples were stored for three months, under the same temperature and humidity conditions in which they were stabilized. Subsequently, the densities were determined and transverse vibration tests were carried out, the results of which were identified as "after treatment" (TA).

The transverse vibration tests consisted of measuring the natural frequency of the vibration perpendicular to the longitudinal direction of the specimen. The Grindosonic® device was used for this purpose. Figure 1 shows the configuration of the transverse vibration nondestructive tests. The initial elastic impulse was applied at the geometric center of the upper face of the specimen, in the direction tangential to it, supported by two rigid single-type supports at a nodal distance of 0.224 L. The dynamic test on each test piece was repeated three times and the average values were considered for further analysis. During the tests, the moments of inertia of the cross section of the specimens corresponding to each test were calculated.

The experiences were carried out in the Wood Mechanics Laboratory of the Wood Technology Engineering Faculty of the Universidad Michoacana de San Nicolás de Hidalgo, in Morelia, Mexico.

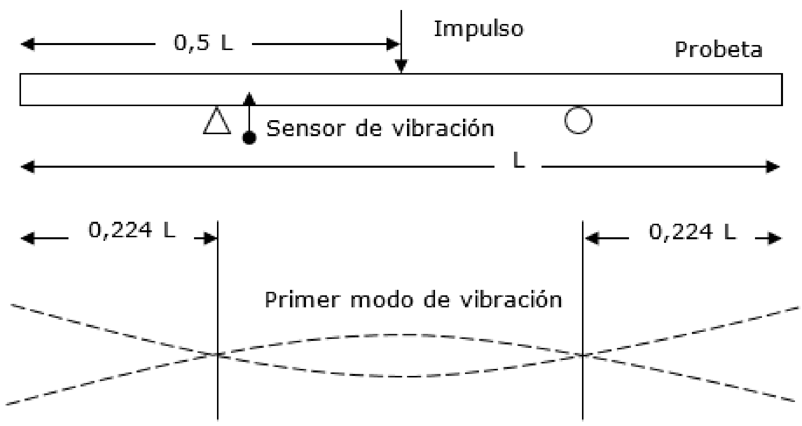

Figure 1. - Vibration test configuration. L = length of the specimen

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For the treatment of impregnation with the hot-cold bath method (Ávila *et al.*, 2012), 30 litres of boron salt solution with a concentration of 3 % were prepared. The mixture consisted of boric acid (39,4 %) and sodium borate (60,6 %). In order to lighten the text, the term treatment will subsequently refer to the impregnation treatment with the hot-cold bath method. The wood was immersed for 8 hours in a water bath with a temperature of 60 °C. The specimens were then immersed for 16 hours in a cold bath with the mixture at a temperature of 23 °C. Subsequently, the retention of the salts was calculated with the formula (1) (Percin *et al.,* 2015) (Equation 1).

$$R = \frac{(P_{DT} - P_{AT}) \times C}{V_{DT}} \qquad (1)$$

Where:

R= Retention (kg m^{-3});
P_{DT}= Weight after treatment (kg);
P_{AT} = Weight before treatment (kg);
V_{DT} = Volume after treatment (m^3);
C = Concentration of the boron salt mixture

The bulk density corresponding to the moisture content of the wood at the time of testing was calculated for each test specimen. The density was calculated with formula (2) according to ISO 13061-2:2014 (International Organization for Standardization, 2014a) (Equation 2).

$$\rho_{CH} = \frac{P}{V} \times 100 \qquad (2)$$

Where:
ρ_{CH} = Density (kg m^{-3}).
P = Weight of the specimen (kg).
V = Volume of the test specimen (m^3).

The moisture content was determined by the difference in weight method according to ISO 13061-1:2014 (International Organization for Standardization, 2014b) with complementary groups of 10 specimens cut from the same batch of wood from which the specimens were made. The dynamic module was calculated using formula (3) (Faydi *et al.,* 2017) (Equation 3).

$$E_{vt} = \frac{4\,\pi^2\,L^4\,f_{vt}{}^2\,\rho_{CH}}{m^4\,r^2} \times \left[1 + \frac{r^2}{l_{vt}{}^2}\,K\right] \qquad (3)$$

Where:

E_{vt} = Dynamic module (N m^{-2});
L = Length of the test tube (m);
l_{vt} = Distance between supports (m);
f_{vt} = Natural frequency (Hz);
ρ_{CH} = Density (kg m^{-3});
m, K = Dimensionless constants (12.65, 49.48);
r = Turning radius of the specimen cross section (m^2);

Con: $r = \sqrt{I/A}$

I = Moment of inertia of the specimen cross section (m^4);
A = Cross-sectional area of the specimen (m^2).

Although the number of samples observed for each species was 40, a sufficient number to consider the large sample theory in the analysis, the sample size needed to validate the statistical tests was calculated a posteriori for an acceptable error of 0.05. This was done for each species and with the values of the retention of the samples corresponding to C = 1 %. This was done for each species and with the values of the retention of the samples corresponding to C = 1 %. In the cases of density and dynamic modulus the values of the control samples were analysed (C = 0 %). The number of samples was calculated with the formula (4) (Gutiérrez y de la Vara, 2012) (Equation 4).

$$n = \frac{2\,\sigma^2}{e^2} \qquad (4)$$

Where:

n = Number of test specimens;
ó = Standard deviation;
e = Acceptable estimation error *(e = 0.05)*

Experimental design

The experimental unit consisted of twelve samples of ten specimens, each corresponding to four concentrations of boron salts for each of the three species: *S. campanulata, F. americana* and *A. plurijuga*. The response variables were retention (R, formula 1), density (ρ_{CH}, formula 2) and dynamic modulus (Evt, formula 3). The impregnation with boron salts was considered the treatment and gives the wood two states: before the treatment (AT) and after the treatment (DT). The boron (C) salt concentration was recognised as the controlled factor and has four levels C = 0 (Control group), C = 1 %, C = 2 % and C = 3 %. A total of 240 observations were made (10 × 4 × 3 × 2 = 240). The moisture content of the wood (CH) was considered a reference variable and was not involved in the experiment. For the response variables the mean ("x”), the standard deviation (ó) and the coefficient of variation $(CV = \sigma/\bar{x})$ were determined. For the densities and dynamic modules the arithmetic variations between the values obtained before and after treatment were calculated:

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$\Delta = [(\bar{x}_{DT}-\bar{x}_{AT})/\bar{x}_{AT}]$. For the average values before and after treatment, linear regressions *(y = ax + b)* and their coefficients of determination (*R*^{2}) were calculated between the dynamic modules and the densities.

RESULTS AND DISCUSSION

Sample size

For the samples of *S. campanulata, F. americana* and *A. plurijuga* the number of samples needed to validate the statistical analysis was lower than the number of samples observed for each species (Table 1). These results assured the validity of the strategy proposed by the experimental design. In particular, the number of test tubes needed to analyze data corresponding to the dynamic modules, which, according to Table 1, was close to but less than the number of observations for each species.

Table 1. - Sample sizes

	Species		
	S. campanulata	*F. americana*	*A. plurijuga*
Variables	n	n	n
R	3	25	9
ρ_{CH}	6	5	1
E_{vt}	33	31	38

R = Retention; ρCH = Density; Evt = Dynamic modulus; n = Number of specimens required per species

Retentions

Average retention values varied from a minimum of 1.3 kg m-3 with a concentration of 1 % for *S. campanulata* to a maximum of 6.4 kg m-3 with a concentration of 3 % for *A. plurijuga* (Table 2). Their coefficients of variation averaged 6.4 % with a minimum of 4.6 % for *S. campanulata* with a concentration of 1 % and a maximum of 16.8 % also corresponding to *S. campanulata* (C = 2 %) (Table 2).

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Table 2. - Densities, dynamic modules and retentions

specie		ρCH AT	Evt AT	C	R	ρCH DT	Evt DT
		(kg m-3)	(MN m-3)	(%)	(kg m-3)	(kg m-3)	(MN m-3)
S. campanulata	x	351	7145	Control	0	351	6723
CH = 8,2 %	CV	6,3	14,4	-	-	6,4	15,5
	x	363	6805	1	2,3	355	7366
	CV	7,4	17,4	-	4,6	7,1	15,8
	x	343	7539	2	4,0	342	7926
	CV	3,7	13,0	-	16,8	3,3	12,2
	x	347	7004	3	6,4	348	7387
	CV	7,8	12,1	-	6,4	6,7	12,6
Averages		351	7218	-	3,2	349	7446
F. americana	x	654	11754	Control	0	658	11708
CH = 9,5 %	CV	5,6	14,0	-	-	5,7	13,9
	x	650	13552	1	1,6	647	11797
	CV	4,4	11,9	-	9,5	4,4	10,4
	x	659	11878	2	3,3	656	11991
	CV	7,3	17,6	-	6,3	7,6	17,4
	x	643	12208	3	5,3	645	12434
	CV	5,3	12,9	-	10,2	5,1	12,0
Averages		652	12348	-	2,6	652	11983
A. plurijuga	x	830	11450	Control	0	830	11487
CH = 11,7 %	CV	2,7	15,4	-	-	2,7	15,9
	x	845	11935	1	1,3	841	11951
	CV	3,2	13,1	-	9,8	3,3	13,6
	x	849	11900	2	2,7	846	11855
	CV	3,2	17,1	-	5,9	3,3	17,0
	x	827	11532	3	3,7	827	11603
	CV	4,6	11,0	-	7,3	4,7	11,2
Averages		838	11704	-	1,9	836	11724

ρCH = Density; Evt = Dynamic modulus; AT = Before treatment; DT = After treatment; C = Concentration; R = Retention; CH = Moisture content; "x" ¯= Average; CV = Coefficient of determination in percent

The magnitudes of the retentions obtained in this research were of the same order as those reported in the literature. In particular, they were similar to those of Sotomayor and Avila (2019), who used the same material and treatment as in the present study. Even so, differences between species and types of tests were observed to determine the dynamic modules. In relation to the densities of the three species, the dispersions of the retentions decreased as the densities decreased. This suggests that average values tend to cluster and decrease (Figure 2). Likewise, the dispersions of the dynamic modules did not indicate any trend in relation to the retentions (Figure 3).

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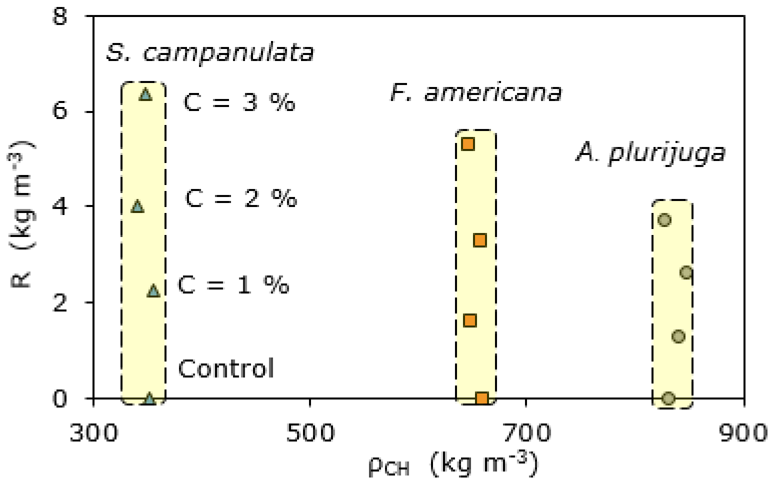

Figure 2. - Retentions (R) grouped in relation to densities *(ρ_{CH})*

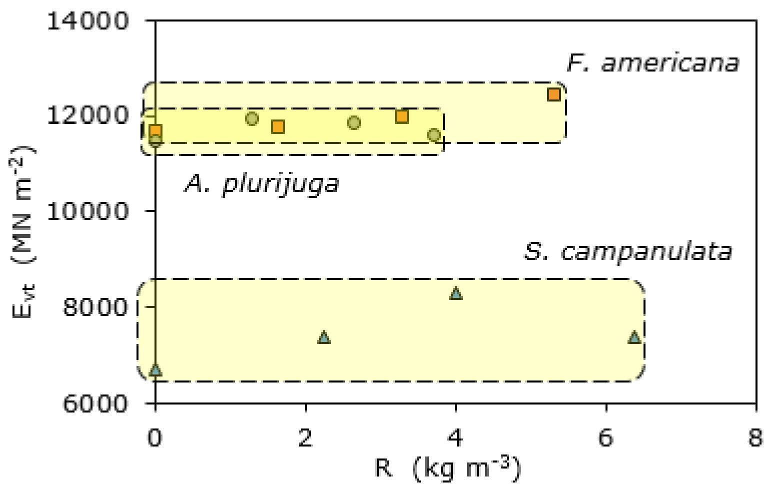

Figure 3. - Dynamic modules *(E_{vt})* grouped in relation to the retentions (R)

Densities

The magnitudes of the densities before and after treatment place *S. campanulata* wood with the lowest density, *F. americana* with intermediate values and *A. plurijuga* with the highest (Table 2). The arithmetical differences tabulated in Table 3 were negative and less than the percentage unit. In addition, the three spacings averaged -0.28 %, which is why it is considered that the treatment does not modify the wood densities. These results were similar to those reported by Sotomayor and Ávila (2019), since, as with the data concerning retentions, it is the same material and treatment. In the same order of ideas, the coefficients of variation of the densities varied in average 5 %, values congruent to those reported in the bibliography (Tamarit and López, 2007; Silva *et al.*, 2010).

Table 3. - Arithmetic differences for densities and dynamic modules

Species	C	$\Delta\rho_{CH}$	ΔE_{vt}
	(%)	(%)	(%)
S. campanulata	Control	-0,01	-5,20
	1	-2,02	8,51
	2	-0,42	5,23
	3	0,31	5,47
Average	-	-0,53	3,50
F. americana	Control	0,21	-0,38
	1	-0,46	-11,64
	2	-0,48	1,03
	3	0,25	2,05
Average	-	-0,12	-2,24
A. plurijuga	Control	0,04	0,31
	1	-0,50	0,08
	2	-0,30	-0,37
	3	0,00	0,61
Average	-	-0,19	0,16

C = Concentration; ΔρCH = Arithmetic difference density; ΔEvt = Arithmetic difference dynamic module.

The values of the retentions are aligned with those of the densities (Figure 2). Furthermore, the correlations between the densities, the dynamic modules and their determination coefficients (Figure 4 and Figure 5), indicate that density is a good predictor of dynamic modules. This applies to all three species, the three concentrations and the control group, as well as the two wood states, i.e. before and after treatment. These corollaries coincide with those recently reported by Sotomayor (2019) for Mexican woods.

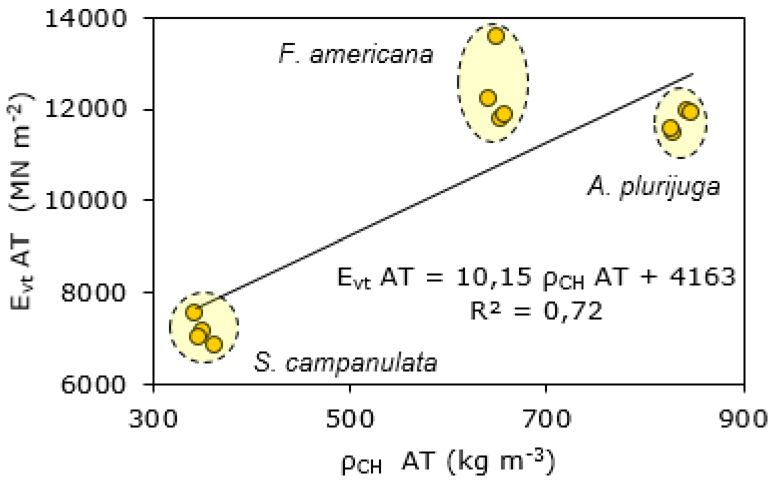

Figure 4. - Dispersions, correlations and coefficients of determination *(R^2)* between dynamic modules *(E_{vt})* and densities *(ρ_{CH})* before treatment *(AT)*

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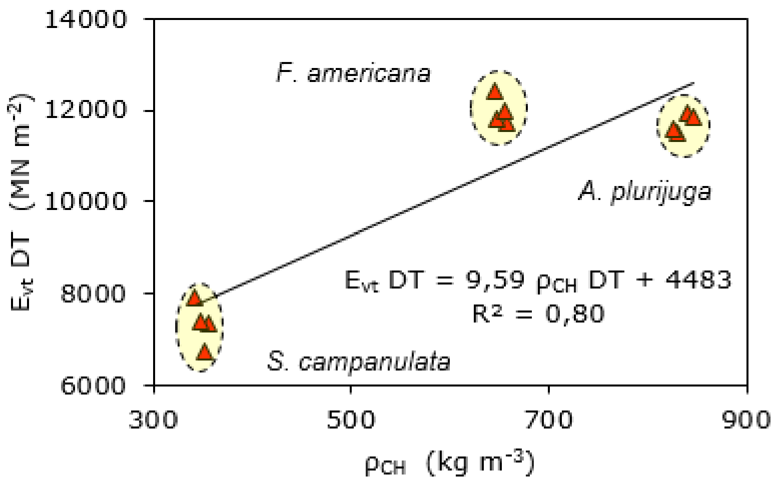

Figure 5. - Dispersions, correlations and coefficients of determination *(R^2)* between dynamic modules *(E_{vt})* and densities *(ρ_{CH})* after treatment *(DT)*

Dynamic modules

The average magnitudes of the dynamic modules of *S. campanulata*, *F. americana* and *A. plurijuga* listed in Table 2 were of the same order as those reported by Sotomayor (2019). The average dynamic modules of *F. americana* were higher than those of *S. campanulata* and *A. plurijuga*. Thus, while the arithmetic differences between the values before and after treatment (Table 3) were positive for *S. campanulata* and *A. plurijuga*, those corresponding to *F. americana* decreased. As a result, the average of the differences for all the samples observed was 0.16 %, a calculation difficult to interpret since the effect of the treatment on the dynamic modules reached a maximum value of 8.51 for S. campanulata (C = 1 %) and a minimum value of -11.64 % for *F. americana* (C = 1 %), that is, an interval of 20.15 %.

The coefficients of variation for the dynamic modules (before and after treatment) averaged 14.1 % (Table 2) and were not affected by the concentrations and/or retention for each sample observed. The dispersions of the dynamic modules are grouped indifferently to the retentions (Figure 3). However, the average values of the four samples corresponding to each species and concentration (Control, C = 1 %, 2 % and 3 %) correlate well with the densities before treatment (Figure 4) and regularly after treatment (Figure 5).

In the hot-cold bath treatment, the retention values increase proportionally to the concentration of boron salts used, but decrease as the densities of each species increase.

The treatment modifies the dynamic modules determined with transversal vibrations, these differences are of the order of 3,5 % for *S. campanulata*, of -2,24 % for K and of 0,16 % for *A. plurijuga*.

The average values of the densities of the three woods, before and after treatment, are good predictors of the dynamic modules.

The hot-cold bath is efficient to impregnate with boron salts woods of *S. campanulata*, *F. americana* and *A. plurijuga.*

Thanks

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The authors have participated in the writing of the work and analysis of the documents.