Article

Addition of boron to pine veneers and ist effect on plywood properties

Adição de boro às lâminas de pinus e seu efeito nas propriedades de compensados

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Abstract

This work evaluated the influence of impregnation of boron compounds, in three concentrations, on *Pinus* sp. veneers, and its effect on the properties of plywood. The experiment was carried out in a completely randomized design, in a 3 × 3 + 1 factorial scheme, with three boron compounds (boric acid, borax, and disodium octaborate tetrahydrate) and three solution concentrations (1%, 3%, and 5%) plus the control. The panels were produced with three or five veneers, depending on the test, impregnated by dipping in boron compound solutions for 15 min and bonded with urea-formaldehyde adhesive. The apparent density, water absorption, thickness expansion, hygroscopicity, resistance to static bending (parallel and perpendicular to the grain) and shear strength were evaluted. All tests were carried out following the European Committee for Standardization, and the results were compared to the Brazilian Association of Mechanically Processed Wood requirements. Based on the results, it is recommended to use boric acid at a concentration of 1% or 3% or the other compounds tested at a concentration of 1%.

Keywords: Apparent density. Shear strength. Static bending. Thickness selling. Water absorption.

Resumo

Objetivou-se avaliar a influência da impregnação de compostos de boro, em três concentrações, em lâminas de Pinus sp., e seu efeito nas propriedades de compensados. O experimento foi realizado em delineamento inteiramente casualizado, em esquema fatorial 3 × 3 + 1, sendo três compostos de boro (ácido bórico, bórax e octaborato dissódico tetrahidratado) e três concentrações (1%, 3% e 5%) mais o controle. Os painéis foram produzidos com três ou cinco lâminas, encolados com uréia-formaldeído, com as lâminas impregnadas por imersão nas soluções de compostos de boro por 15 min. Foram avaliadas a densidade aparente, absorção de água, expansão de espessura, higroscopicidade, resistência à flexão estática (paralela e perpendicular à grã) e resistência ao cisalhamento. Todos os testes foram realizados de acordo com o Comitê Europeu de Padronização. Para o controle de qualidade, os resultados foram comparados com as exigências da Associação Brasileira de Madeira Processada Mecanicamente. Com base nos resultados, recomenda-se usar ácido bórico na concentração de 1% ou 3% ou os demais compostos testados na concentração de 1%.

Palavras-chave: *Densidade aparente. Resistência ao cisalhamento. Flexão estática. Inchamento em espessura. Absorção de água*

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Introduction

According to the Brazilian Association of Mechanically Processed Wood (ABIMCI, 2022), plywood production in Brazil reached 3.08 million m^3 in 2018, with 73,8% exported, in which both pinewood (mainly) and native tropical wood (kapok - *Ceiba pentandra* and paricá - *Schizolobium amazonicum*) were used as raw material. However, because of the good trunk shape of pine, ease of lamination and bonding, and lower environmental concerns, plywood manufacturers have been using its wood since the early 1990s (Almeida *et al*., 2012; Mendes *et al*., 2013; Sanquetta *et al*., 2019). To meet the demand, the planting of *Pinus* spp. forests have increased and currently occupy 1.93 million hectares (19,54% of the total area), concentrated in Southern Brazil. According to the Brazilian Tree Industry (IBA, 2022), pine wood is mainly transformed into lumber and panels, such as plywood. The physical-mechanical properties of plywood are closely related to species used as raw material, including genetic traits. Despite their low durability, softwoods are easier to bond than hardwoods and are favored for their cellular structure, which influences its porosity and permeability, with a clear effect on adhesive bonding (Asante *et al*., 2022; Kollmann; Kuenzi; Stamm, 1975; Zhuang; Cloutier; Koubaa, 2023).

Plywood is considered a multipurpose material since it can be designed for structural and decorative use (Moezzipour *et al*., 2013). However, when produced with less durable veneers, it is necessary to apply chemicals to improve their durability (Demir; Aydin, 2023; Sugahara *et al*., 2022). The chemicals can be applied to veneers by dipping or spraying (for stain and mold fungi); together with the adhesive (for insects), and by vacuum-pressure (for decaying fungi and insects), after manufacturing for outdoor uses.

However, one of the problems related to the use of chemicals for preserving wood composites is the incompatibility of their formulations with industrial adhesives (Alade *et al*., 2022; Aydin; Colakoglu, 2007; Özçifçi, 2006). Thus, they should not impair the physical-mechanical properties of wood after bonding, and consequently the quality of the panel ((Alade *et al*., 2022).

Boron compounds are the most investigated products for this purpose (Lesar *et al*., 2011) and have been used in Australia and New Zealand since the 1930s (Williams, 1996). Because of their diffusibility, they are applied to freshly cut veneers by dipping or spraying (Bagheri *et al*., 2022). These methods allow the diffusion of the product into the wood structure without impairing its physical-mechanical properties (Colakoglu *et al*., 2003; Lebow, 2010; Lesar *et al*., 2011; Wallis, 1970).

In Brazil, the majority of plywoods is manufactured without the application of preservative substances, which becomes a problem, especially when these products are exposed to xylophagous organisms (Nunes *et al*., 2023). Therefore, dry-wood termites attack residential, office, and school furniture frequently. This causes premature replacement, disposal of furniture in unsuitable locations, and the proliferation of these insects. Furthermore, the addition of chemical substances to the glue (to join the veneers) does not prevent termite attacks, as they have the habit of building their galleries horizontally in the veneer, passing from one layer of plywood to another through cracks or bonding imperfections.

Therefore, impregnating the veneers with preservative substances should be practiced, instead of adding them to the glue. Boron compounds were used for this purpose, to be less toxic and more environmentally friendly (Bagheri *et al*., 2022). Even so, research is possible to detect which compounds or concentrations are most effective in controlling xylophagous organisms, without causing damage to the quality of the bond line, and consequently, to the physical-mechanical properties of plywood. The hardwoods previously mentioned have problems with dry-wood and subterranean termites, and coleoborer attacks (*Dinoderus minutus* and *Lyctus* spp.), also needing to be impregnated with boron compounds.

This study aimed to evaluate the influence of impregnation of boron compounds, in different concentrations, on *Pinus* sp. veneers, and its effect on the physical-mechanical properties of plywood.

Material and methods

Collection and treatment of veneers

Pinus sp. rotary-peeled veneers (1.5 mm thickness) were obtained from a company located in Imbituva, Paraná, Southern Brazil, and artificially dried to 6% moisture content (MC) and stored at room temperature.

The veneers were classified according to the presence of knots and cracks, mass, and homogeneously divided among the treatments. Veneers were cut into 60 × 60 cm and dipped, at room temperature (25-30 °C), in different solutions (50 L of water) of boron compounds (Table 1) for 15 min, while the control veneers were dipped in pure water. The immersion time followed Purslow (19874). The adsorption of the preservative solution, for permeable and thin lumber, as the pine veneers used in the research, occurs in the first 5-10 min of immersion or dipping, with a tendency to stabilize after 15 min. According to Purslow (1974), absorption decreases with the square root of the immersion or dipping time. After that, they were stored at room temperature until reaching the equilibrium moisture content (≈ 12%).

The concentrations were established based on literature, since for tropical regions (average temperatures normally exceeding 25 °C), borax should have a concentration of up ≥ to 5% in the solution (Willerding; Vianez, 2003). Above this, precipitation of boron compounds occurs, leading to concentration errors. When necessary, the solutions were homogenized to avoid the precipitation of boron compounds. As the objective of this study was to compare the effect of the different compounds for the three concentrations (Table 1), it was not concerned with the chemical balancing of concentration in the boric acid equivalent (BAE) of each compound.

The mean retentions of BAE, boron element, or boron trioxide (kg of H_3BO_3; B or B_2O_3 per cubic meter of wood) for each treatment was calculated, and its effects on protection against mold and dry-wood termites published (Nunes *et al.*, 2023). However, the addition of chemicals to improve the performance of wood panels to xylophagous organisms can negatively affect some of their physical-mechanical properties.

Plywood manufacturing

Plywood was manufactured in a laboratory on an automatic hydraulic press (SOLAB, SL 12/150, Piracicaba, Brazil), with two electrically heated horizontal plates at 140 °C, with a pressing area of 60 cm × 60 cm. The pressing time and pressure were 10 min and 980 kPa, respectively.

Three veneers were used per panel, but the shear strength test had five veneers. Since the panels were intended for interior use, liquid urea-formaldehyde (UF) adhesive was used, catalyzed with 24% ammonium sulfate [$(NH_4)_2SO_4$], calculated at 2% of the solids content of adhesive, and wheat flour as binder. The glue was formulated at a ratio of 100:40:40 (adhesive: binder: water) and each simple glue line had 340 $g.m^{-2}$. When composing veneers, the fibers of the neighboring veneers were at a 90° angle as European Committee for Standardization - CEN, European Standard - EN 636 (CEN, 2015).

Nine panels (five with three veneers + four with five veneers) were produced per treatment (Table 1), along with the control (untreated). They were stored in a climate-controlled room (25 ± 2 °C and 65 ± 5 % relative humidity - RH) until equilibrium moisture content (≈ 12 %).

Physical-mechanical tests

The MC was determined according to the standard EN 322 (CEN, 1993a), using 20 specimens (50 mm width × 150 mm length) per treatment, four per panel (two parallel and two perpendicular to the grain). The apparent density was determined according to the standard EN 323 (CEN, 1993b), using 20 specimens (50 mm width × 50 mm length) per treatment, four per panel. The water absorption and thickness swelling, both after 2 h and 24 h of water immersion, were determined (Abdul Khalil *et al.*, 2010; Dieste *et al.*, 2008; CEN, 1993c) using 15 specimens (50 mm width × 50 mm length) per treatment, three per panel.

Table 1 – **Solutions used for impregnation of *Pinus* sp. veneers**

Treatment	Concentration (%)	Boron compounds (g)	
		Boric acid	Borax
1	1	500	-
2	3	1500	-
3	5	2500	-
4	1	-	500
5	3	-	1500
6	5	-	2500
7*	1	197	303
8*	3	591	909
9*	5	984	1516

Note: *boric acid: borax (1:1.54), according to the relation between borax and boric acid equivalent (BAE), i.e., the mass of borax/mass of boric acid (× 4): BAE (381.37/247.32 = 1.54). This dry solution produces Polybor, known as Timbor which contains 117.3% of boric acid (BAE) (Richardson, 1993). It is also cited as disodium octaborate tetrahydrate (DOT).

The mechanical tests were carried out on a universal testing machine (EMIC, DL 10000 N/S 8622, São José dos Pinhais, Brazil). The modulus of rupture (MOR) and elasticity (MOE) in bending strength were determined according to the standard EN 310 (CEN, 1993d, 2004), using 20 specimens (50 mm width × 150 mm length) per treatment (10 parallel and 10 perpendicular to the grain), four per panel (two parallel and two perpendicular to the grain), with a 125 mm span and 3 $mm.min^{-1}$ of loading speed. After that, the MC of specimens was measured and the results of MOR and MOE were standardized to ≈ 12% MC according to Equation 1, as described in the standard NBR 7190-1 (ABNT, 2022).

$$F_{12\,\%} = F_{M\,\%}[1 + \frac{3(M_{\%} - 12)}{100}] \quad \text{Eq. 1}$$

Where:

$F_{12\,\%}$ is the standardized resistance to 12 % moisture content (MPa);

$F_{M\,\%}$ is the resistance at M % moisture content (MPa); and

$M_{\%}$ is the moisture content (%).

The shear strength was determined according to the standard EN 314-1 (CEN, 2004), using 12 specimens (25 mm width × 150 mm length) per treatment, three per panel. The specimens were immersed in distilled water for 24 h and tested at 100 kN, using a loading speed of 4 $mm.min^{-1}$. The shearing area was 25 mm × 25 mm. After that, the specimens were classified according to the percentage of apparent cohesive wood failure by comparison. Finally, the plywood was classified regarding bonding quality according to EN 314-1 (CEN, 2004) and EN 314-2 (CEN, 1993e).

Statistical analysis

The experiment was carried out in a completely randomized design in a 3 × 3 + 1 factorial arrangement (boron compounds × solution concentrations, plus control), totaling 10 treatments. Assumption tests were done (Cochran: normality; Shapiro-Wilk: homogeneity of variance, both to $p < 0.05$), and then the data were submitted to analysis of variance (ANOVA) with the F test ($p < 0.05$).

For the initial evaluation of the results, we considered only the factorial scheme, excluding the control. In situations where there was a significant effect of the type of boron compound, the Tukey test ($p < 0.05$) was applied to compare the means. In cases where there was a significant effect of the concentration, regression analysis was applied, using the technique of orthogonal polynomials with decomposition in linear and quadratic effects. Additionally, the means of the treatments were compared to the control by the Dunnett test ($p < 0.05$).

Results and discussion

Physical properties

According to ANOVA, the interaction between the boron compound and concentration was significant (F test; $p < 0.01$) for MC. According to the Tukey test, there was no significant difference among means for boron compounds only at 1% concentration (Table 2).

Table 2 – **Moisture content by treatment**

Boron compound	**Concentration (%) / Moisture content (%)**		
	1	**3**	**5**
Boric acid	9.77 ± 0.13 a	10.55 ± 0.35 a	10.30 ± 0.29 b
Borax	9.80 ± 0.25 a	10.09 ± 0.26 b	10.34 ± 0.26 b
DOT	9.93 ± 0.39 a	10.53 ± 0.25 a	10.67 ± 0.26 a
General mean	9.83 ± 0.26	10.39 ± 0.29	10.47 ± 0.27

Note: Means followed by the same letter in the column do not differ (Tukey test; $p > 0.05$). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

The absolute means of the treatments ranged from 9.77% to 10.67%, respectively for boric acid at 1% and DOT at 5%, which is a small range of 0.90 percentage points (p.p.). Despite the statistical significance between some treatments, the differences in MC were so small that they would not have a practical influence on the performance of the panels. This result also indicates good quality control during the conditioning of panels. The MC negatively influences the gluing of veneers, which can affect the physical-mechanical properties of panels. However, Pipíška *et al*. (2023) did not observe any negative effect of 2%, 5%, and 7% MC on the bending and gluing properties of plywood. Thus, as the difference between the MCs of veneers used was less than 1.0 p.p., this will not negatively influence the properties of panels.

All panels (including the control) met the MC since their means were between 9% and 12% (ABIMCI, 2022). This result reinforces the previous discussion that the statistical differences in MC among the treatments probably would not influence the performance of panels.

Little has been reported about the influence of boron compounds on wood hygroscopicity, equilibrium moisture content, and water absorption. However, it is known that they are considered hygroscopic (Baraúna *et al*., 2020; Kartal; Ayrilmis; Imamura, 2007), besides having good solubility in water and being easily leachable ((Baraúna *et al*., 2020; Jorge; Nunes; Botelho, 2004; Zou *et al*., 2018). The differences in means of different boron compounds at 3% and 5% concentrations may be attributed more to material rather than to compound, as discussed previously, due to the small absolute ranges in the average MCs at the same concentration.

The effect of solution concentration on MC for each boron compound was checked by regression analysis, whose estimated equations are shown in Figure 1. All parameters of the adjusted equations were significant (F test; $p < 0.01$), and the compounds had different behavior according to the concentration.

Despite the different behavior, for all boron compounds, higher MC increases occurred from 1% to 3% borate content, corresponding to 0.78 p.p., 0.29 p.p., and 0.60 p.p., respectively for boric acid, borax, and DOT. From 3% to 5%, the MC decreased (0.25 p.p.) only for boric acid. In the case of DOT and borax, the MCs increased by 0.25 p.p. and 0.14 p.p., respectively. The increase in MC of impregnated veneers (plywood) may have been by the bulking effect, which is caused by the impregnation of some salt solutions into wood (Paes *et al*., 2015).

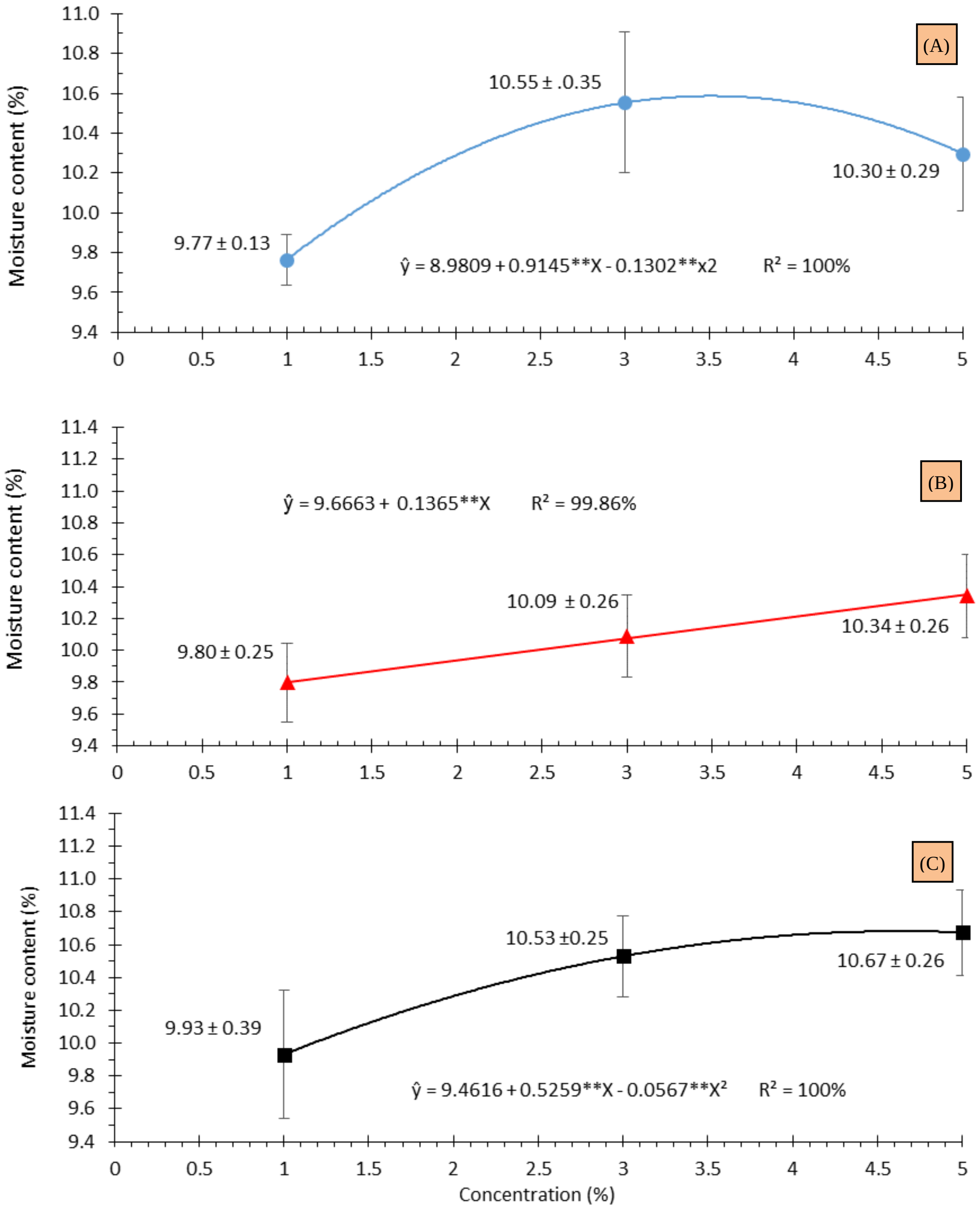
The means of treatments (Table 2) were compared to the control (9.53 %) by the Dunnett test, which indicated that only the mean of boric acid at 1% (9.77%) did not differ significantly ($p > 0.05$). The boron compounds increased wood hygroscopicity (Baraúna *et al*., 2020; Jorge; Nunes; Botelho, 2004; Kartal; Ayrilmis; Imamura, 2007; Wang; Liu; Lyu, 2019). However, the highest range compared to control was 1.14 p.p. (DOT at 5%), which is also difference lower than those previously reported, which were not able to negatively influence the bending and gluing properties of plywood (Pipíška *et al*., 2023). Differences in MC of veneers and panels produced with them are common to occur in panels from the same lot as well, and it is impossible to have 100% rigorous control over the moisture levels of the same batch of veneers or panels produced from them.

According to ANOVA, the interaction between the boron compound and concentration was significant (F test; $p < 0.01$) for apparent density (Table 3). According to the Tukey test, there was a significant difference (F test; $p < 0.05$) between the boron compounds only at 3% concentration for boric acid.

Since the effect of the boron compound was not significant for other concentrations, and no tendency regarding absolute means could be noticed for different concentrations, we suggest that the density of veneers had a stronger influence on apparent density than the boron compound. It happened even despite the classification of the veneers before the panels' manufacturing. Another factor that might have contributed to this non-significant difference was the low retention achieved during the impregnation of the veneers (Nunes *et al*., 2023). Such retention may be related to the impregnation method, as well as by the MC of the veneers at the time of dipping.

Additionally, boric acid and borax have 1.51 $g.cm^{-3}$ and 1.73 $g.cm^{-3}$ density, respectively (Smallwood *et al*., 2004. Since they are similar, we expected the concentration to have a stronger effect on the density of panels than the boron compound, which was not confirmed according to the analysis presented in Figure 2. The impregnation with boron compounds causes an increase in basic and anhydrous density of wood (Augustina *et al*., 2020; Baraúna *et al*., 2017).

Figure 1 - **Effect of boron compound concentration on moisture content**



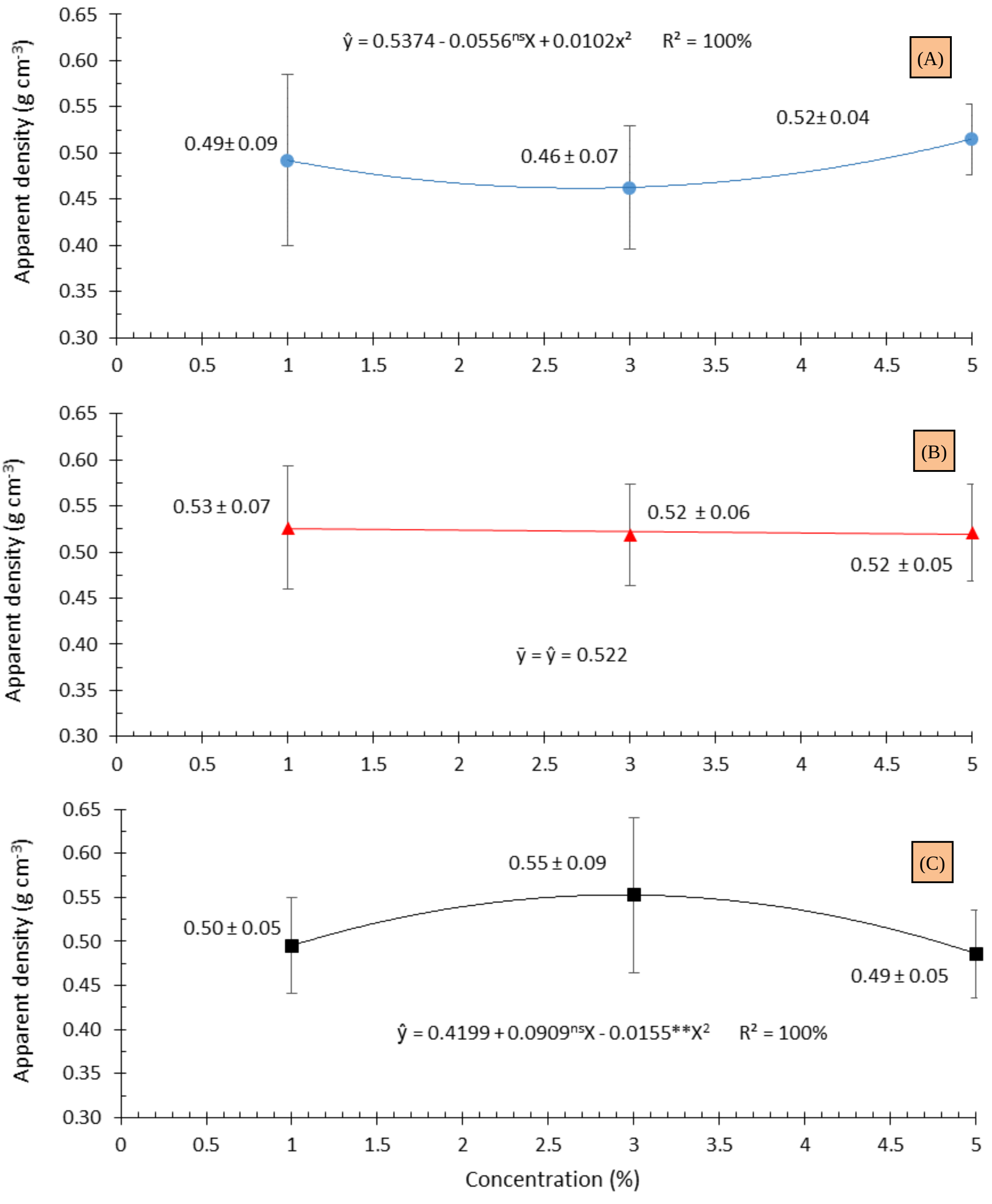
Note: boric acid + borax ≈ DOT (disodium octaborate tetrahydrate). A: Boric acid; B: Borax; C: DOT; ** significant (F test; $p < 0.01$). Mean ± standard deviation.

Table 3 - **Apparent density by treatment**

Boron compound	Concentration (%)/Apparent density ($g.cm^{-3}$)		
	1	3	5
Boric acid	0.49 ± 0.09 a	0.46 ± 0.07 b	0.52 ± 0.04 a
Borax	0.53 ± 0.07 a	0.52 ± 0.06 a	0.52 ± 0.05 a
DOT	0.50 ± 0.05 a	0.55 ± 0.09 a	0.49 ± 0.05 a
General mean	0.51 ± 0.06	0.51 ± 0.07	0.51 ± 0.06

Note: means followed by the same letter in the column do not differ (Tukey test; $p > 0.05$). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 2 - **Effect of boron compound concentration on apparent density. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate)**



Note: A: Boric acid; B: Borax; C: DOT; ^{ns} no significant (F test; p > 0.05); ** significant (F test; p < 0.01). Mean ± standard deviation.

According to regression analysis (Figure 2), the behavior of boron compounds was different in function of concentration, where the effect of concentration on apparent density was not significant for borax ($p > 0.05$). The adjusted parameters of equations were significant (F test; $p < 0.01$) for boric acid and DOT. For boric acid, apparent density declined from 1% to 3% and then increased from 3% to 5%. For DOT the effect was the opposite.

The means of treatments (Table 3) were compared to the control (0.470 $g.cm^{-3}$) by the Dunnett test, which indicated that only the means of boric acid at 1 % (0.527 $g.cm^{-3}$) and DOT at 3% (0.553 $g.cm^{-3}$) were significantly different ($p < 0.05$). The absolute means of treatments ranged from 0.463 $g.cm^{-3}$ to 0.553 $g.cm^{-3}$, respectively for boric acid at 3% and DOT at 3%, which is a small range of 0.090 $g.cm^{-3}$. The overall analysis of results reiterates that the density of veneers (material heterogeneity) had a stronger influence on apparent density than preservative solutions. Also, the low retention values of boron compounds in veneers, which ranged from 0.138; 0.264; and 0.324 kg m^{-3} (boric acid); 0.091; 0.23; 0.369 kg m^{-3} (borax); and 0.115; 0.264; 0.511 kg m^{-3} (DOT), calculated as boric acid equivalent (BAE), respectively for concentrations of 1; 3; and 5 (%) in boron compound solution. These concentrations were effective against dry-wood termites (*Cryptotermes brevis*) and mold fungus *Penicillium* sp. (Nunes *et al*., 2023).

It is noteworthy that none of the retentions obtained reached the minimum of 3.89 kg m^{-3} for Timbor® (≈ DOT), what would be enough to prevent subterranean termite attacks (Jorge; Nunes; Botelho, 2004). This is because the diffusibility obtained by the dipping method is more effective in freshly sawn wood (high MC), which facilitates the diffusion process of boron compounds. As the veneers were previously dried (≈ 10%), their low MC hampered the diffusion of boron compounds (Nunes *et al*., 2023).

However, retentions of 0.8 - 1.0 kg of BAE m^{-3} are required, to prevention against xylophagous organisms (Freeman; McIntyre; Jackson, 2009), including false and true powderpost beetles. For dry-wood termites (*Incisitermes minor*), a retention of 0.533 kg DOT m^{-3} provided 25% of termite mortality (Kartal; Terzi; Yoshimura, 2020). Nevertheless, to achieve 80% or more termite mortality, retention greater than 6.8 kg m^{-3} would be required.

It is appropriate to highlight that, the Brazilian Association of Mechanically Processed Wood (ABIMCI, 2002) has no quality requirements regarding apparent density for plywood manufactured with three veneers. Furthermore, the addition of chemical products, to improve the resistance against xylophagous, should have little or no influence on their physical-mechanical properties, mainly regarding the resistance of bond lines (Nunes *et al*., 2023).

According to ANOVA, the interaction between boron compound and concentration was significant for water absorption (WA) for both 2 h (F test; $p < 0.01$) and 24 h (F test; $p < 0.01$) of immersion (Table 4). For the same concentration, there was no correspondence in the statistical result in both periods evaluated.

For WA2, there was a significant difference ($p < 0.05$) between the means of boron compounds only at 3% concentration, where boric acid had the highest mean (10.1%). The same statistical result occurred for apparent density, but boric acid had the lowest mean (0.463 $g.cm^{-3}$). The water uptake of wood and its derivatives is related to density and therefore to the proportion of voids in the structure (Siau, 1995). Less dense materials tend to absorb more water than denser materials (Brito *et al*., 2020).

For WA24, there was a significant difference ($p < 0.05$) between the means of boron compounds for all concentrations. No clear pattern could be noticed for boron compounds in water absorption. The effect of solution concentration on WA for each boron compound was checked by regression analysis (Figure 3). The same behavior was noticed for WA2 and WA24.

The effect of concentration on WA was not significant (F test; $p > 0.05$) only for DOT. For boric acid, WA increased from 1% to 3% concentration and then decreased at 5%, where the absolute mean of the latter had an intermediate position. For borax, the effect of concentration on WA was rising and linear.

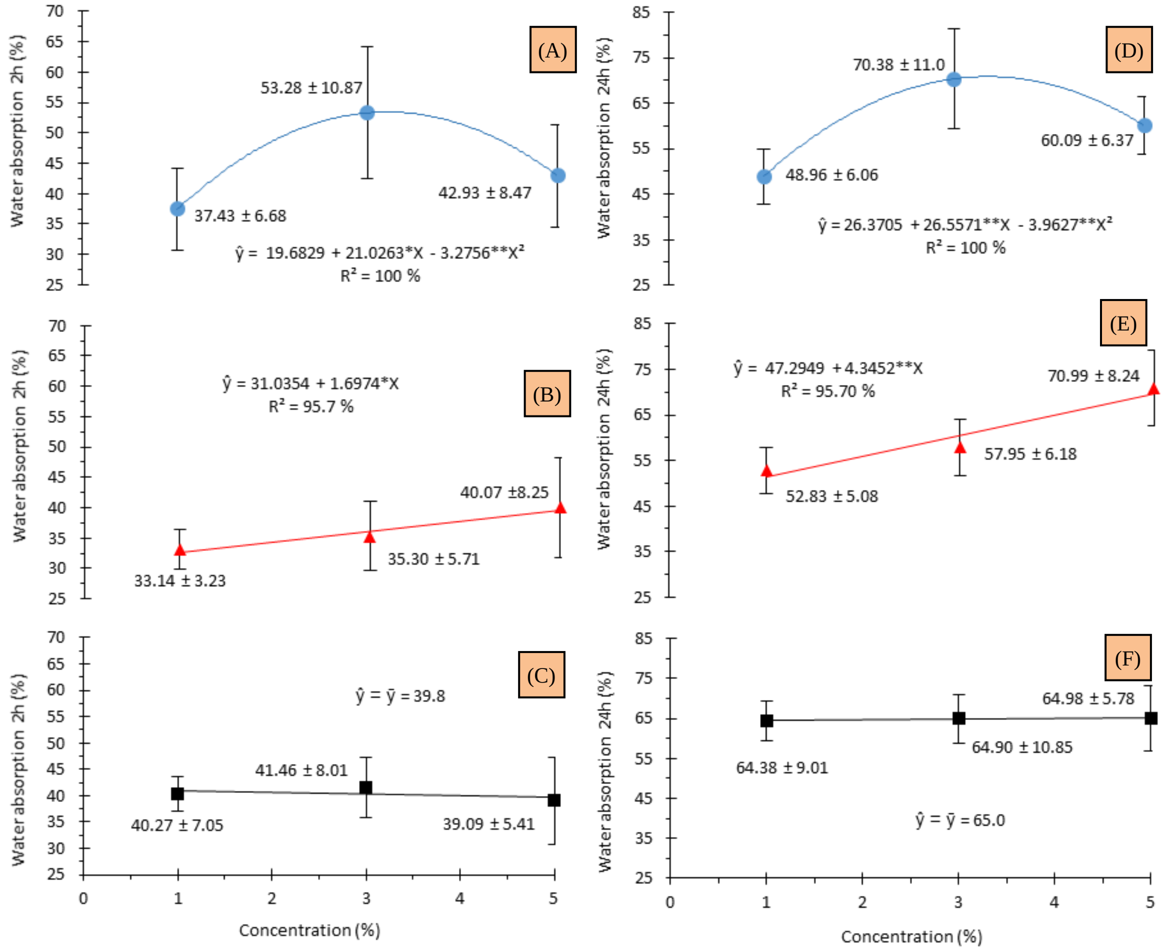
The means of the treatments (Table 4) were compared to the control (45.2%/WA2, 62.7%/WA24) by the Dunnett test. For DOT, there was not a significant difference ($p > 0.05$) compared to control for both WA2 and WA24. The same result was obtained for boric acid at 5% concentration, and borax (5% concentration/WA2 and 3% concentration/WA24). The means of other treatments were significantly different ($p < 0.05$) from the control. For 1% concentration, the means of WA2 and WA24 of boric acid and borax were lower than the control, and the same was true for borax at 3% concentration. This indicates a positive effect of preservative solutions.

Table 4 – **Water absorption after 2h (WA2) and 24h (WA24) of immersion by treatment**

Boron compound	Concentration (%) / Water Water absorption - WA (%)					
	1		3		5	
	WA2	WA24	WA2	WA2h	WA2	WA24
Boric acid	37.43±6.68a	48.96±6.06b	53.28±10.87a	70.38±11.00a	42.93±8.47a	60.09±6.37b
Borax	33.14±3.23a	52.83±5.08b	35.30 ± 5.71b	57.95 ± 6.18b	40.07±8.25a	70.99±8.24a
DOT	40.27±7.05a	64.38±9.01a	41.46 ± 8.01b	64.90±10.85a	39.09±5.41a	64.98±5.78ab
General mean	36.95±5.63	55.39±6.71	43.20± 8.20	64.45±9.72	40.70±7.42	65.74±7.05

Note: means followed by the same letter in the column do not differ (Tukey test; $p > 0.05$). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 3 - **Effect of boron compound concentration on water absorption after 2h (A, B, C) and 24 h (D, E, F) of immersion**



Note: boric acid + borax ≈ DOT (disodium octaborate tetrahydrate). A, D: Boric acid; B, E: Borax; C, F: DOT; * significant (F test; $p < 0.05$); ** significant (F test; $p < 0.01$). Mean ± standard deviation.

A negative effect was noted for boric acid at 3% concentration for both WA2 and WA24, and borax at 5% concentration. For these treatments, the means were significantly ($p < 0.05$) higher than the control. Little is reported about the influence of boron compounds on WA. Some authors affirm that wood treated with inorganic flame retardants (such as boron compounds) tends to be more hygroscopic (Kartal; Ayrilmis; Imamura, 2007), which happened only with two out of nine treatments tested.

According to ANOVA, the interaction between boron compound and concentration was significant for thickness swelling (TS) for both 2 h (F test; $p < 0.01$) and 24 h (F test; $p < 0.01$) of immersion (Table 5).

Only at 3% concentration, the same statistical result was found for both periods evaluated, where borax had the highest means, followed by boric acid and DOT, which did not differ significantly from each other. At 1% concentration, borax tended to have higher TS. At 5% concentration, there was no effect of the boron compound on TS2, but for TS24 boric acid and DOT had the highest means.

The effect of solution concentration on TS for each boron compound was investigated by regression analysis, whose estimated equations are shown in Figure 4. The effect of concentration on TS was not statistically significant (F test; $p > 0.05$) only for DOT, the same behavior as noticed for water absorption test (Figure 3).

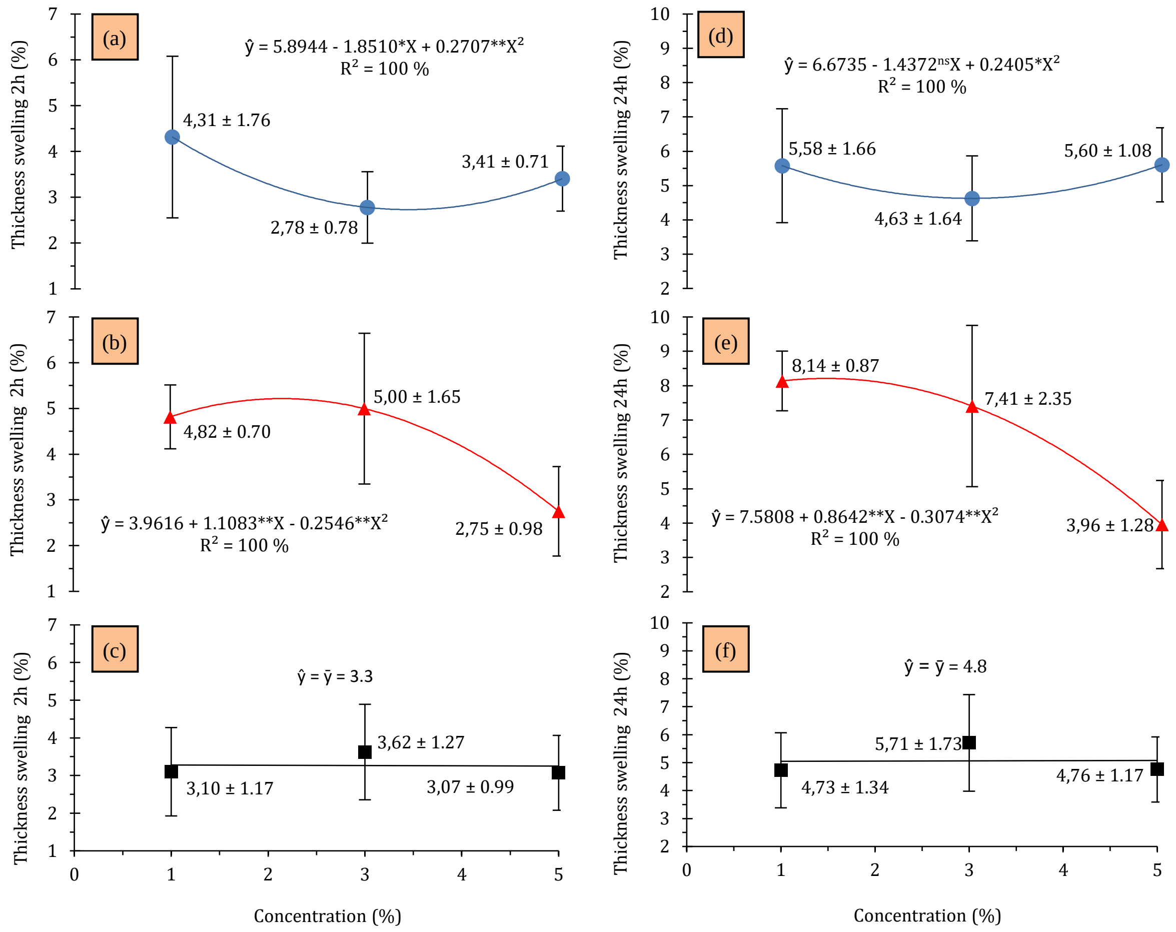
For boric acid, TS2 decreased from 1% to 3% concentration and then increased at 5% concentration, where the latter had an intermediate mean. For TS24, the behavior was slightly different because the mean at 5% was the same as at 1% concentration.

Table 5 - **Thickness swelling after 2h (TS2) and 24h (TS24) of immersion by treatment**

Boron compound	Concentration (%) / Thickness swelling - TS (%)					
	1		3		5	
	TS2	TS24	TS2	TS24	TS2	TS24
Boric acid	4.31±1.76 a	5.58±1.66 b	2.78±0.78 b	4.63±1.24 b	3.41±0.71 a	5.60±1.08 a
Borax	4.82±0.70 a	8.14±0.87 a	5.00±1.65 a	7.41±2.35 a	2.75±0.98 a	3.96±1.28 b
DOT	3.10±1.17 b	4.73±1.34 b	3.62±1.27 b	5.71±1.73 b	3.07±0.99 a	4.76±1.17ab
General mean	4.08±1.26	6.15±1.25	3.78±1.24	5.90±1.80	3.05±0.94	4.71±1.24

Note: means followed by the same letter in the column do not differ (Tukey test; p > 0.05). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 4 - **Effect of boron compound concentration on thickness swelling after 2h (A, B, C) and 24 h (D, E, F) of immersion**



Note: boric acid + borax ≈ DOT (disodium octaborate tetrahydrate). A, D: Boric acid; B, E: Borax; C, F: DOT; ^{ns} no significant (F test; p > 0.05); * significant (F test; p < 0.05); ** significant (F test; p < 0.01). Mean ± standard deviation.

However, for borax, the TS2 slightly increased from 1% to 3% concentration and then had a steep decrease at 5% concentration. For TS24, the behavior was slightly different because the means constantly decreased from 1% to 5% concentration. Nonetheless, for DOT, no effect of concentration on thickness swelling behavior was observed. For most of the situations analyzed, a high standard deviation was observed, when compared to means of observations. This may have been caused by the lack of homogeneity in the mass of analyzed samples, in which the denser ones tend to swell more in thickness when adsorbing water, than those with lower density. This is related to the volume of wood substance and voids per unit area (Siau, 1995).

The means of the treatments (Table 5) were compared to the control (4.3%/TS2, 4.9%/TS24) by the Dunnett test. For TS2, the means of all treatments were significantly lower ($p < 0.05$) than the control or did not differ from it ($p > 0.05$). Boric acid at 3% (2.8%) and DOT at 1% (3.0%) matched the former situation.

Regarding TS24, the means of impregnated wood at any concentration with boric acid and DOT did not differ from the control ($p > 0.05$). The same was true for borax at 5%. However, the effect of borax at 1% and 3% concentrations was negative on TS24, because their means were significantly higher ($p < 0.05$) than the control. These results may be associated with the retention of boron compounds used, the effect of time, and the impregnation mechanism adopted. This is required since the aim of impregnating wood is to improve its resistance to xylophagous, with minimal influence on its physical-mechanical characteristics (Nunes *et al.*, 2023).

Mechanical properties

According to ANOVA, the interaction between boron compound and concentration was significant for bending strength parallel to the grain for both MOE (F test; $p < 0.01$) and MOR (F test; $p < 0.01$). According to the results (Table 6), borax had the highest absolute means of MOE at 1% and 3% concentration, which did not differ significantly from DOT. For 5% concentration, the effect of the boron compound was not significant on MOE (F test; $p > 0.05$), which was the same for MOR at 1% concentration. Borax had the highest mean of MOR at 3% concentration but had the lowest at 5% concentration, whereas DOT had the highest absolute mean (did not differ significantly from boric acid).

The effect of solution concentration on MOE and MOR in bending strength parallel to the grain was checked by regression analysis (Figure 5). For boric acid, MOE and MOR had the same behavior in function of concentration, decreasing from 1% to 3% concentration and then increasing up to 5% concentration, where the latter means were the highest.

For borax, MOE and MOR were the opposite, increasing from 1% to 3% concentration and decreasing up to 5% concentration, where the latter means were the lowest. For DOT, the effect of concentration on MOE was significant, with a constant increase from 1% to 5% concentration. Nonetheless, the effect was not significant for MOR.

The means of the treatments (Table 6) were compared to the control (50 MPa/MOR and 5829 MPa/MOE) by the Dunnett test. The statistical results were the same for MOE and MOR compared to the control for all treatments, except for borax at 5% concentration. For boric acid, the concentrations at 1% and 5% had no effect ($p > 0.05$) on the bending strength. However, the effect was negative at 3%, where the means were lower than the control.

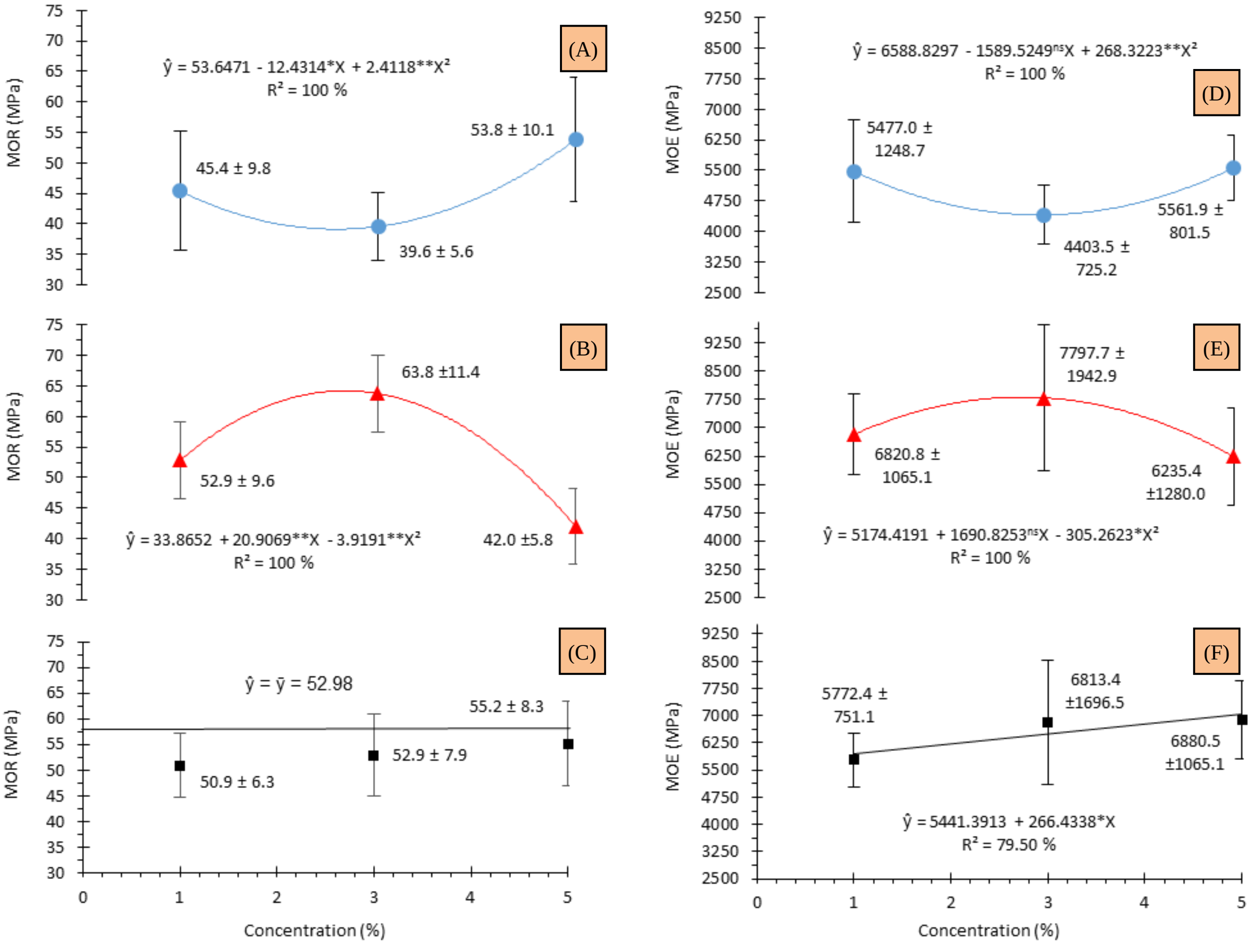
For borax, the concentrations at 1% had no effect ($p > 0.05$) on the bending strength. Nevertheless, the effect was positive at 3%, where the means were higher than the control. Concentration at 5% had different behavior for MOE and MOR, with no significant difference from control for MOE and lower mean for MOR. For DOT, there was no effect of concentration on bending strength parallel to the grain when compared to the control. For most of the situations analyzed, similar to what happened for the thickness swelling (TS), a high standard deviation value was observed. This may have been caused by a lack of homogeneity among analyzed samples, having influenced the MOR and MOE values, regardless of the boron compounds and concentrations used.

Table 6 – **Bending strength parallel to the grain: modulus of rupture (MOR) and elasticity (MOE) by treatment**

Boron compound	Concentration (%) / Bending strength parallel to the grain - MOE and MOR (MPa)					
	1		3		5	
	MOR	MOE	MOR	MOE	MOR	MOE
Boric acid	45.4±9.8a	5477.0±1248.7b	39.6±5.6c	4403.5±725.2b	53.8±10.1a	5561.9±801.5a
Borax	52.9±9.6a	6820.8±1065.1a	63.8±11.4a	7797.7±1942.9a	42.0±5.8b	6235.4±1280.0a
DOT	50.9±6.3a	5772.4± 752.1ab	52.9±7.9b	6813.4±1696.5a	55.2±8.3a	6880.5±1065.1a
General mean	49.73±8.56	6023.4±1021.96	52.1±8.3	6338.20±1454.86	50.33±8.06	6225.93±1048.86

Note: means followed by the same letter in the column do not differ (Tukey test; $p > 0.05$). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 5 - **Bending strength parallel to the grain: effect of boron compound concentration on modulus of rupture - MOR (A, B, C) and elasticity - MOE (D, E, F)**



Note: boric acid + borax ≈ DOT (disodium octaborate tetrahydrate). A, D: Boric acid; B, E: Borax; C, F: DOT; [ns] no significant (F test; $p > 0.05$); * significant (F test; $p < 0.05$); ** significant (F test; $p < 0.01$). Mean ± standard deviation.

The increased concentration of boron compounds negatively affected the physical-mechanical properties of particleboards (Özlüsoylu; Istek; Yalçinkaya, 2017). For medium-density fiberboard (MDF), negative effects on MOR values were found (Ustaomer; Usta, 2012). A similar trend was also reported for *Fagus orientalis* wood, with a decline in mechanical resistance and a rise in retention rates with the increase in concentrations of boron compounds (Fídan; Adanur, 2019). Samples impregnated with borax were more resistant than those impregnated with boric acid. Nevertheless, the impregnation of compounds in the wood veneers did not exert the same pattern of variation in bending strength. In which they did not exert effect positive or negative compared to the control.

The interaction between the boron compound and concentration was significant for bending strength perpendicular to the grain for both MOE (F test; $p < 0.01$) and MOR (F test; $p < 0.05$). The same statistical results were observed for both MOE and MOR for all concentrations of boron compounds tested (Table 7).

At 1% and 5% concentrations, there was no effect (F test; $p > 0.05$) of the boron compound on bending strength perpendicular to the grain. At 3%, DOT had the highest means of MOE and MOR, differing from others, being non-significant.

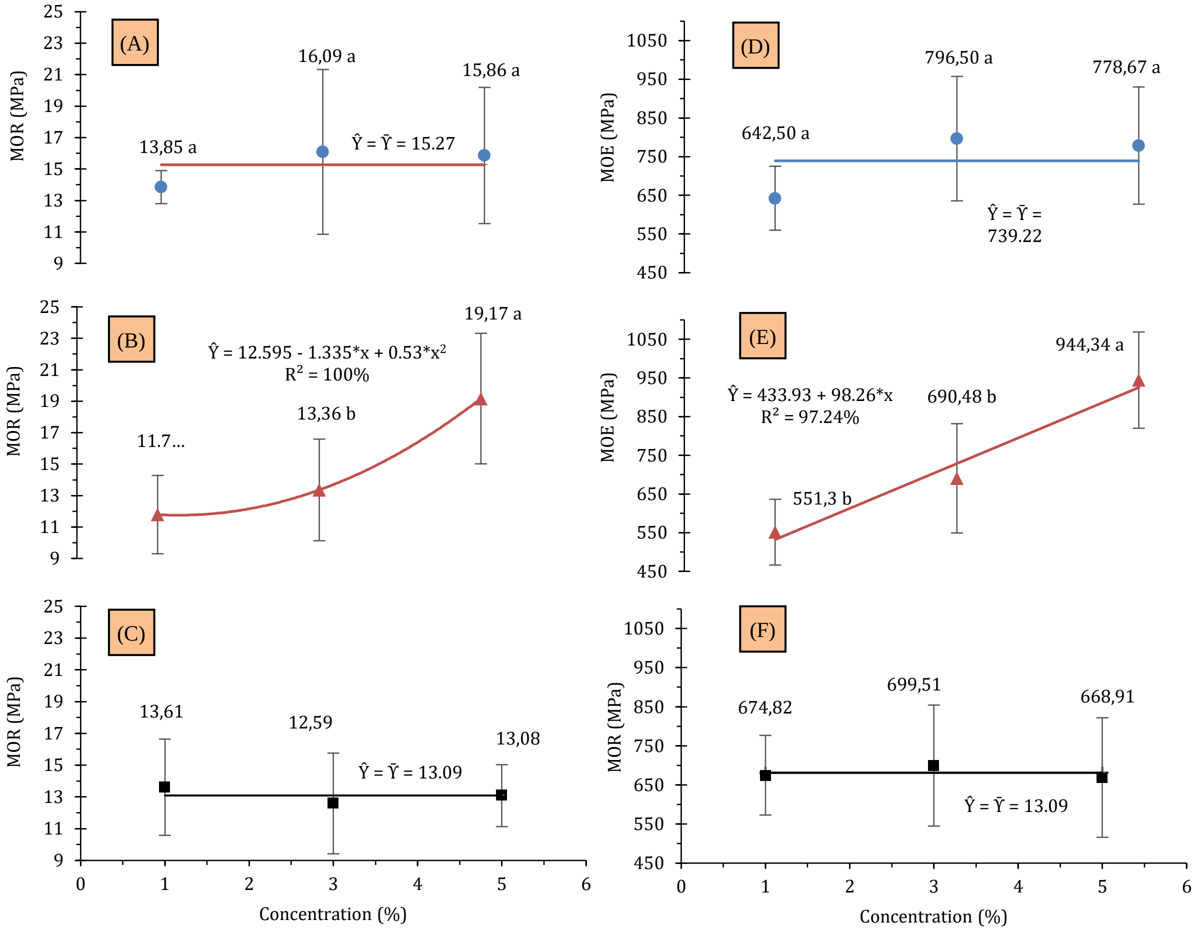
The effect of solution concentration on MOE and MOR in bending strength perpendicular to the grain was investigated by regression analysis, whose estimated equations are shown in Figure 6. The effect was the same for MOE and MOR, where concentration had no effect (F test; $p > 0.05$) on the bending strength for boric acid and borax. For DOT, the resistance increased from 1% to 3% and had a steep decrease to 5%, where the latter means were the lowest.

Table 7 - **Bending strength perpendicular to the grain: modulus of rupture (MOR) and elasticity (MOE) by treatment**

Boron compound	Concentration (%) / Bending strength perpendicular to the grain - MOE and MOE (MPa)					
	1		3		5	
	MOR	MOE	MOR	MOE	MOR	MOE
Boric acid	13.85±1.05a	642.50± 82.71a	11.79±2.49b	551.30±84.98b	13.61±3.03a	674.82±101.9a
Borax	16.09±5.24a	796.50±160.87a	13.36±3.23b	690.48±141.33b	12.59±3.17a	699.51±154.8a
DOT	15.86±4.33a	778.67±151.54a	19.17±4.15a	944.34±124.48a	13.08±1.95a	668.91±152.8a
General mean	15.27±3.97	739.23±131.91	14.77±3.58	728.71±118.59	13.10±2.71	681.08±134.7

Note: means followed by the same letter in the column do not differ (Tukey test; p > 0.05). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 6 - **Bending strength perpendicular to the grain: effect of boron compound concentration on modulus of rupture - MOR (A, B, C) and elasticity - MOE (D, E, F)**



Note: boric acid + borax ≈ DOT (disodium octaborate tetrahydrate). A, D: Boric acid; B, E: Borax; C, F: DOT; ^{ns} no significant (F test; p > 0.05); ** significant (F test; p < 0.01). Mean ± standard deviation.

The means of the treatments (Table 7) were compared to the control (16 MPa/MOR and 705 MPa/MOE) by the Dunnett test. Only the MOE of DOT at 3% was different ($p < 0.05$) from the control, with a higher mean (945 MPa). All the other means of MOE and MOR did not differ ($p > 0.05$) from the control.

Although no statistical analysis was performed for this comparison, the means of MOR and MOE parallel to the grain were higher than those perpendicular to the grain. Taking the control as a reference, the means of MOE and MOR parallel to the grain were, respectively, 8.3 and 3.2 times higher than those perpendicular to the grain.

Most treatments did not affect the static bending test compared to the control, and positive effects were found. This is a good result because it means that impregnation with boron compounds had no negative effects on this property. However, for other types of panels and also for wood, increase, decrease or no effect of boron compounds on the physical-mechanical properties of wood and reconstituted panels has been observed (Colakoglu *et al*., 2003). Thus, boron compounds did not affect bonding strength (Özçifçi, 2008). However, there are cases in which boron compounds improved the vibro-mechanical properties of wood (Zhang *et al*., 2023).

Nonetheless, for particleboards (Özlüsoylu; Istek; Yalçinkaya, 2017), MDF (Ustaomer; Usta, 2012), and *Fagus orientalis* wood (Fídan; Adanur, 2019), it was noticed a reduction in mechanical resistance and an increase in retention rates with increase in concentrations of boron compounds. Other studies also reported that higher levels of concentration or greater retention of boron compounds resulted in lower values of compressive strength parallel to the grain researchers (Simsek; Baysal; Peker, 2010; Toker *et al*., 2008), what was also observed in the present study.

According to Brazilian Association of Mechanically Processed Wood (ABIMCI, 2022) all panels (including the control) were approved regarding static bending parallel to the grain, because they had MOE and MOR higher than 3110 MPa and 27.8 MPa, respectively . However, no treatment met the requirements regarding static bending perpendicular to the grain, indicating that the panels must not be used in this kind of loading.

According to ANOVA, the interaction between boron compound and concentration was significant for shear strength - SS (F test; $p < 0.01$). As shown in Table 8, there was a significant difference (F test; $p < 0.05$) between boron compounds only at 3% and 5% concentrations.

At 3%, all means differed, and boric acid was the most resistant, followed by DOT and borax. At 5%, DOT and borax had the highest means (not significantly different) and borax had the lowest, which was a similar result compared to 3%. The effect of solution concentration on shear strength was determined by regression analysis, whose estimated equations are shown in Figure 7, where it can be seen that each boron compound had a completely different behavior from each other.

For DOT, the effect of solution concentration was not significant (F test; $p > 0.05$). However, the effect was significant ($p < 0.05$) for the other boron compounds, which had opposite behavior. For boric acid, shear strength slightly increased from 1% to 3% and decreased steeply up to 5%, which had the lowest mean. For borax, the shear strength constantly decreased from 1% to 5% concentration.

The means of shear strength (MPa) and bond line failure (%) of treatments (Table 8) were compared to the control (1.37 MPa and 30.8%) by the Dunnett test. For shear strength, three treatments (boric acid at 1% and 3%, DOT at 1%) out of nine did not differ ($p > 0.05$) from the control, which presented the best results. For other treatments, the effect was negative because the shear strength was lower than the control.

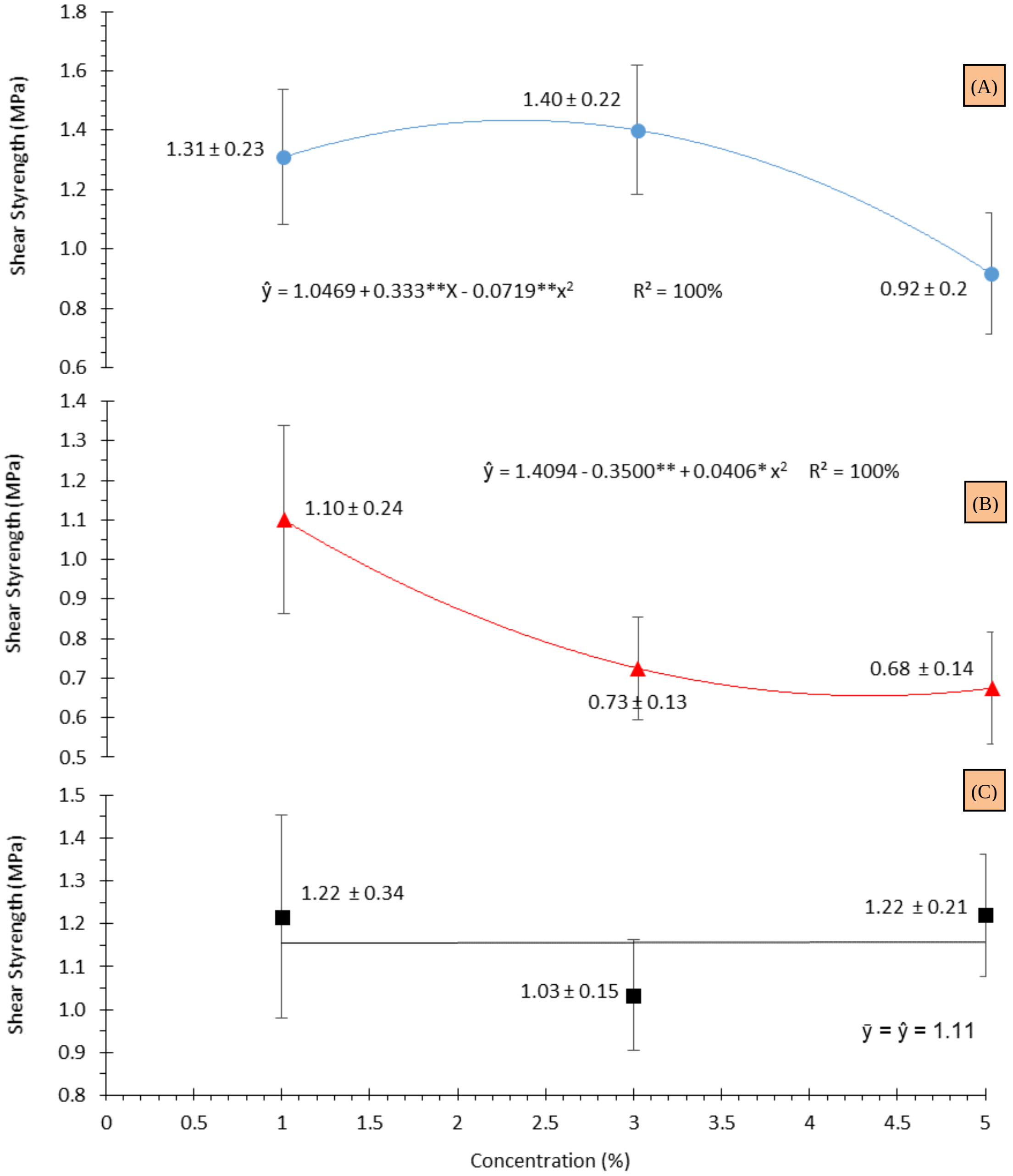
When testing the shear strength, the aim is to get more failure in the veneers than in the bond line, which means efficient bonding. Four out of nine treatments (the previous three, plus borax at 1%) did not differ (F test; $p > 0.05$) from the control regarding the percentage of bond line failure. Similar to shear strength, no treatment had better results than the control.

Table 8 – **Shear strength and percentage of apparent cohesive wood failure by treatment**

Boron compound	**Concentration (%) / Shear strength - SS (MPa) and Wood failure – WF (%)**					
	1		**3**		**5**	
	SS (MPa)	**WF (%)**	**SS (MPa)**	**WF (%)**	**SS (MPa)**	**WF (%)**
Boric acid	1.31±0.23a	27.92±31.66	1.40±0.22a	17.92±16.71	0.92±0.20a	9.17 ± 9.25
Borax	1.10±0.24a	13.33±12.85	0.73±0.13c	5.83±9.49	0.68±0.14b	7.50±13.23
DOT	1.22±0.34a	21.67±15.13	1.03±0.15b	6.67±3.89	1.22±0.21a	16.25±14.94
General mean	1.21±0.28	20.97±20.10	1.05±0.18	10.14±10.33	0.94±0.20	10.97±12.53

Note: means followed by the same letter in the column do not differ (Tukey test; $p > 0.05$). Mean ± standard deviation. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate).

Figure 7 - **Effect of boron compound concentration on shear strength. Boric acid + borax ≈ DOT (disodium octaborate tetrahydrate)**



Note: A: Boric acid; B: Borax; C: DOT; * significant (F test; $p < 0.05$); ** significant (F test; $p < 0.01$). Mean ± standard deviation.

An overall analysis indicates that the boron compounds harmed the bonding quality, except at 1% concentration. The boron compounds do not affect the practical bonding quality but tend to reduce the shear strength (Özçifçi, 2008). Thus, as contextualized for static bending, no there effect of boron compounds, nor of concentration of solution or retention used on shear strength in some properties of wood and plywood was observed (Fídan; Adanur, 2019; Özlüsoylu; Istek; Yalçinkaya, 2017; Ustaomer; Usta, 2012; Zhang *et al.*, 2023).

Nonetheless, for results of shear strength and delamination of *Picea abies* lamellae bonded with melamine-urea formaldehyde and polyurethane, the addition of boric acid (0.1% and 0.5% boron) to adhesive did not harm the bonding performance (Lesar *et al.*, 2011). On the other hand, it improved shear strength and the percentage of wood failure with polyurethane. Thus, as observed for other properties evaluated in the present study, the increase of boron compounds did not affect or negatively affect some properties of plywood, depending on the boron compound, concentrations, or retention, as also reported in literature (Fídan; Adanur, 2019; Özlüsoylu; Istek; Yalçinkaya, 2017; Toker *et al*., 2008).

Despite the negative results, six out of nine treatments (boric acid at 1% and 3%, borax at 1%, and DOT at any concentration) met the shear strength requirements (> 1.0 MPa) (CEN, 1993e), regardless of the percentage of bond line failure.

Conclusions

Considering the good performance in physical-mechanical properties, we recommend the use of boric acid at 1% or 3% concentration, or the other compounds at 1% concentration.

Boron compounds made the plywood more hygroscopic, except for those treated with boric acid at 1% concentration. However, the increased hygroscopicity did not degrade plywood quality.

Plywood treated with boric acid at 1% and borax at 1% had lower water absorption compared to control. The same result was achieved by boric acid at 3% for thickness swelling after 2 h. In general, the boron compounds did not affect thickness swelling.

Plywood treated with borax at 3% concentration had a higher static bending parallel to the grain compared to the control. In general, the boron compounds did not affect the bending strength perpendicular to the grain. For shear strength, plywood treated with boric acid at 1% and 3% had the best results.

In general, the panels met the quality requirements of the Brazilian Association of Mechanically Processed Wood (ABIMCI) regarding MC, apparent density, and bending strength parallel to the grain.

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