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A SYNERGIC EFFECT OF WATER-BASED ACRYLIC RESIN WITH BORIC ACID ON LEACHABILITY

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ABSTRACT

In this study, the Scots pine wood samples were impregnated (single treatment) with boric acid combined with two types of water-based acrylic resin (pure acrylic and semi-translucent acrylic emulsion) to limit the boron leaching and improve the decay resistance. The results showed dimensional stability in anti-swelling efficiency and water absorption improved in wood specimens treated with boric acid and acrylic types. While the leachability was over 90% for only 3% boric acid-impregnated wood (control), it was calculated at 36% for acrylic emulsions-impregnated wood. Although there were no weight losses for the unleached woods, it was up to 9% for leached woods impregnated with acrylic resin and emulsion. The 25% acrylic emulsion had no weight losses after the leaching test for *Coniophora puteana* and *Trametes versicolor.* The boric acid combined with acrylic resin can improve the leaching resistance with the synergic effect, enhancing resistance against biological threats.

KEYWORDS: Anti-swelling, acrylic resin, boric acid, decay resistance, ICP analyses.

INTRODUCTION

Boron is a familiar biocide and fire retardant, so it is not new. The efficiency of boron-based preservatives against wood-degradative organisms such as fungi, insects, or termites is proven (Tsunoda 2001, Furuno et al. 2003, Temiz et al. 2008, Thevenon et al. 2009). The fire resistance of boron compounds has also been well-known for a long time (Blasi et al. 2007, Wang et al. 2004). Moreover, the relatively low toxicity of boron compounds against mammals has offered an opportunity to be an alternative to wood preservatives (Jaouadi 2021, Morrel et al. 2011). Boron is also an effective chemical than copper and zinc (Lloyd et al. 2001, Obanda et al. 2008). The water exposure of boron compounds generates tetrahydroxyborate $[B(OH)_4]^-$, which mainly provides biological resistance against the functions of wood-degrading organisms (Obanda et al. 2008). However, the leachability of boron compounds from wood is the main drawback and limits the use of boron compounds, especially in ground contact applications (Furuno et al. 2006). In recent years, researchers have worked on limiting or reducing boron leaching by using vegetable oils, tannins, water repellents, several monomers, and polymer systems to increase the use of environmentally friendly boron compounds in wood preservation (Di Blasi et al. 2007, Hwang et al. 2005, Kartal and Green 2003, Kartal et al. 2004). Boron compounds such as boric acid forms 1-methoxy-2hyroxy benzene and 1,2-dimethoxy benzene groups in lignin, while it is 1,2-dihydroxy benzene groups with tannin (Pizzi and Baecker 1996). However, boron compounds do not chemically bond with wood hydroxyl groups entirely, increasing leachability (Caldeira 2010). Peylo and Willeitner (1995) also stated that the leachability of boron compounds in wood continues under 20% moisture content. It is also suggested that one of the best ways to inhibit the diffusibility is to keep boron impregnated wood away from high humidity. There are also many studies about limiting the leachability of boron compounds. Lesar et al. (2012) liquefied Norway spruce sawdust and impregnated it with boric acid. The application demonstrated the decrease in the leaching of boric acid from liquefied wood. Organo boron compounds, which interact with a part of lignin, are widely evaluated to gain wood leachability resistance (Deveci et al. 2017, Yalinkilic et al. 1998a,1999). Vegetable oils also improve the boron retention in the wood by providing a water barrier. Lyon et al. (2007) highlighted that boron retention depends on the drying properties of oils. The impregnated wood with boric acid and vegetable oils showed resistance against termite and fungal degradation, while linseed oil is only decay resistant.

In recent studies, acrylic and acrylic-silicon systems that can increase the water-repellency and dimensional stability of wood have been introduced and focused on modifying wood by treating it with appropriate resin systems. When used with boron and other wood preservatives, these acrylic-based systems have been found to have the potential to improve preservative performance, reduce preservative leaching rates, improve the water-repellent properties of wood, and increase biological resistance to wood-degrading fungi (Obanda et al. 2008, Salman et al. 2014, Whang et al. 2005). Kartal and Green (2003) used natural polymers (pectin, starch, sucrose, guar gum), synthetic polymers (carboxymethylcellulose), and N‘Nnaphthaloyl-hydroxylamine (NHA) to limit the leachability. While the highest leachability was found in wood impregnated with boron (alone), boron leachability was reduced with the synergistic effect of the NHA component. When wood was treated with silane compounds, glycidyl ether, and methyl methacrylate to increase the boron leachability, the polymers improved dimensional stability, decreased boron disintegration, and enhanced resistance against fungi and termites (Kartal et al. 2004, 2009). While the polyvinyl alcohol treatment reduced the boron leachability from wood, the boron release could not be prevented entirely (Mohareb et al. 2011). In the methyl methacrylate (MMA) impregnation treatment combined with heat treatment to reduce boron leachability, the dimensional stability of the wood was improved, and the leaching resistance was provided (Priadi et al. 2020). The appropriate conditions make wood sensitive to biological agents. The high humidity also affects boron compounds, which can be quickly released from the wood. Treatment with resin improves the dimensional stability of wood and gains water repellency (Deka and Saikia 2000, Mourant et al. 2009).

The main objective of this study was to improve the leachability resistance of boron in wood to improve the biological resistance. Wood specimens were impregnated with boric acid with two types of water-based acrylic resin (single treatment). The effect of the mixture (boron and resin) on the water absorption (WA) and thickness swelling (TS) was examined. Inductively coupled plasma (ICP) spectroscopic analysis also determined the boric acid release from wood. The wood samples were exposed to brown-rot fungi (*Coniophora puteana*) and white-rot fungi (*Trametes versicolor)* to determine the effect of boron compounds against decay fungus.

MATERIAL AND METHODS

Wood specimens, 20 x 20 x 30 mm for water absorption, thickness swelling, and leaching tests, and 5 x 15 x 15 mm for decay test, were obtained from Scots pine sapwood (*Pinus sylvestris* L.). The Scots wood samples were free of knots and resins, and no visible evidence of infection by mold, stain, or fungi was evident. Boric acid (H_3BO_3) was provided from Boren in Turkey. The water-based pure acrylic and semi-translucent acrylic emulsion were used as modifying resins. The characteristic features of these acrylic resins were given in Tab. 1.

Tab. 1: Water-based acrylic resin types.

Resins	Solids by weight (%)	pH	Viscosity (mPa·s)	MFFT (°C)	Group Code
Pure acrylic	50	7.0-8.5	50-500	2	X
Acrylic emulsion	40	7.9	75	20	Y

Note: MFFT is the minimum film formation temperature.

Wood specimens were conditioned at 23 ± 2°C and 65 ± 5% relative humidity (RH) before treatment. The impregnation process was carried out as a single treatment. The boric acid concentration was 3%. The different concentration of water-based acrylic resins (5, 10, 15, and 25%) was prepared by boric acid solution (test specimens). Control specimens were prepared by treatment with 3% boric acid only. The impregnation process consisted of 15 min vacuum (685 $Hg{\cdot}mm^{-1}$) in a desiccator. After the impregnation, wood samples were left to complete polymerization for 24 h in room conditions (25°C). The weight gain was calculated by weight before and after the treatment.

Untreated and treated specimens were exposed to brown-rot fungi *Coniophora puteana* (Schumach.) P. Karst. (Mad-515), and white-rot fungi *Trametes versicolor* (L.) Lloyd (mad-697) according to standard EN 113 (1994). The media were steam-sterilized at 120°C for 20 min before being poured into petri dishes. After inoculation, petri dishes were held at 23 ± 2°C and 65 ± 5% RH so that the fungi could spread over the entirety of the petri dishes. After the samples were steam-sterilized under the same conditions, the fungal test was started. Every measurement was taken from clean and dry samples. Six wood samples were used for each group. The weight loss was calculated by dry weight before and after the decay test.

The control and test specimens were entirely soaked in the water at 20 ± 1°C in the beakers. The samples were weighed and measured after 6 h, 24 h, and 48 h. The water absorption (WA), thickness swelling (TS), and the percentage of anti-swelling efficiency (ASE) were calculated according to Eqs. 1, 2, and 3, resp.

$$WA= [(W_2-W_1)/W_1] \times 100 \quad (1)$$
$$TS = [(S_2-S_1)/S_1] \times 100 \quad (2)$$
$$ASE (\%) = [(S_c-S_t)/S_c] \times 100 \quad (3)$$

where: W_2 - the wet weight of the sample, W_1 - the dry weight, S_2 - the wet volume of the sample, S_1 - the dry volume of the sample, S_c - the untreated specimens' volumetric swelling coefficient, and St - the treated specimens' volumetric swelling coefficient.

The leaching test was conducted according to the standard of AWPA E11-06 (2016). Before the leaching test, all samples were conditioned at 23 ± 2□ and 65 ± 5% RH. Wood samples were submerged in 300 mL of distilled water in 500 mL beakers. Specimens were vacuumed to impregnate water for 20 min. The wood samples were left during 14 days in the water at 23 ± 2°C and 65 ± 5% RH. The water was replaced every 6 h, 24 h, 48 h, and after every 48 h for 14 days. At the same time, the leachates were collected for analysis. Six samples were tested for each group.

The control and test specimens were prepared according to AWPA A7-93 (1993). The specimens were grinded using a laboratory-scale Wiley mill (IKA MF10, IKA-Werke, Staufen, Germany). The amount of 0.5 g wood samples in 7.5 mL nitric acid (65%, Merck) were added to the beakers and heated until the brown smoke seemed. After that, 5 mL hydrogen peroxide (35%, Merck) was added to the beakers. Finally, the samples were filtered with a membrane filter with a diameter of 0.45 µm. The filtered samples were diluted.

The leachates obtained from the leaching test during 14 days were held in sealed flasks. The leachates and filtered samples were analyzed with an inductively coupled plasma (ICP) spectrometer (PerkinElmer, Optima 7000 DV, USA). The percentage of leaching boron content from the wood was calculated by proportioning the released boron content to the initial boron content. The results were evaluated statistically according to the analysis of variance (ANOVA). The differences between the groups were determined using the Duncan test ($p < 0.05$).

RESULTS AND DISCUSSION

Boron leachability

The effect of water-based acrylic resin on the leachability of boric acid was evaluated by ICP spectroscopy analysis. The boric acid released from the wood samples was given in Tab. 2.

Tab. 2: The leached boron from wood samples after 14 days.

	Control	X5	X10	X15	X25	Y5	Y10	Y15	Y25
Leached boron (%)	91.5^a (1.13)	78.6^b (1.88)	67.2^c (1.25)	64.5^{cd} (0.67)	50.2^e (1.20)	69.5^c (1.13)	65.6^c (2.35)	59.9^d (2.30)	36.5^f (0.66)

Note: Letters show the homogeneity groups, and values in parentheses are the standard deviations.

The results showed that releasing boric acid from the wood can be limited, but it is inevitable. The highest removal was obtained from wood samples impregnated with only 3% boric acid. In contrast, the lowest one was the combination of boric acid and 25% acrylic resin Y. Although almost all boric acid (91.37%) leached from the control samples, it was, however, not wholly leached from the wood. Kartal et al. (2004) stated that the decrease in the concentration of boron increased the leachability.

Meanwhile, the release of boric acid decreased to 36.47% with acrylic resins. Both acrylic resins had a significant effect on leachability. Meanwhile, acrylic resin Y provided the best leachability resistance for all the concentrations. The statistical differences between the groups were also shown in Tab. 3. As seen, the control group disintegrated from wood samples impregnated with acrylic resin. As the resin concentration increased, the dimensional stability of the wood increased (Tab. 2). The interaction of wood with water is limited by acrylic resin impregnation. Therefore, the boric acid leachability was limited, which affects biological resistance.

Tab. 3: Results of ANOVA analysis of variance for boric acid leaching.

	Sum of squares	df	Mean square	F	Sig.
Between groups	5915.434	8	739.429	213.714	0.000
Within groups	62.278	18	3.460		
Total	597.712	26			

Note: df is degree of freedom; F shows the difference between the groups; Sig is significance level.

Weight gain, water absorption, and anti-swelling efficiency

The weight percentage gain is essential to determine the efficiency of impregnation chemicals in the usage area. The weight gain percent (WGP) of wood samples was calculated after the impregnation (Tab. 4). The WGP values of samples varied between 95.25% and 115.43%. The highest WGP values were obtained from boric acid treatment with 5% acrylic resin combinations. As the water-based acrylic resin concentration increased, the retention value decreased. However, the difference between the retention values was not statistically significant (Tab. 5).

Tab. 4: WGP value of samples (%.)

Groups	Control	X5	X10	X15	X25	Y5	Y10	Y15	Y25
WGP (%)	106.7 (28)	115.4 (51)	96.4 (52)	95.6 (44)	76.6 (32)	115.3 (51)	103.5 (49)	95.4 (12)	98.5 (52)

Note: Values in parentheses are the standard deviations.

Tab. 5: Results of ANOVA analysis of variance for WGP ($p < 0.05$).

	Sum of squares	df	Mean square	F	Sig.
Between groups	6871.819	8	858.977	0.406	0.911
Within groups	95313.348	45	2118.074		
Total	102185.167	53			

Note: df is the degree of freedom; shows the difference between the groups; Sig. is the significance level.

Wood is known to change its dimensions depending on environmental humidity (Popescu et al. 2016). Besides dimensional stability, the mechanical and biological properties are also affected by the moisture content of wood (Chang and Chang 2002). Moreover, the humidity also affects the boron mobility in the wood. One way to provide the wood with dimensional stability is impregnation with resin (Deka and Saikia 2000). Wood samples were impregnated with two different water-based acrylic resins to decrease boron's leachability and dimensional stability in this study. The water absorption and thickness swelling values of samples are given in Tab. 6. Acrylic resin impregnation improved the dimensional stability of wood. As seen in Tab. 6, thickness swelling values decreased for both radial and tangential sections with both resins compared to control samples. Moreover, the change for both wood directions was significant as the resin content increased from 5% to 25%. The hydroxyl and oxygen-containing groups in the wood are responsible for bonding with water molecules (Rowell and Banks 1985). The resin impregnation inhibited the bonding between water molecules and hydroxyl groups of wood (Deka and Saikia 2000). The acrylic resin significantly limited the thickness swelling in the first 6 h. After that, the increase was a little rapid. However, the increase also remained below the control samples. Likewise, water absorption values of resin-impregnated wood specimens decreased as resin content increased. While the highest water absorption and thickness swelling values were obtained from control samples, the lowest one was resins at a concentration of 25%; the cell lumen filled with resin and limited the water absorption.

Tab. 6: The thickness swelling and water absorption of wood samples (%).

Groups	6 h		24 h		48 h		Water absorption		
	Tangential	Radial	Tangential	Radial	Tangential	Radial	6 h	24 h	48 h
Control	4.60 ± 0.41	1.83 ± 0.13	5.61 ± 0.45	2.40 ± 0.20	5.80 ± 0.55	2.86 ± 0.21	49.8 ± 4.01	57.1 ± 4.51	65.8 ± 5.56
X5	4.15 ± 0.36	1.82 ± 0.20	5.38 ± 0.60	2.28 ± 0.21	5.75 ± 0.50	2.39 ± 0.24	44.2 ± 4.21	54.4 ± 4.78	62.7 ± 5.76
X10	4.01 ± 0.35	1.66 ± 0.17	5.37 ± 0.55	2.26 ± 0.18	5.71 ± 0.60	2.32 ± 0.25	43.8 ± 4.12	52.4 ± 5.01	59.8 ± 5.23
X15	3.71 ± 0.27	1.43 ± 0.12	5.24 ± 0.52	2.24 ± 0.17	5.41 ± 0.42	2.29 ± 0.18	40.7 ± 3.89	49.9 ±4.46	56.4 ± 5.01
X25	3.27 ± 0.36	1.34 ± 0.11	4.67 ± 0.41	2.06 ± 0.25	5.14 ± 0.38	2.22 ± 0.17	33.0 ± 2.98	43.3 ± 3.78	52.1 ± 4.78
Y5	4.11 ± 0.44	1.71 ± 0.10	5.15 ± 0.50	2.71 ± 0.23	5.50 ± 0.35	2.79 ± 0.23	48.1 ± 4.24	54.7 ± 3.98	61.1 ± 4.98
Y10	4.04 ± 0.37	1.65 ± 0.18	4.95 ± 0.49	2.26 ± 0.30	5.36 ± 0.55	2.41 ± 0.13	44.1 ± 3.97	52.5 ± 4.53	59.3 ± 5.32
Y15	3.82 ± 0.25	1.62 ± 0.15	4.89 ± 0.49	2.14 ± 0.15	5.30 ± 0.57	2.27 ± 0.18	42.5 ± 4.06	51.2 ± 4.65	58.3 ± 4.36
Y25	2.98 ± 0.22	1.29 ± 0.19	4.17± 0.35	2.03 ± 0.16	4.58 ± 0.48	2.25 ± 0.12	22.5 ± 1.79	34.7 ± 3.26	44.13 ± 3.87

The percentage of anti-swelling also presents that acrylic resins gained wood water repellency properties. The treatment with acrylic resin improved the ASE of wood by up to 33%, as seen in Fig. 1. As the concentration increased, the ASE values of samples increased. The highest ASE value obtained from the highest resin concentration was 25%. When compared to resins, Y provided more water repellency effect to wood. In previous studies, methyl methacrylate (MMA) was evaluated to decrease the leachability of boron. Şolpan and Guven (1999) used MMA, which gained 12% ASE to wood. Kartal et al. (2004) also utilized the MMA and obtained up to 75% ASE with the additives. However, the hygroscopic properties of boron compounds could also influence the ASE values of wood (Yalinkilic et al. 1998b).

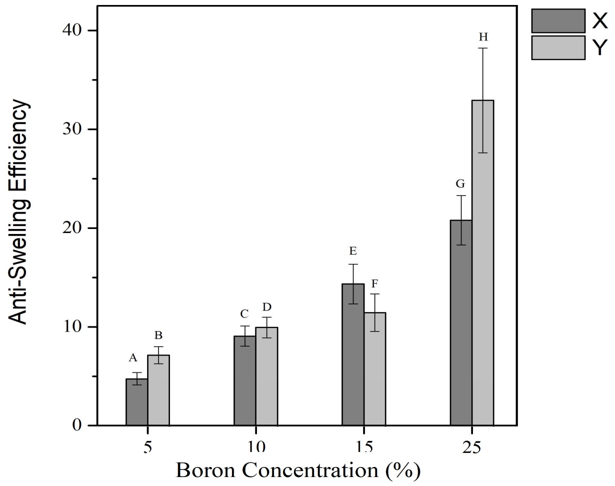

Fig. 1: Anti-swelling efficiency of wood samples (letters A-H show homogeneity groups).

Decay resistance

The fungal decay test is one of the factors which determines the efficiency of the preservative. The efficiency of boron compounds against biological threats is well-known, as explained above. However, the leachability of boron compounds is an essential obstacle that decreases wood resistance against fungi, insects, and termites. The mass losses of wood samples exposed to brown-rot fungi, *C. puteana,* and white-rot fungi *T. versicolor* were given in Fig. 2. The mass losses of wood samples without any preservatives were 35%, 29% for brown and white-rot fungus, respectively, while 18% and 15% for leached control-BA.

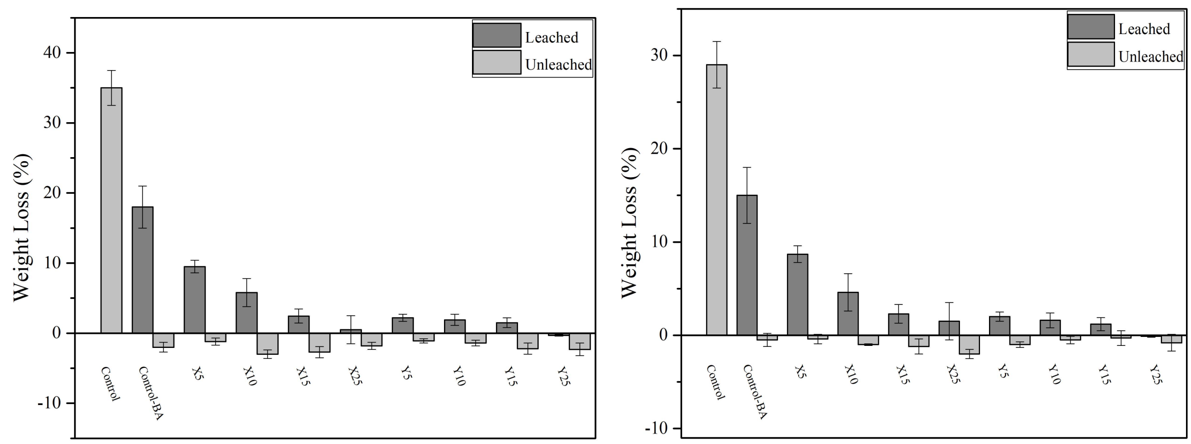

Fig. 2: The mass losses of wood samples exposed to: a) brown-rot fungi, b) white-rot fungi.

The weight losses due to white-rot fungi were less than brown-rot fungi. Brown rot fungi are associated with softwood species used in this study (Popescu et al. 2010). Meanwhile, there was no mass loss for unleached samples for brown-rot fungi, while the mass loss was under 10% for the impregnated with the combination of boric acid and acrylic resins after the leaching test. Similarly, the combination of boric acid and acrylic resin inhibited the activity of white-rot fungi, *T. versicolor*. Unleached samples did not lose weight, while it was up to 9% for leached wood samples impregnated with the combination of acrylic resin and boric acid. Hashemi et al.

(2010) found out that the mass losses of BA-treated wood were under 2%, which also decreased as BA concentration increased.

Tsunoda (2001) stated that 2 $kg \cdot m^{-3}$ of boric acid retention against fungi is adequate. Moreover, they found that 0.9 $kg \cdot m^{-3}$ inhibited *T. versicolor* while it was a 6.5% mass loss for *F. plustris*. According to WGP values in Tab. 1, over 4 $kg \cdot m^{-3}$ boric acid penetrated wood samples according to retention values in this study. Therefore, unleached wood samples have sufficient resistance against decay fungus. The lowest weight losses were obtained from the combination of 25% acrylic resin Y and boric acid. There were no weight losses for wood samples impregnated with a combination of acrylic resin Y and boric acid exposed to both fungi due to having above 2 $kg \cdot m^{-3}$ of boric acid after the leaching test. As the concentration of acrylic resin increased, wood was more stable against water responsible for releasing boron. Therefore, the acrylic resin impregnation decreased the boric acid leachability, which improved the decay resistance of samples.

CONCLUSIONS

Boron compounds are well-known wood preservatives against biological threats. The mobility or release of boron from wood is the main problem. The most crucial trigger for the leaching of boron is humidity. In this study, wood specimens were impregnated with a combination of water-based acrylic resin and 3% boric acid to reduce the release of boron and improve the biological resistance against fungi. According to previous studies, the retention values showed that wood samples had adequate boric acid per m^3 above 2 $kg \cdot m^{-3}$. The WA and TS values also demonstrated the increasing dimensional stability of wood samples. The acrylic resin filled the cell lumens, which limited interaction with water. Therefore, the less moisture content, the less leachability. The ICP spectroscopy analysis revealed a decrease in the leachability of boron. As the concentration of acrylic resin increased, the release of boron decreased. The leachability test results were also accompanied by the decay test results. Boric acid is more effective against fungus unless it is unleached. However, the boric acid content in the wood decreased after the leaching test, despite the acrylic resin. Although the boron content decreased, adequate resistance was provided as the concentration of acrylic resin increased. The highest fungal resistance was obtained from the 25% resin concentration. Moreover, the lowest weight loss was obtained from 25% acrylic resin Y and boric acid. According to obtained results, the water-based acrylic resins can be evaluated as reducing boron release, which improves the decay resistance of wood.

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