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Dimensional Stability and Wettability of Modified Samama (*Anthocephallus macrophyllus*) Wood with Boron, Citric Acid, and Heat Treatment

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ABSTRACT

This research aimed to address the leaching phenomenon of boron preservatives in wood. The subsequent investigation focuses on wood's dimensional stabilization and wettability after a two-stage impregnation process. Samama (*Anthocephallus macrophyllus*) wood was impregnated with boron (boric acid, borax, and a combination of both) at a pressure of 7 atm for 4 hours, with each preservative's concentration set at 5%. After oven drying at 60°C until achieving a 15% moisture content, the next step involves a second-stage impregnation with citric acid (at a 5% concentration) under the same pressure and duration. The final step included heat treatment at 80°C and 160°C for 4 hours. The research results indicate that boron and citric acid enhance the dimensional stabilization of samama wood. The best dimensional stabilization treatment combines boric acid, borax, citric acid, and heat treatment at 160°C. This study confirms that citric acid improves the dimensional stabilization of samama wood, whether used with boron or not. Compared to treatments without citric acid and heating, the two-stage impregnation reduces boron leaching by up to 30%. The research also recommends that all treatments exhibit good finishing properties.

1. Introduction

Wood impregnation with various substances has been a fundamental focus for enhancing wood quality. The objectives of impregnation are diverse, ranging from enhancing the surface aesthetics of wood (Can and Sivrikaya 2019), improving dimensional stabilization (Demirel et al. 2018), and extending the service life (Nguyen et al. 2020). One well-known substance in wood impregnation is boron. This preservative material is effective in protecting wood from destructive organisms such as termites and fungi (Palanti et al. 2017; Priadi et al. 2023). However, boron impregnation has a drawback – its susceptibility to being easily washed away by water and moisture (Verly Lopez et al. 2020), limiting its long-term durability (Kartal et al. 2020).

Previous research has made various efforts to enhance the effectiveness of boron impregnation in wood. At least two techniques have been employed: modifying the substance (Beyli et al. 2018; Huang et al. 2018) and modifying the process (Nguyen et al. 2020). The use of binding agents after boron impregnation and thermal modification remains relevant for further investigation. Particularly, the binding agent falls into the category of environmentally friendly materials–citric acid. Citric acid possesses chemical properties that can form stable bonds with the hydroxyl groups of wood (Augustina et al. 2023; Lee et al. 2020). Previous studies have reported that heat treatment to arrange these polyesterification bonds is effective at a temperature of at least 140°C (Larnøy et al. 2018; Lee et al. 2020). Citric acid results in a permanent fixation on samama wood after one hour of compression treatment at 180°C (Rumbaremata et al. 2019). It can potentially reduce the issue of boron leaching due to water and moisture, enhancing its durability.

This research aims to observe changes in the dimensional stabilization, wettability, and surface roughness of samama wood after impregnation with boron, binding with citric acid through heat treatment, or a combination of both. Another aspect under examination is the resistance of boron within samama wood after interaction with high water and moisture levels.

2. Materials and Methods

2.1. Material Preparation

The wood used in this research was samama (*Anthocephallus macrophyllus*) sourced from 9-year-old trees with a diameter of 10 cm and a height of 13 m. The wood was obtained from a community forest in Bogor, carefully chosen from disease-free stands with cylindrical trunks, and harvested at a 50 cm distance from the ground. The wood, cut into 100 cm lengths, was transported to the laboratory in a wet condition. Subsequently, the wood was transformed into boards with a thickness of 10 cm, passing through the pith, and then dried using a wood drying kiln at a temperature of 40°C until reaching a moisture content of 15%.

For the physical property tests, samples were extracted from the 10 cm section beyond the pith and cut into two sizes. The first sample measured 2 cm × 2 cm × 2 cm (thickness, width, length) and was used for density testing. The second set of samples, measuring 2.5 cm × 2.5 cm × 10 cm (thickness, width, and length), was prepared in triplicate for water absorption, leaching, and dimensional stabilization tests. Samples for surface roughness and wettability testing were sized at 2 cm × 5 cm × 5 cm (thickness, width, and length). Each test was conducted with five replications.

2.2. Impregnation Preparation

The first impregnation solution consisted of boric acid (BA), borax (B) and a combination of boric acid and borax. Each solution was dissolved in distilled water with a concentration of 5%. The second solution was citric acid with the same concentration as the first, i.e., 5%. Details are presented in **Table 1**.

2.3. Impregnation and Heat Treatment

Three solutions were placed in plastic containers, namely boric acid, borax and a mixture of boric acid and borax. The concentration of each solution was 5%. Test samples were immersed in these solutions until fully submerged, then placed in a prototype-scale pressure cylinder (5 m in length and 1.5 m in diameter) at a pressure of 7 atm. The duration of this process was 4 hours. Subsequently, the test samples were drained, weighed to determine preservative retention, and oven-dried at 60°C until reaching a moisture content of 5%. This activity took 5 days and involved weighing (Priadi et al. 2023).

The citric acid impregnation procedure mirrored that of boron impregnation. The concentration of the solution was also 5%. However, after impregnation with citric acid (CA), test samples were drained and heated in an oven, each at different temperatures, namely 80°C and 160°C. The test samples were then conditioned at room temperature of ± 25°C with a relative humidity of ± 80% for 7 days for subsequent testing.

Table 1. Samama wood types, boron and citric acid impregnation

No.	Type	Treatment code	Impregnation I	Impregnation II	Heat treatment (°C)
1	A	NOO-80	-	-	80
2	B	NOO-160	-	-	160
3	C	NOC-80	-	Citric acid	80
4	D	NOC-160	-	Citric acid	160
5	E	BAO-80	Boric acid	-	80
6	F	BAO-160	Boric acid	-	160
7	G	BAC-80	Boric acid	Citric acid	80
8	H	BAC-160	Boric acid	Citric acid	160
9	I	BXO-80	Borax	-	80
10	I	BXO-160	Borax	-	160
11	K	BXC-80	Borax	Citric acid	80
12	L	BXC-160	Borax	Citric acid	160
13	M	BBO-80	Boric acid-borax	-	80
14	N	BBO-160	Boric acid+borax	-	160
15	O	BBC-80	Boric acid+borax	Citric acid	80
16	P	BBC-160	Boric acid+borax	Citric acid	160

2.4. Testing

Test sample density was calculated based on the British Standard (BSI 1957). Water absorption and leaching tests followed ASTM D 1413-76 (ASTM 1976). Test samples were immersed in distilled water gradually for 2 hours, 24 hours, and 48 hours. Each immersion period was interrupted by oven-drying at a temperature of 103 ± 2°C until a constant weight was achieved. The leaching value was calculated from the difference in dry oven weight after impregnation and dry oven weight after immersion. Water absorption was calculated from the difference between the wet weight of the test sample after each immersion period and the dry oven weight after immersion.

Dimensional stabilization testing also occurred during immersion for 2 hours, 24 hours, and 48 hours. Measurements of dimensions were taken before and after the immersion process. The volume expansion of test samples was calculated from the air-dry condition to each water immersion process. Anti-swelling efficiency was calculated based on the volume expansion ratio between the control and treated test samples. In addition to these two factors, tangential and radial dimensional shrinkage testing was conducted. Shrinkage was measured from the wet to dry oven conditions. The final step involved calculating the Tangential/Radial Shrinkage Ratio (T/R Ratio).

Surface roughness testing adhered to the ISO 4287:1997 standard (ISO 1997). The test samples' roughness levels were measured using a surface roughness tester (Mitutoyo SJ-210). Measurements were conducted five times on the tangential cross-section, following the direction perpendicular to the fiber. The surface hardness measurement parameter was the arithmetical mean roughness (Ra).

Wettability testing was performed using the drop test technique (Cahyono et al. 2024). Distilled water was dropped onto the surface of samama wood using a micropipette. The droplets were recorded with a USB camera (dino-lite am211) at a magnification of 40 times for three minutes. The video data were then extracted every 3 seconds using GOM Player software. The contact angle was measured from each image using Image-J software version 1.42. The constant value for the rate of change of the contact angle (*K*) was obtained from Equation 1. The software used was Statistica version 10.

$$\theta = \frac{\theta i.\, \theta e}{\theta i + (\theta e - \theta i) exp\,[K(\frac{\theta e}{\theta e - \theta i})t]} \quad (1)$$

where θ is the contact angle at a specific time (°), θI is the initial contact angle of the droplet (°), θe is constant contact angle (°), t is time (seconds), and K is the constant rate of change of the contact angle (liter/second).

2.5. Data Analysis

The data from various tests on the stepwise impregnation of samama wood with boron, citric acid and heat treatment were analyzed using a factorial, completely randomized design. The factors involved were Factor A (boron compounds) involving 4 levels (boric acid, borax, boric acid+borax, no boron). Factor B included 2 levels (citric acid and without citric acid), and Factor C involved heat treatment involving two levels (80°C and 160°C). Duncan's test followed the analysis to see if the variance analysis (ANOVA) yielded a significant effect at a 95% confidence interval.

3. Results and Discussion

3.1. Density

The concentration of boron and citric acid used in this study was only 5%, resulting in no drastic change in the density of the test samples. The most significant change occurs in sample O (boric acid+borax, citric acid, 80°C). The increase is less than 8% (**Fig. 1**), indicating the formation of bonds between citric acid and the cell wall polymer (Lee et al. 2020). Citric acid falls into the category of polycarboxylic acids, capable of forming double ester bonds and producing a three-dimensional cross-linked network (Teacă and Tanasă 2020). Furthermore, the analysis of variance results shows that wood density is influenced by the interaction between boron, citric acid, and heat treatment ($p = 0.012$).

Interestingly, a reverse phenomenon is observed in some test samples after heat treatment, resulting in a density reduction of up to 4.32%. Heating causes the amorphous region to transform into crystalline structures (Chien et al. 2018). High-temperature heating, ranging from 120°C to 150°C, leads to the loss of wood extractives (Tarmian and Mastouri 2019; Todaro et al. 2015). The higher the temperature used, the greater the loss of extractives (Výbohová et al. 2018). In addition to extractives, high-temperature heating degrades cellulose in wood (Cahyono et al. 2021). The degradation of chemical components is closely related to the increase in temperature and its duration (Cao et al. 2022).

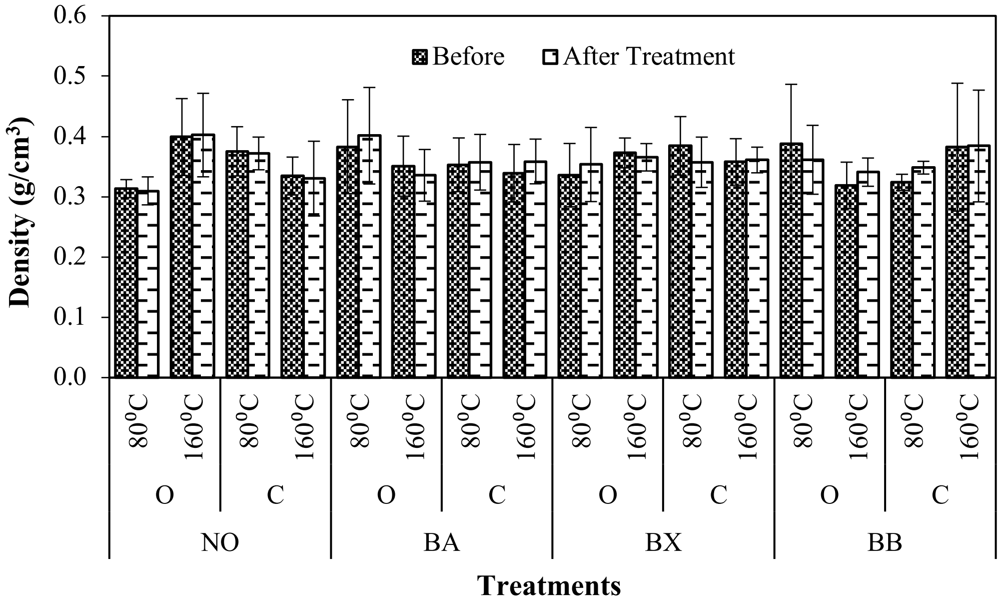

Fig. 1. Density of samama wood in various treatments (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.2. Water Absorption

Fig. 2 illustrates that water absorption (WA) in the test samples is reduced after the impregnation process. The most extreme reduction is observed in the sample Boric Acid+Citric Acid+HT160°C (48-hour immersion), with WA showing a value of 66.45%. This value is only 1/3 compared to the control sample. Citric acid impregnation forms bonds between the free OH groups of cellulose and the carboxyl groups of CA. These bonds replace the OH groups from water (Mihulja et al. 2021). The decrease in WA also occurs because the cavities are filled with impregnating material. This is supported by the analysis of variance results explaining that WA is influenced by the interaction between boron compound treatment and heating (p = 0.004).

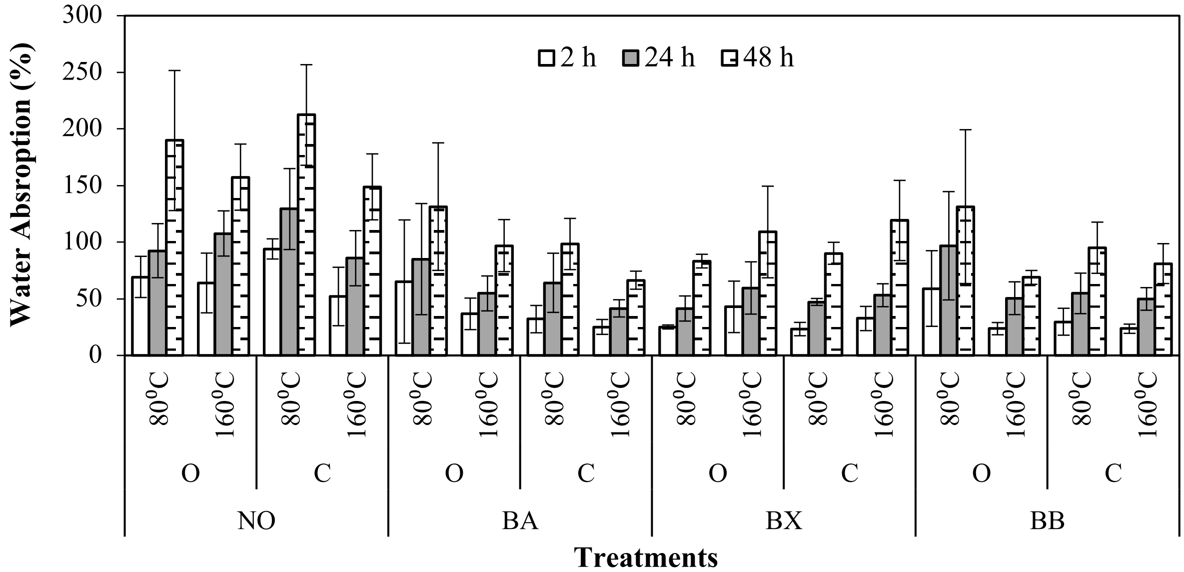

Fig. 2. Water absorption of samama wood after impregnation of boron, citric acid and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.3. Leaching

Citric acid can resist leaching by up to 30%. This value is found in the sample impregnated with a mixture of BA+BX and heat treatment of 80°C (**Fig. 3**). The analysis of variance results indicates that the leaching value is influenced by the interaction between boron compounds and citric acid ($p = 0.031$). This result reinforces the adhesive properties of citric acid, which can resist boron leaching in samama wood. Citric acid is an adhesive substance that forms cross-linking bonds with the hydroxyl groups of wood (Lee et al. 2020).

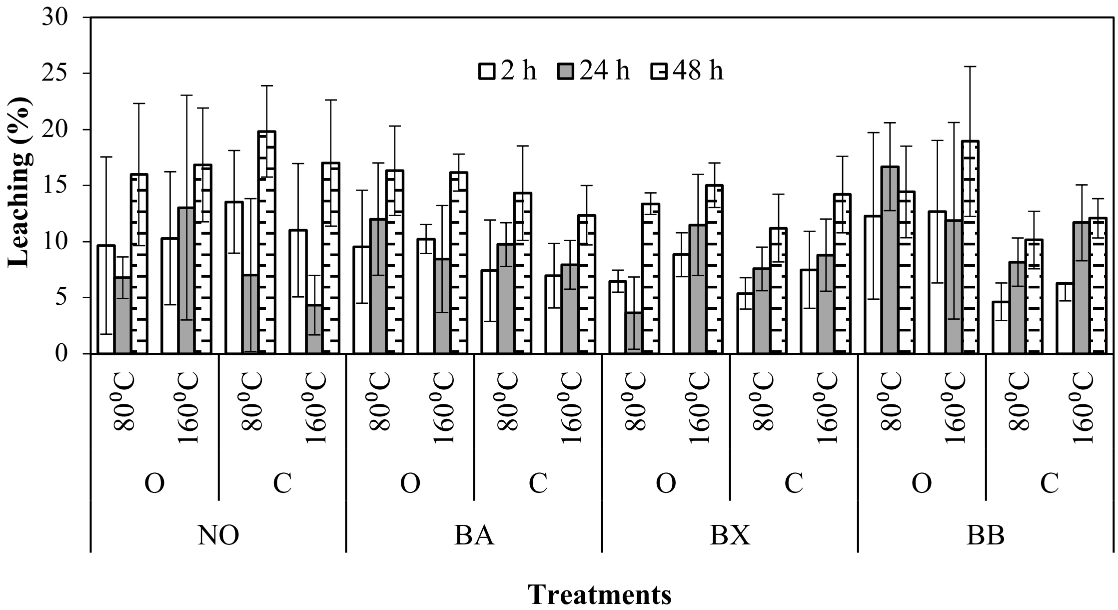

Fig. 3. Changes in leaching of samama wood after impregnation stage I, stage II, and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.4. Volume Expansion and Anti-Swelling Efficiency (ASE)

The impregnation of boron, citric acid, and heat modification have been proven to improve the dimensional stability of samama wood. First, it is indicated by the reduced volume expansion of samama wood in all test samples (**Fig. 4**). Next, it is demonstrated by the positive values of ASE for all treatments (**Fig. 5**). The highest ASE value (58.76%) is found in the sample M (boric acid+borax, and heat treatment at 80°C). The ASE value is statistically influenced by impregnation stages I and II. All boron impregnation treatments (whether in boric acid, borax, or a combination of both) have higher ASE values than treatments without boron. The hygroscopic characteristics of boric acid and borax are beneficial in managing water distribution in wood.

Citric acid impregnation also increases ASE and even better when compared to boron (samples G and K). As explained in water absorption parts, CA impregnation reduces the number of wood's OH groups, which potentially bond with water molecules. The impact is the decreasing swelling volume and the increasing ASE (Augustina et al. 2023; Priadi et al. 2023). Furthermore, the treatment combination of boron+citric acid also causes ASE to reach 52.91%.

Heat treatment at 160°C produces higher ASE than at 80°C. Samples experiencing this phenomenon are samples F, K, and L. This supports the statement by Bao et al. (2016) regarding the increased ASE of pine wood after treatments at temperatures ranging from 150°C to 180°C.

Meanwhile, Cheng et al. (2016) used an even lower temperature, 130°C. An increase in the hydrophobic properties of Chinese fir wood has occurred at this temperature. These changes in surface properties result in an improvement in dimensional stability.

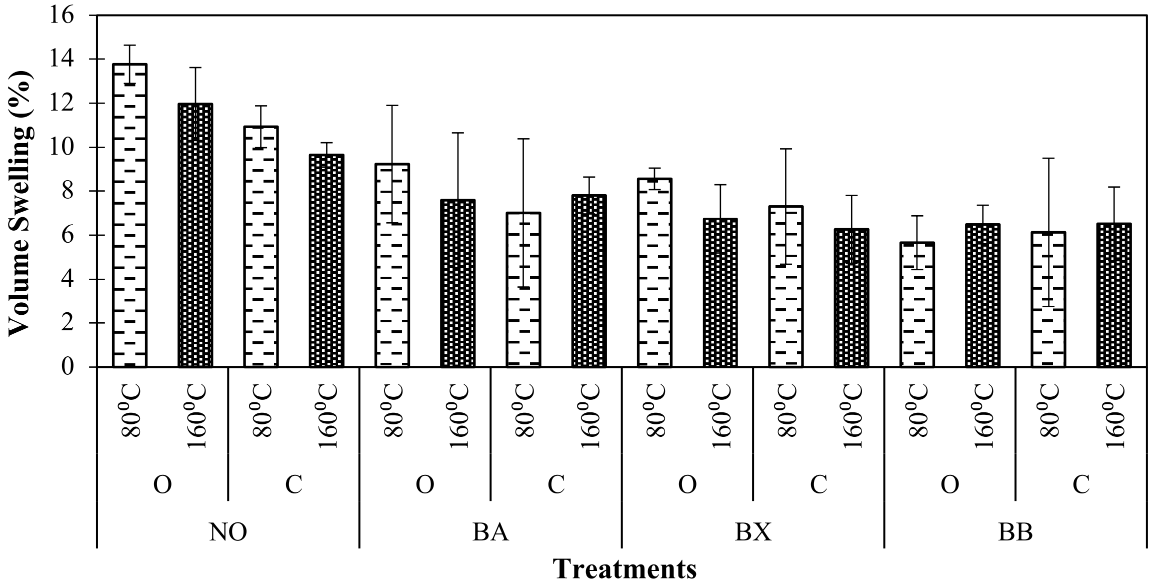

Fig. 4. Volume swelling of samama wood after two-stage impregnation and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

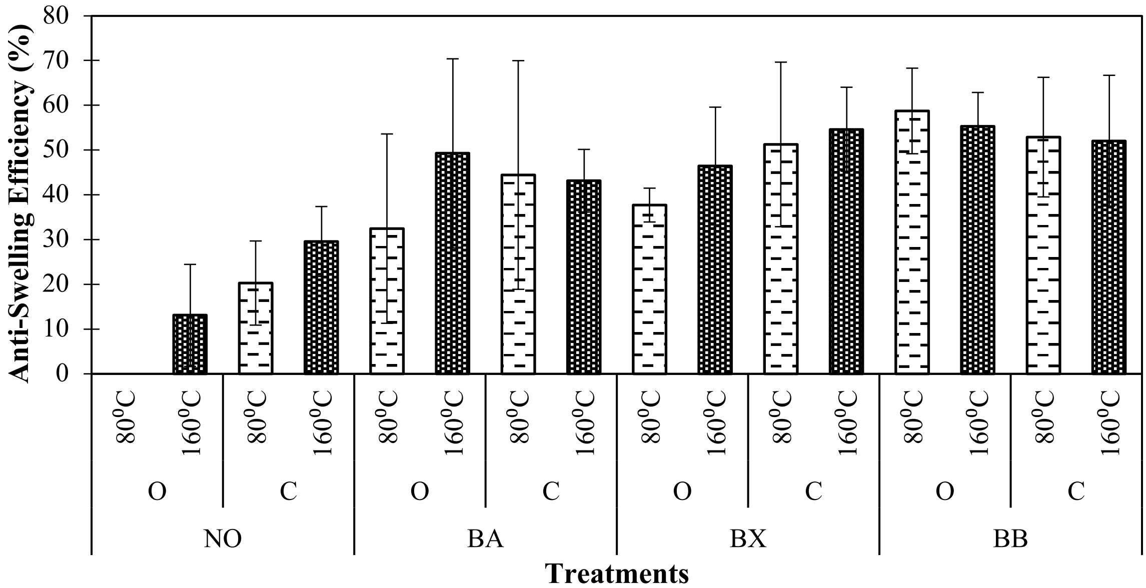

Fig. 5. Anti-swelling efficiency of samama wood after two-stage impregnation and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.5. Dimensional Shrinkage and T/R Ratio

Boron impregnation, citric acid and heat treatment have proven to reduce the dimensional shrinkage of samama wood. The best reductions are 59.02% for tangential shrinkage and 66.35% for radial shrinkage compared to the control (**Fig. 6**). The interaction of boron compounds and citric acid significantly influences tangential shrinkage. Additionally, radial shrinkage in samama is influenced by citric acid impregnation and the interaction between boron and HT (p-value = 0.35).

Fig. 7 shows that the T/R ratio decreases after treatment, except for samples G, M, O and P. When comparing impregnation solutions I and II, the T/R ratio ranges from the lowest to the highest: Boric Acid, Borax, and a combination of both. This is supported by the analysis of variance results showing that the T/R Ratio is closely related to boron and HT (p-value = 0.047). Citric acid bonds with hydroxyl groups in a wood decrease in the tangential direction. The impact is the decrease in T/R Ratio (Rumbaremata et al. 2019).

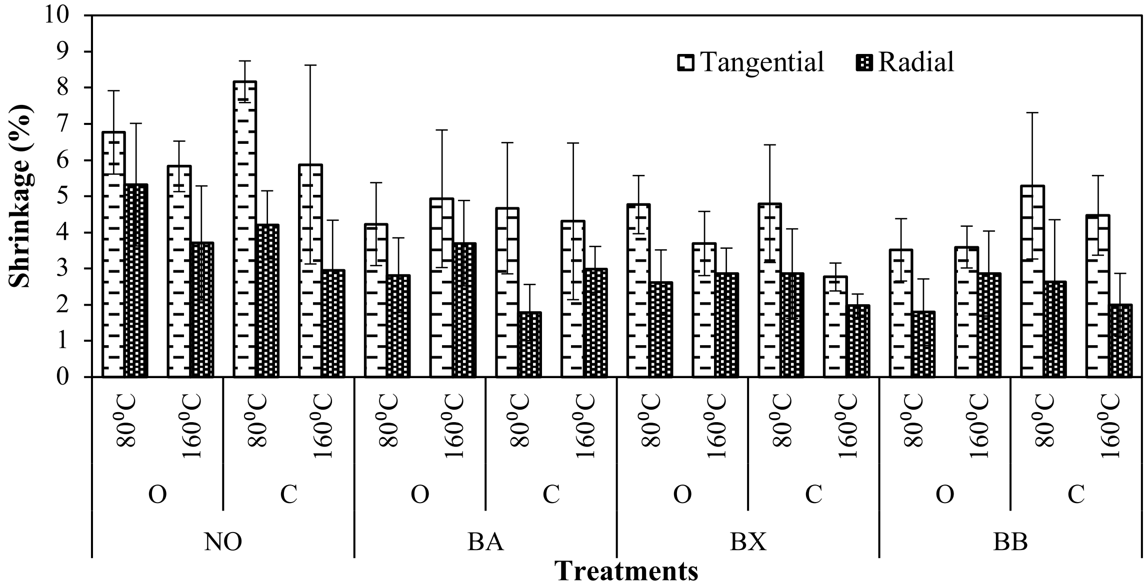

Fig. 6. Tangential and radial shrinkage of samama wood after two-stage impregnation and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

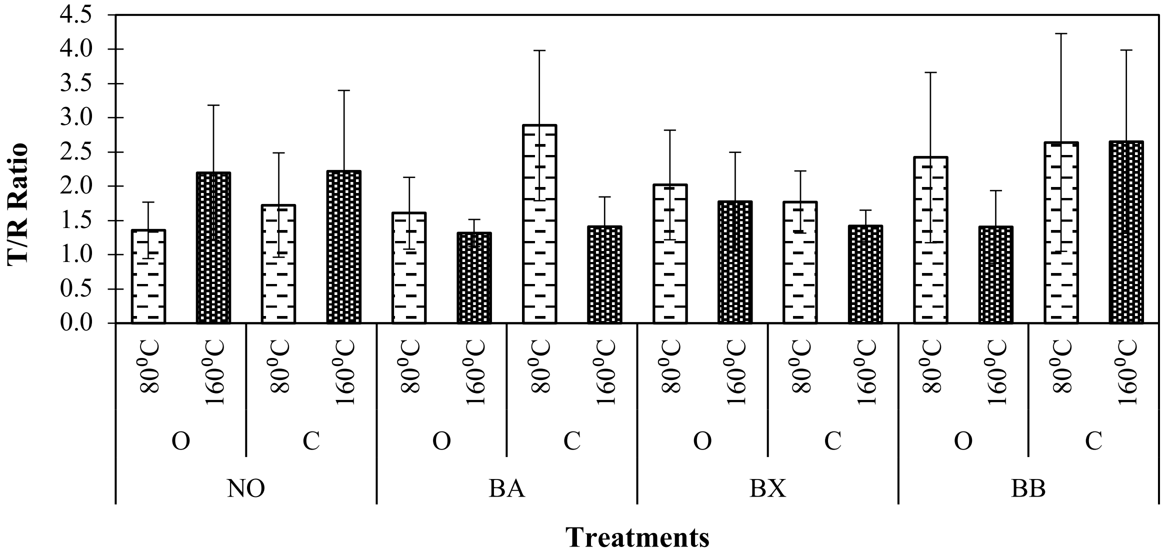

Fig. 7. T/R ratio of samama wood after two-stage impregnation and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.6. Surface Roughness

All impregnation and heat treatment processes increase the surface roughness (*Ra*) of samama wood (**Fig. 8**). The increase ranges from 12.07% to 31.05% compared to untreated samama wood. The analysis of variance results shows that both individual factors and the interaction of all factors (Boron, CA and HT) contribute to the increase in surface roughness (p-value < 0.0001). Further, Duncan's test results indicate that wood impregnated with boron has a higher *Ra* than the control. This is consistent with the findings of Can and Sivrikaya (2019) on the finishing surface of scots pine.

Impregnation stage II (citric acid) reduces the Ra of samama wood, especially in samples impregnated in stage I with boric acid + borax. Furthermore, Heat treatment as a single factor reduces Ra. Sandberg et al. (2017) reported that temperature correlates with changes in Ra. The reduction in Ra due to high temperatures (above 120°C) can be up to 35%. The cause is the biochemical constituents within the cell walls.

Surface roughness is closely related to finishing properties. A rough wood surface requires more finishing material than a smooth wood surface. Conversely, rough wood has the advantage of better adhesion during outdoor testing (Darmawan et al. 2018). Another factor dominating surface quality is the anatomical structure of the wood. Samama wood impregnated with boron and citric acid shows an increase in Ra, but the values still fall into the wood category with good finishing quality, comparable to untreated African and Merbau wood (Darmawan et al. 2020).

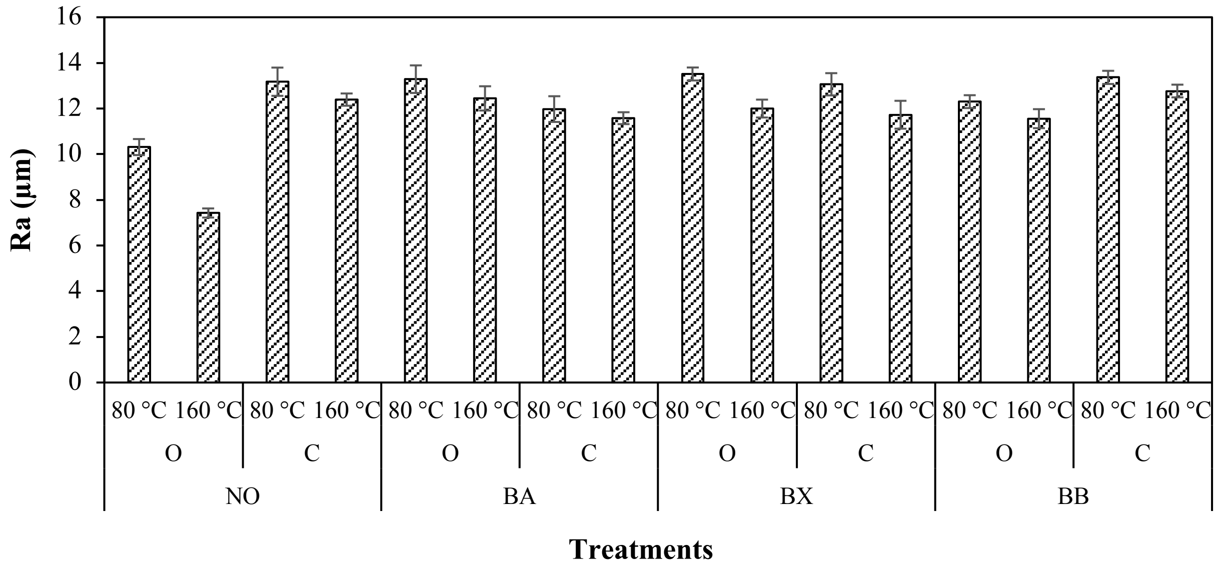

Fig. 8. Changes in surface roughness of samama wood before and after two-stage impregnation and heat treatment (NO = no boron, BA = boric acid, BX = borax, BB = boric acid+borax, O = no citric acid, C = citric acid).

3.7. Wettability

The research results indicate that the K value in wood impregnated with BA is lower than in the control wood, while wood impregnated with borax and the combination of boric acid+borax shows higher K values than the control wood. This indicates that borax treatment contributes to the hydrophobic properties of wood. Different chemical compositions lead to this phenomenon, as boric acid absorbs less water than borax (Kartal et al. 2020). Wood preserved with borax shows greater hygroscopicity than boric acid (Obounou-Akong 2015).

Conversely, the opposite phenomenon is found in citric acid impregnation. Citric acid has been shown to increase the *K* value. This is beneficial during the finishing process. Furthermore, heat treatment at 160°C results in a reduction in the K value. This happens due to a change in the pH of the wood surface (Žigon et al. 2023). Two-stage impregnation leads to better wetting of samama wood compared to the control. This treatment is expected to form strong bonds with finishing materials.

Table 2. Difference in K Value for various treatments

No.	Type	θi (°)	θe (°)	*K* (liter/second)
1	A	105.21	12.62	0.14
2	B	108.26	37.25	0.07
3	C	61.17	0.74	0.31
4	D	84.61	20.56	0.18
5	E	106.70	4.61	0.13
6	F	106.05	25.33	0.06
7	G	78.47	4.08	0.24
8	H	93.35	22.79	0.14
9	I	43.53	2.03	0.41
10	I	75.63	0.56	0.85
11	K	50.66	10.74	0.25
12	L	83.15	11.24	0.12
13	M	59.72	0.20	0.79
14	N	88.43	4.90	0.24
15	O	58.43	2.75	0.17
16	P	68.36	19.47	0.13

4. Conclusions

The dimensional stabilization of samama wood improves significantly after a two-stage impregnation with boron, citric acid, and heat treatment. Consistent treatment shows that the most effective approach for dimensional stabilization is the impregnation of samama wood with a combination of boric acid+borax, citric acid, and heat treatment at 160°C. In this study, the presence of citric acid implies a 30% reduction in boron leaching. Heat treatment provides two benefits: it enhances the adhesive properties of citric acid and contributes to the dimensional stabilization of samama wood. Another implication of heat treatment is the reduced surface roughness (*Ra*) of samama wood. While the impregnation of boron and citric acid increases *Ra*, the values still fall within the category of easily finishable. This is further supported by the increased wettability value observed after impregnation.

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