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The Hydrophilicity of Samama Wood Surface Quality after Boron, Methyl Methacrylate Impregnation, and Heat Treatment

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ABSTRACT

Indicators of surface roughness and surface-free energy can represent the improvement in wood surface quality. In this study, those two indicators were used to determine the change in the samama (*Anthocephalus macrophyllus*) wood surface following two modification steps, namely impregnation and heat treatment. The first step was boron impregnation in two forms applied separately, i.e., boric acid and borax. The second step was impregnation of methyl methacrylate followed by heat treatment at 90°C and 180°C. Surface roughness was determined following the ISO 4287:1997 standard, and surface free energy was analyzed using the Rabel Method. The results showed that the radial surface of samama wood naturally had a lower roughness than the tangential surface. Impregnation with boric acid, borax, and methyl methacrylate increased the wood surface roughness. However, heat treatment at 180°C tended to smooth the rough surface. Total surface free energy was altered after borax and methyl methacrylate impregnation. However, heat treatment seemingly withdrew the alteration. The polar components and dispersion contributed to total surface free energy with different compositions. In this study, the change in surface roughness was not congruent with the change in total surface free energy.

1. Introduction

Boron has long been used as a wood preservative, and the engineering of boron utilization has become an interesting topic (Ghani 2021; Nguyen et al. 2020). Boron can be easily applied to low-density hardwood and softwood (Cahyono et al. 2022b; Kartal et al. 2022). The main disadvantage of boron as a wood preservative is that it is only loosely bound in wood (Lloyd et al. 2020). Apart from being easily leached, boron is also susceptible to environmental issues. Thus, studies on boron are usually concurred with other more environmentally friendly materials. Those shortcomings could be controlled through physical engineering, chemical, mechanical, or combinations (Priadi et al. 2021, 2023; Salman et al. 2014).

The combination of physical and chemical methods that have succeeded in enhancing the boron fixation in the wood is the addition of bulk agents and heat treatment. Those methods could be applied separately and concurrently (Fadia et al. 2023). Boron in sugi wood (*Criptomeria japonica*) with a modified heat treatment was leached after 10 days of weathering period (Mendis et al. 2023). Heat treatment on Scots pine sapwood (*Pinus sylvestris*) and beech heartwood (*Fagus sylvatica*) at 150°C could retain boron by 2.7% and 4.1%, respectively. An increase in temperature up to 220°C increased the boron retention to 4.2% and 5.1% for both woods, respectively (Salman et al. 2014). Bulk agent impregnation followed by heat treatment is aimed to form a polymer from monomer compounds, e.g., styrene and methyl methacrylate (Stolf et al. 2017). Those two monomers were reported to have successfully impeded boron leaching. Furthermore, samama wood modified with boron, methyl methacrylate, and heat treatment showed more varied color gradation (Priadi et al. 2020). Heat treatment induced a darker color in white pine (*Pinus koraiensis*) and royal paulownia (*Pauwlonia tomentosa*) wood. This current study also confirmed that changes in wood color have become a consumer preference in choosing a type of wood (Hidayat et al. 2017). Tracking the success of wood quality impregnation generally only evaluates changes in physical, mechanical, and chemical component properties. Rarely does it proceed with identifying the surface quality. Therefore, further investigation of the modified wood's surface quality is necessary for effective wood finishing and adhesion.

Two parameters often used to determine surface quality are surface roughness and surface free energy (SFE). An SFE survey of 10 Indonesian wood species examined surface roughness, SFE, and wettability varnish on wood surfaces. The survey determined that the lower the SFE, the lower the wettability (Martha et al. 2020). In this regard, several studies have reported the relation between boron, surface roughness, and SFE. Nano-boron impregnation improved the surface quality of Scots pine (*P. sylvestris*) wood more effectively than boric acid (Can and Sivrikaya 2019; Tomak et al. 2018). Boric acid has been proven to improve the surface roughness of Scots pine, beech (*Fagus orientalis*), and chestnut (*Castanea sativa*) wood, thereby increasing the adhesion strength of these three types of wood (Ozdemir et al. 2015). Boric acid and borax repair the surface adhesion strength of varnishes used in woods. Furthermore, SFE was found to decrease in a poplar veneer after boron addition, although the difference was considerably insignificant (Li et al. 2013). Heat treatment reduced the SFE of radiata pine wood (Fu et al. 2019).

Samama (*Anthocephalus macrophyllus*) is a fast-growing tree. The mature wood of samama wood forms at the age of 8 years (Cahyono et al. 2015). It has a relatively low specific gravity (0.44); however, its quality can be easily engineered using compaction, impregnation, and gluing methods (Augustina et al. 2023; Rumbaremata et al. 2019). Nevertheless, those studies have not discovered the surface quality due to the treatments. This study on boron impregnation, methyl methacrylate, and heat treatment on samama wood aimed to examine the relationship between those aforementioned wood engineering methods with surface roughness and SFE components. Those components are rarely addressed, even though they are useful in determining the quality of gluing and wood finishing, especially after impregnation and polymerization.

2. Materials and Methods

2.1. Material Preparation

The samama wood used in this study originated from Curug Nangka, Sukajadi Village, Bogor Regency, West Java Province, about 16 km from Bogor City (6° 39' 56" S, 106° 43' 35" E). The samama trees were planted by the community in 2008 and were cut nine years after planting. Before the cut, the trees were selected based on the bole straightness and defect clearance. Three trees with a diameter of 39–42 cm and a clear bole height of 14–16 m were selected and cut.

Subsequently, it was followed by bole sortation that was started at the basal stem about 50 cm from above ground. That section was cut into a disc with a thickness of 20 cm. The discs were then brought to the laboratory and kiln-dried to a 10–12% moisture content. Test samples, 15 cm × 5 cm × 10 cm in size, were then made, and the radial and tangential surfaces were defined. The test samples were only taken from mature wood, which was at an 8 cm distance from the pit (Cahyono et al. 2015).

2.2. Boron Impregnation and Monomer Polymerization Processes

Boron impregnation was done in several steps. The first step was carried out using a pressure of 5 atm for 4 hours in a pressurized tube. The boron concentration was 5%, each in boric acid (trihydroxy-diboron) and borax (sodium tetraborate decahydrate). Following the step in the pressurized tube, the test samples were air-dried. The second impregnation stage was then conducted using methyl methacrylate with mepoxe M (methyl ethyl ketone peroxide) as the catalyst. The composition of methyl methacrylate and catalyst was 10:1 (v/v). This second impregnation was also done in the pressurized tube with the same pressure and time regime (5 atm, 4 hours). The next step was to heat the test samples in two temperature treatments, which were 90°C and 180°C for 4 hours.

2.3. Determination of Surface Roughness

The ISO 4287:1997 was used as the standard for the surface roughness test. Each surface (**Table 1**), radial and tangential, was tested on 5 points marked as x. The equipment used to measure the surface roughness in this test was a surface roughness tester (Surftest SJ-210 Series, Mitutoyo, Japan) coupled with the software USB communication tools for the portable surface roughness measurement. The testing direction was perpendicular to the fiber direction. The diameter of the used diamond tip was 5 mm, the tracing length was 6 mm, the speed was 0.5 mm/s, and the cut-off was 0.8 mm. The equipment measured the arithmetical average roughness (*Ra*), the root means square (*Rq*), and the average maximum height of the profile (*Rz*).

Table 1. Type of the surface based on treatments

Type of surface	Boron impregnation	Methyl methacrylate	Heat treatment (°C)
A	Untreated	Untreated	90
B	Untreated	Untreated	180
C	Untreated	Treated	90
D	Untreated	Treated	180
E	5% boric acid	Untreated	90
F	5% boric acid	Untreated	180
G	5% boric acid	Treated	90
H	5% boric acid	Treated	180
I	5% borax	Untreated	90
J	5% borax	Untreated	180
K	5% borax	Treated	90
L	5% borax	Treated	180

2.4. Determination of Dynamic Wettability

The surfaces of samama wood (radial and tangential) were dripped with four different liquids, i.e., aqua distillate, glycerol, methanol, and toluene. Each liquid was dripped three times at different points in a diagonal pattern. The first drip was on the bottom left (close to the observer), the second was on the center, and the third was on the top right (far from the observer). The drips were recorded by a USB camera (Dino-Lite, AM2111, Taiwan) with a 2-megapixel resolution. The duration of observation for each drop was 3 minutes, and 40x magnification was used. The data collected from the camera was videos in mpeg4 format that then was extracted into the motion of the liquid drips at 10 seconds duration for glycerol and aqua distillate, 1/20 second for methanol, and 1 second for toluene.

The contact angle was analyzed using the freeware Image-J Version 1.45 on the Windows 10 platform and drop analysis plug-in. The angle was the average value of the left and right contact angles. That value was then presented in a chart of contact angle gradation based on the time change (**Fig. 1**). The equilibrium of contact angle (θe) was determined using the S/G model (Cahyono et al. 2022a). The model was presented in Equation 1.

$$\theta = \frac{\theta_i . \theta_e}{\theta_i + (\theta_e - \theta_i) exp\left[K\left(\frac{\theta_e}{\theta_e - \theta_i}\right)t\right]} \quad (1)$$

where θ is the contact angle at a specific time, θi is the initial contact angle, and t is wetting time.

The model in Equation 1 was also used to determine the constant of the contact angle change rate (K). Both values were estimated using the Statistica Software Version 10 and the non-linear regression model.

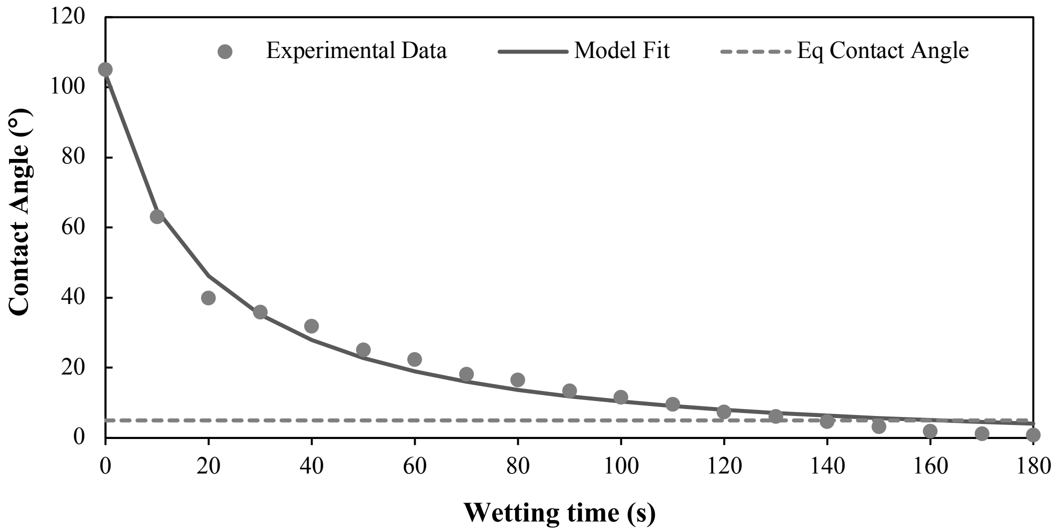

Fig. 1. Determination of the equilibrium contact angle (θe) using the S/G model.

2.5. Determination of Surface Free Energy (SFE) Component

Rabel Model was used to analyze SFE as presented in Equation 2 (Martha et al. 2020).

$$(1 + \cos\theta_e)\frac{\gamma_l}{\left(\gamma_l^d\right)^{1/2}} = (\gamma_s^d)^{1/2} + \left(\gamma_s^p\right)^{1/2}\left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{1/2} \quad (2)$$

where θe is the equilibrium contact angle, γ_l is the value of total surface tension, γ_l^d is dispersive surface tension, γ_s^d is a dispersive component of SFE, γ_s^p is the polar component of SFE, and γ_l^p is polar surface tension.

The Equation 2 is a linear regression (Y = A + BX). The Y and X components are presented in Equation 3 and Equation 4. Furthermore, the intercept (A) and slope (B) are presented in Equation 5 and Equation 6. The values of Y and X were calculated based on four SFE-measured standard solutions (**Table 2**). The total SFE is presented in Equation 7. The total gradation of SFE is presented as the percentage change of the total SFE (SFE Change) of the treated test sample (i.e., SFE) compared to the total SFE of the untreated test sample (i.e., SFE1) (Equation 8).

$$Y = (1 + \cos\theta_e)\frac{\gamma_l}{(\gamma_l^d)^{1/2}} \quad (3)$$

$$X = \left(\frac{\gamma_l^p}{\gamma_l^d}\right)^{1/2} \quad (4)$$

$$A = (\gamma_s^d)^{1/2} \quad (5)$$

$$B = (\gamma_s^p)^{1/2} \quad (6)$$

$$SFE = A^2 + B^2 \quad (7)$$

$$SFE\ Change = \frac{SFE_1 - SFE}{SFE_1} x100\% \quad (8)$$

Table 2. The surface tension component of some standard liquids (Yuningsih et al. 2019)

Liquids	γ_l^p **(mJ.m^{-2})**	γ_l^d **(mJ.m^{-2})**	γ_l **(mJ.m^{-2})**
Water	21.8	51	72.8
Methanol 50%	12.9	22.7	35.6
Toluene	2.3	26.1	28.4
Gliceryn	30	34	64

Notes: γ_l^p= polar component, γ_l^d= dispersive component, and γ_l= the value of total surface tension.

2.6. Data Analysis

The obtained data were analyzed with the factorial in completely randomized design. The first factor was boron application in three levels: 0%, 5% boric acid, and 5% boron. The second factor was methyl methacrylate application in two levels: 0% and 100%. The third factor was heat treatment at two temperature levels: 90°C and 180°C. The fourth factor was the surface of the wood, which consists of two levels, namely tangential and radial surfaces. After significant results were obtained, the data were further analyzed with the Duncan Test at a 5% significance level.

3. Results and Discussion

3.1. Surface Roughness

The tangential surface of the unimpregnated samama wood was noticeably smoother following the heat treatment at 180°C. Methyl methacrylate impregnation added the *Ra* of the tangential surface, and it appeared to have more smoothness following the heat treatment at 180°C (**Table 3**). The borax impregnation showed similar results to that of the methyl methacrylate impregnation. The combination of boric acid and methyl methacrylate increased the roughness of the tangential surface (type G). The increased *Ra* was also detected on the wood impregnated with borax, followed by methyl methacrylate treatment (type K). After heat treatment at 180°C, the wood surface impregnated with borax and methyl methacrylate showed a smoother appearance.

Similar to the tangential surface, the radial surface without methyl methacrylate and borax treatment showed a smoother surface following the heat treatment at 180°C. However, the wood surface type C and D displayed the opposite results. The combination of boric acid and heat treatment at 180°C (type F) exhibited a smoother surface than that of the type E surface (without heat treatment at 180°C). Similar results were also shown by the wood surface type H (boric acid + methyl methacrylate). Borax impregnation, methyl methacrylate, and heat treatment at 180°C presented a radial surface with the highest *Ra*.

Table 1. The surface roughness of the treated samama wood

Type of Surface	Tangential			Radial		
	Ra **(µm)**	***Rq*** **(µm)**	***Rz*** **(µm)**	***Ra*** **(µm)**	***Rq*** **(µm)**	***Rz*** **(µm)**
Untreated, 90°C	16.78 ± 2.42	20.77 ± 2.96	98.80 ± 14.59	15.02 ± 2.30	19.19 ± 2.56	91.77 ± 13.34
Untreated, 180°C	14.99 ± 0.97	19.13 ± 1.40	97.96 ± 9.36	14.68 ± 1.30	18.92 ± 1.92	86.67 ± 5.26
Untreated, MMA, 90°C	17.61 ± 1.07	22.25 ± 1.92	111.58 ± 15.40	15.42 ± 2.90	19.55 ± 3.14	93.35 ± 13.07
Untreated, MMA, 180°C	16.43 ± 1.83	20.83 ± 2.46	105.00 ± 15.29	16.33 ± 2.34	20.74 ± 2.90	94.29 ± 16.12
5% BA, 90°C	14.14 ± 1.76	18.06 ± 2.02	91.22 ± 9.24	18.26 ± 2.21	23.30 ± 3.21	104.78 ± 12.83
5% BA, 180°C	16.77 ± 3.51	21.08 ± 4.14	107.08 ± 17.03	17.08 ± 2.25	21.52 ± 3.30	96.87 ± 17.91
5% BA, MMA, 90°C	21.12 ± 1.40	26.62 ± 1.71	126.03 ± 10.95	18.19 ± 4.20	22.99 ± 4.64	105.01 ± 15.23
5% BA, MMA, 180°C	18.68 ± 1.80	23.65 ± 1.97	117.69 ± 7.41	17.45 ± 1.21	22.26 ± 1.81	98.53 ± 13.25
5% B, 90°C	17.94 ± 1.01	22.66 ± 1.64	113.10 ± 12.91	13.73 ± 1.81	17.39 ± 2.31	78.80 ± 9.64
5% B, 180°C	17.27 ± 1.81	21.68 ± 2.51	109.87 ± 15.64	16.16 ± 3.59	20.28 ± 4.56	88.36 ± 20.58
5% B, MMA, 90°C	21.12 ± 1.40	26.62 ± 1.71	126.03 ± 10.95	18.19 ± 4.20	22.99 ± 4.64	105.01 ± 15.23
5% B, MMA, 180°C	18.43 ± 2.21	23.25 ± 3.08	116.15 ± 16.38	18.36 ± 2.45	23.33 ± 3.00	110.99 ± 14.22

Notes: MMA is methyl methacrylate, BA is boric acid, and B is borax.

The analysis of variance showed that borax and methyl methacrylate impregnation affected the smoothness of the samama wood surface (**Table 4**). However, the radial and tangential surfaces responded differently to the treatment. Meanwhile, heat treatment was found to contribute less to the changes in surface roughness. Methyl methacrylate polymerization started at 90°C; however, no significant change was detected on the surface roughness when the temperature was increased to 180°C. The Duncan Test affirmed that the radial surface impregnated with borax without heat treatment (type I) had the smoothest surface. The tangential surface of samama impregnated with borax, methyl methacrylate, and heat treatment at 90°C showed the highest *Ra*, *Rq*, and *Rz*.

The radial surface of the untreated samama wood was smoother than the tangential surface of the same untreated wood. The different orientations of the ray cells on the radial and tangential surfaces cause different frictions between the ray cells and the saw blade. Following Methyl methacrylate impregnation, the roughness increased, and a smoother radial surface and tangential surface were consistently exhibited. Impregnation of bulking agents tends to enhance the roughness of the wood surface. This finding is in accordance with the previous study, which noted that bulking agents increased the surface roughness of fir wood (Harandi et al. 2020). Methyl methacrylate impregnation was noticed to have a less bulking effect in the tangential direction than in the radial direction of jabon wood (*Anthocephalus cadamba*) (Hadi et al. 2013). Borax impregnation increased the surface roughness of samama wood, particularly on the radial surface. The increase ranged from 6.7% to 15.2%. The formation of boric acid crystals on the wood surface contributes to the increase in surface roughness of pine, beech, and chestnut wood from 10.2% to 68.7% (Ozdemir et al. 2015). However, boric acid and borax did not show a significantly different effect. This finding confirmed the previous study in which no difference was observed in the surface roughness after boric acid and borax impregnation (Can and Sivrikaya 2019).

Table 4. Results summary of analysis of variance for surface roughness and SFE

Factors	Significance					
	Surface Roughness			SFE		
	Ra	***Rq***	***Rz***	**Polar**	**Dispersion**	**Total SFE**
Boron	0.002	0.002	0.011	0.000	0.012	0.000
MMA	0.000	0.000	0.000	0.000	0.322	0.000
HT	0.843	0.734	0.396	0.051	0.521	0.001
Surface	0.047	0.079	0.000	0.906	0.894	0.792
Boron*MMA	0.098	0.067	0.028	0.018	0.003	0.051
Boron*HT	0.214	0.248	0.138	0.670	0.667	0.382
Boron*Surface	0.011	0.009	0.046	0.921	0.832	0.772
MMA*HT	0.792	0.927	0.805	0.002	0.000	0.038
MMA*Surface	0.460	0.416	0.624	0.324	0.586	0.069
HT*Surface	0.353	0.378	0.973	0.518	0.076	0.873
Boron*MMA*HT	0.218	0.218	0.485	0.149	0.001	0.837
Boron*MMA*Surface	0.112	0.112	0.022	0.787	0.776	0.757
Boron*HT*Surface	0.012	0.011	0.026	0.208	0.290	0.115
MMA*HT*Surface	0.537	0.468	0.287	0.002	0.009	0.003
Boron*MMA*HT*Surface	0.770	0.778	0.867	0.574	0.898	0.421

Notes: Significance level = 0.05, MMA is methyl methacrylate, and HT is heat treatment.

3.2. Surface Free Energy

Table 5 demonstrates that the total SFE of the samama wood surface decreased following heat treatment at 180°C. The reduction was up to 30.49% on the radial surface, yet only 4.03% on the tangential surface. The reduction of total SFE was also detected after the wood was impregnated with methyl methacrylate (type C). The reduction of total SFE by 34.78% was noted in the radial surface of samama wood impregnated with methyl methacrylate and heat treatment at 180°C (type D). Moreover, the tangential surface was found to have a smaller reduction of total SFE compared to the radial surface.

The boric acid impregnation reduced the total SFE. The reduction was bigger after heat treatment at 180°C treatment (type F). The radial surface type G (boric acid and methyl methacrylate impregnation) exhibited an identical total SFE with the radial surface type F. The reduction of total SFE in the radial surface type H (boric acid, methyl methacrylate, heat treatment at 180°C) was slightly higher than that of type F. Nevertheless, all radial surfaces of samama wood impregnated with boric acid showed a higher total SFE change than the tangential surfaces.

The total SFE of Samama increased following borax impregnation. The phenomena were observed on the surface types I, J, and K. The increased total SFE contributed to the polar components, while the dispersion components tended to be stable. This tendency was confirmed by ANOVA results, which showed the significant effect of boron impregnation on polar and dispersion components and total SFE ($p \leq 0.05$). Previous studies noted that borax impregnation, methyl methacrylate, and heat treatment at 180°C caused the increase in the lignin content of samama wood. The polar and non-polar characteristics of lignin contribute to the change in total SFE (Cahyono et al. 2022b; Croitoru et al. 2018; Moore et al. 2015).

The ANOVA showed that boron impregnation, methyl methacrylate, and heat treatment impacted the total SFE (**Table 4**). Meanwhile, the surface factors responded otherwise. In contrast, there was a different *Ra* response between the radial and tangential surfaces. Furthermore, the *Ra* was not affected by heat treatment. Therefore, no concordance was observed between total SFE and *Ra* (**Fig. 2**). The linear regression between the total SFE and *Ra* showed a small coefficient of determination (R^2= 0.011).

Table 5. The total SFE of the samama wood following the treatments

Type	Surface	SFE ($mJ.m^{-2}$)			Total SFE Change (%)
		Polar	Dispersion	Total	
A	R	45.7	13.1	58.9	
	T	33.1	18.1	51.2	
B	R	18.8	22.1	40.9	30.49
	T	33.5	15.7	49.2	4.03
C	R	18.5	20.0	38.5	34.55
	T	22.8	22.5	45.3	11.54
D	R	22.9	15.5	38.4	34.78
	T	21.7	16.5	38.2	25.48
E	R	33.6	16.9	50.5	14.26
	T	26.3	19.2	45.5	11.20
F	R	16.9	24.8	41.7	29.20
	T	19.0	23.3	42.4	17.27
G	R	18.6	22.7	41.3	29.89
	T	21.9	21.6	43.5	15.09
H	R	29.3	13.2	42.5	27.88
	T	28.6	14.9	43.5	15.02
I	R	49.2	13.2	62.4	-5.98
	T	41.9	15.8	57.7	-12.60
J	R	40.1	16.5	56.6	3.86
	T	40.5	12.5	52.9	-3.37
K	R	26.1	19.0	45.0	23.50
	T	39.6	17.0	56.7	-10.64
L	R	33.0	18.4	51.5	12.53
	T	26.7	19.5	46.2	9.80

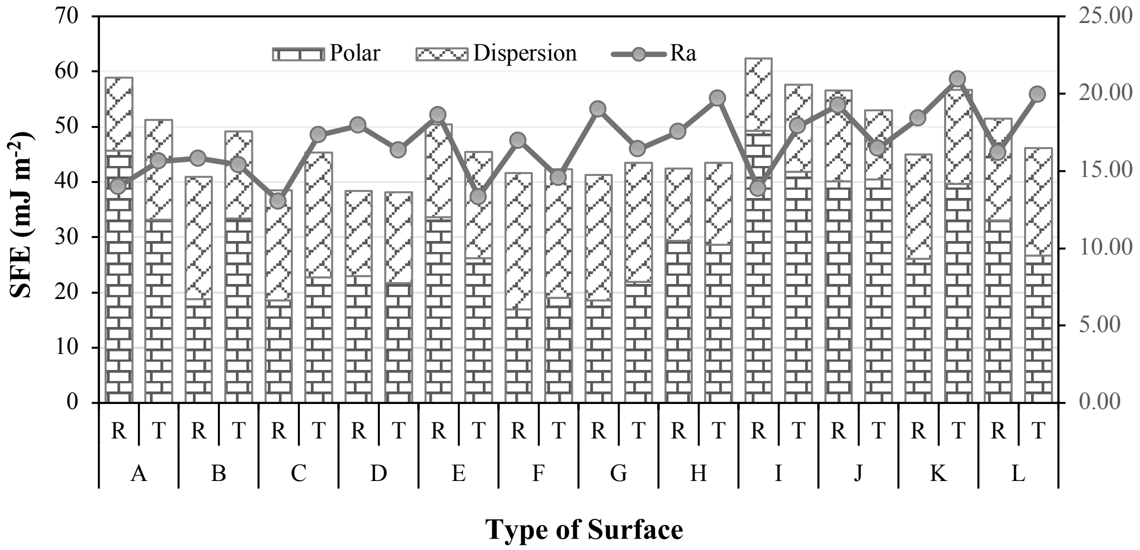

Fig. 2. The total SFE and *Ra* of samama wood.

The gradation of total SFE of samama wood following boron and methyl methacrylate impregnation indicated an enhanced surface quality for further processing, i.e., finishing. The highest total SFE (62.4 mJ.m^{-2}) was noted on the radial surface of type I, and the lowest was observed on the tangential surface of type D (38.2 mJ.m^{-2}). A study by Darmawan et al. (2020) noted that African wood (*Maesopsis eminii*) with a total SFE of 53.61 mJ.m^{-2} produced the best varnishing bond quality. However, boron impregnation has also been reported to reduce the bond strength of beech wood (*F. orientalis*) plywood with up to 17.2% UF adhesive.

4. Conclusions

Boric acid, borax, and methyl methacrylate impregnation increased the roughness of tangential and radial surfaces of samama wood. Heat treatment at 180°C enhanced the surface smoothness of untreated- and methyl methacrylate-impregnated-samama wood. The boric acid, borax, and methyl methacrylate impregnation also altered the polar, dispersion, and total SFE components. The polar component dominated the change in total SFE. The radial surface impregnated with borax produced the highest total SFE, while the lowest was noticed in the tangential surface impregnated with methyl methacrylate, followed by heat treatment at 180°C. These results affirm that there is no relationship between surface smoothness and the total SFE of the modified-samama wood.

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