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PERFORMANCE OF FILM-FORMING FINISHES ON THERMALLY MODIFIED BOLO (*GIGANTOCHLOA LEVIS*)

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The effect of thermal modification on the finishing properties of the bamboo species, bolo (*Gigantochloa levis*), was investigated. Bolo was thermally modified in steam at 175 and 200 °C for 30 minutes, followed by the application of Nitrocellulose lacquer (NC), polyurethane (PU) and water-based PU (PUW). Thermal modification lowers pH, gloss and degree of lightness (L*) and increases dry film thickness. On the other hand, the adhesion property and gouge hardness of solvent-based finishes such as NC and PU significantly improved with heat treatment at 175 °C. At 200 ˚C, the hardness of both topcoats further improved, along with the adhesion property and acid resistance of PUW. Scanning electron micrograph revealed an improved adhesion bond on NC and PU applied on thermally modified at 175 °C.

Keywords: Finishing, nitrocellulose lacquer, polyurethane, thermal modification, water-based polyurethane

INTRODUCTION

Thermal modification or heat treatment is the process of subjecting the wood to high temperatures in the absence of oxygen which results in chemical changes in the material (ITWA 2003, Homan and Jerissen 2004, Jirous-Rajcovic & Miklesic 2019).

The technology exists for two decades now and is known to enhance the dimensional stability and durability of wood, darken its color and decrease its hygroscopicity (Militz 2002, de Moura et al. 2012, Miklesic & Jirous-Rajkovic 2016). Improvement in wood properties is attributed to changes in the chemical structure of the heat-treated wood (Militz 2002, de Moura et al. 2012, Herrera et al. 2015, Miklesic & Jirous-Rajkovic 2016).

Thermal modification is done in steam, nitrogen or oil environment ranging from 160 to 260 °C (Esteves & Pereira 2009, Militz 2002). Thermal modification using steam is considered eco-friendly since it generates no waste, uses no chemicals and requires only heat and water as inputs (ITWA 2003).

Most studies on thermal modification deal with the physicomechanical properties of treated wood and durability tests on various tree species, including *Abies nordmanniana, Eucalyptus globulus, Fagus sylvatica, Fraxinus excelsior, Pinus pinaster, Picea abies, Polyscias nodosa, Quercus robur, Corymbia citriodora* and *Pinus taeda* (Esteves et al. 2007, Jimenez et al. 2011, Candelier et al. 2013, Herrera et al. 2015, Kesik et al. 2015, Miklesic & Jirous-Rajkovic 2016, Herrera et al. 2018, Gaff et al. 2019, Paes at al. 2021).

Bamboo has been promoted as an alternative material to wood (Chaowanna 2013). In recent years, studies on the thermal modification of bamboo have also been conducted. In the Philippines, for example, *Bambusa spinosa* (kauayan-tinik) has been successfully heat-treated using steam at 150 and 200 °C for 30 and 60 min (Natividad & Jimenez-Jr 2015). Likewise, *Bambusa blumeana* and *Dendrocalamus asper* has also been thermally modified in a steam environment at 175 and 200 °C for 30 min, with 175 °C imparting improved dimensional stability (Jimenez et al. 2021).

In Malaysia, *B. vulgaris* and *Gigantochloa scortechinii* were treated with oil as a heating medium using variable temperatures and treatment durations (Wahab et al. 2005, Wahab et al. 2019). Both species showed a reduction in strength properties. This concurred with the report by Manalo and Acda (2009), where a reduction of the strength of *B. blumeana, B. spinosa* and *D. asper* was observed upon hot oil treatment using virgin coconut oil at 160–200 °C for 30 to 120 min.

Oil treatment of *G. levis* (bolo) at 180 °C for 60 min showed improvement in its physical and mechanical properties and durability (Wahab et al. 2016). Changes due to the thermal modification of bamboo, such as bolo, could affect the performance of the applied finish.

Heat-treated wood has been reported to be a good substrate for various coating materials (Jirous-Rajcovic & Miklecic 2019). A study on malapapaya showed that it could be coated with either synthetic or natural finish, with no significant difference in adhesion strength between thermally modified and unmodified wood (Jimenez & Palisoc 2012). On the other hand, European ash thermally modified at 212 °C under steam and finished with UV-curable and water-borne coatings displayed enhanced adhesion properties. Moreover, a change in wood color is a welcome result as it eliminates the need to apply stain to accentuate the material's aesthetic property or change any light shade. Every wood and bamboo species perform differently based on the coating materials used. The type of finish to be applied depends on the client's preference.

The Philippine market is dominated by solvent-based wood coatings like nitrocellulose lacquer (NC) and polyurethane (PU). Known for its excellent rubbing quality, NC is quick-drying, low-cost and can be easily applied and repaired. On the other hand, PU is known for its film hardness, wear and scratch resistance, and resistance to many solvents, acids and alkalis (Rines 2020). Between the two, NC is most commonly used by local small and medium enterprises in the furniture and handicraft sector. To reduce the volatile organic contents (VOCs) found in wood finishes, manufacturing companies have introduced water-based clear coats like polyurethane (PUW) into the market. These are expected to have almost the same properties as their solvent-based counterparts minus the strong smell and the hazardous effects on human health and the environment (Charron 1998).

In this study, three commercial film-forming finishes (NC, PU and PUW) were studied to determine the effect of thermal modification at two different temperatures (175 and 200 °C) on the finishing properties of bolo bamboo. Bolo was selected since it is one of the economically important bamboo species in the Philippines (PCAARD 1991). It is used in making furniture, handicrafts, baskets and engineered bamboo (Rojo et al. 2000, Cabangon 2014). This study presents the improvement in the finishing property of thermally modified bolo.

MATERIALS AND METHODS

Bamboo collection and thermal modification

Bolo was collected from the Carolina Bamboo Garden, a bambusetum in the town of Antipolo, Province of Rizal, Philippines (20 km east of Manila). Mature bolo aged 3–4 years were used in this study. Each bamboo pole was cut into 1.2 m long samples prior to thermal modification.

Samples were thermally modified in the FPRDI lab-scale pressurised steam heat treatment chamber at 175 and 200 °C for 30 min. The temperature in the chamber was increased gradually until the desired treatment temperature was attained. Steam pressure was kept at 206 kPa throughout the treatment process. For the purpose of comparison of finishing performance, kiln-dried bolo samples were prepared to serve as control.

The pH determination

Aqueous extracts of kiln dried and thermally modified bolo for pH determination were prepared based on Pedieu et al. (2007) method. Scraped samples were hammermilled and then oven-dried for 24 hr. Five grams of dry samples were then refluxed in 25 ml of deionised water for 20 min and then filtered with filter paper. After the extracts had cooled to room temperature, their pH was measured using a bench-type pH meter.

Sample preparation and finishing

Thermally modified and kiln-dried bolo internodes were cut into lab-size samples (approximately 17 cm × 7 cm). All the specimens were then scraped using a knife and sanded at 100-180-320 grits schedule prior to finishing.

The finishes comprised three different liquid coatings prepared following the manufacturers' instructions and sprayed on the bamboo samples using the straight finishing system. The characteristics of the commercial finishes, after thinning, were determined (Table 1).

Table 1 Characteristics of the commercial finishes used and environmental condition during finish application

Finish	Solid content (%)	Density (g ml^{-1})	Viscosity (cP)	Condition during finish application (ambient)	
				Temperature (°C)	RH (%)
Nitrocellulose lacquer (NC), solvent-based	71.33	0.8991	41	31	65
Polyurethane (PU), 2-component	102.33	0.9576	25	29	69
Water-based polyurethane (PUW), 1 component	100.00	1.0320	106.67	31	67

Optical, physical and performance properties testing

Finished bolo samples were conditioned inside the laboratory for 1 month prior to physical and performance testing of the applied finishes. Gloss was measured using polygloss following ASTM D 523-14 (2017). Gloss at a geometry of 60 ° incidence angle scaled on black glass standard of 100 GU was reported. On the other hand, color was determined using spectrocolorimeter at a geometry of 45/0 using a light source of 8* visible wavelength and D65/10 illuminant. The color was expressed via CIE L*a*b*. Five readings were taken per sample for both gloss and color on 10 replicates per finish.

Thickness was established using a thickness gauge following ASTM D6132-13 (2017). A pea-sized amount of couplant was placed in the area to be tested. The thickness gauge was pressed and the measurement was taken. Five sets of measurements were taken per sample with 10 replicates.

The adhesion property was evaluated via the Adhesion by Tape Test Method, ASTM D3359-17 (2017). Three sets of lattice-cut forming 100 squares (2 mm × 2 mm) were made on the samples. A masking tape was pressed on the grid and removed after 2 min. Percentage of coating removed from the substrate was reported. Adhesion was tested in 10 replicates.

Film hardness was tested via Pencil Hardness Method, ASTM D3363-20 (2017). Wood pencils with different hardness levels (6H-5H-4H-3H-2H-H-F-HB-B-2B-3B-4B-5B-6B) were pushed onto the finished surface with the aid of a Pencil Hardness Tester. The tester had a load of 750 ± 10 g and a pencil inclined at 45 ± 1°. The gouge hardness of the films was determined in 10 replicates.

Lastly, the resistance to liquids test was conducted following ISO 4211-79 (1979) using liquids such as 44% acetic acid, 48% ethyl alcohol and coffee. An absorbent pad was soaked in the test liquids, then placed on the finished samples and covered with glass. After 1 hr, the glass cover was removed and left to stand for another 30 min before removing the test pad. After 24 hr, the panels were cleaned using a cleaning solution. The test area was examined, and marks left by the liquids were assessed on a scale of 5 (no damage) to 1 (finish wholly or partially removed).

Microstructure testing

Finished bolo samples were selected randomly to observe the characteristics of applied finishes on thermally modified bolo with scanning electron microscope (SEM). A 0.5 × 0.5 cm sample was cut from a finished kiln-dried and treated bolo, then coated with a highly conductive gold-palladium film. The cross-section of the samples was subjected to SEM with a high vacuum mode, with an accelerating voltage of 5 kV.

Statistical analysis

Statistical analysis using a two-factor factorial in completely randomised design (CRD) with treatment and type of finish as factors was used for the following properties: gloss, color, thickness, adhesion and film hardness, while Kruskal Wallis Test was used for resistance to liquids. Tukey's Honestly Significant Difference (HSD) was used to group means if found significant. Analysis was done using SAS Analytics Pro 2017.

RESULTS AND DISCUSSION

Analysis of variance (ANOVA) was performed to check the differences in the interaction of treatment and finish. Statistically, the comparison revealed that degree of lightness (L*), gloss, dry film thickness, adhesion and gouge hardness were highly significant (p = < 0.0001), while the a* (red/green hue) and b* (yellow/blue hue) were non-significant. For resistance to liquids, the Kruskal-Wallis test showed that only resistance to acid was highly significant (p = < 0.0001), and coffee and alcohol were non-significant. There were also significant differences in the pH upon thermal modification (p = < 0.0001). Generally, there was a decrease in L* and an increase in gloss and dry film thickness with the increase in treatment temperature. In terms of the performance of applied finishes, adhesion and resistance to acid improved at 175 °C. While the gouge hardness improved as the treatment temperature increased.

Effect of thermal modification on pH and color

One of the expected results that can easily be tested on heat-treated wood is the change in pH (ITWA 2003). Table 2 shows the pH change in the bamboo samples due to thermal modification. Untreated bolo and thermally modified at 175 °C (TM175) showed slightly basic pH at 7.96 and 7.85, respectively.

As the steam temperature rose to 200 °C, the samples became slightly acidic. This means that the heightened treatment temperature correlated to the acidity increase. While there was a significant difference among the three treatments (p = < 0.0001), major pH changes occurred at 200 °C. Change in pH is associated with the release of acetic acid during treatment which is the precursor of the chemical change in the cellulose component of wood (ITWA 2003, Herrera et al. 2015, Miklesic & Jirous-Rajcovic 2016).

Table 2 The pH values of bolo samples

Treatment	pH	Tukey's grouping
Unmodified	7.96	A
TM-175	7.85	B
TM-200	6.40	C

Average of 5 replicates, means with the same letter are not significantly different at a 99% confidence level

Visual inspection showed that the treated bolo turned darker as the temperature increased (Figure 1). The test of degree in lightness supported the visual observation as shown by the reduced L* with higher steam temperature (Figure 2).

Effect of thermal modification on optical and physical properties

Table 3 presents the effect of thermal modification on the L*, gloss and dry film thickness of samples applied with different clear finishes. According to studies, treatment temperature and time affect the color of thermally modified wood (Miklesic & Jirous-Rajcovic 2010, 2016, Herrera et al. 2015, 2018). ANOVA showed that the type of finish had no significant effect on L* while the treatment and interaction of treatment and finish had highly significant effects at a 99% level of confidence. Regardless of the finish, all control samples gave lighter color as reflected by higher L* values of 47.17, 46.65 and 45.67 for NC, PU and PUW-coated specimens, respectively.

As thermal modification was applied and temperature increased, the color of the finished bolo became darker, i.e., L* values went down for both NC and PU. Meanwhile, PUW-finished samples recorded an increased L* at TM200. During treatment, some poles of bolo TM200

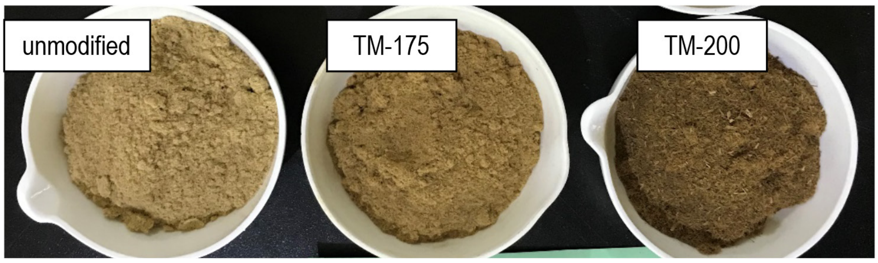

Figure 1 Variation in color of hammermilled samples due to different treatment temperatures

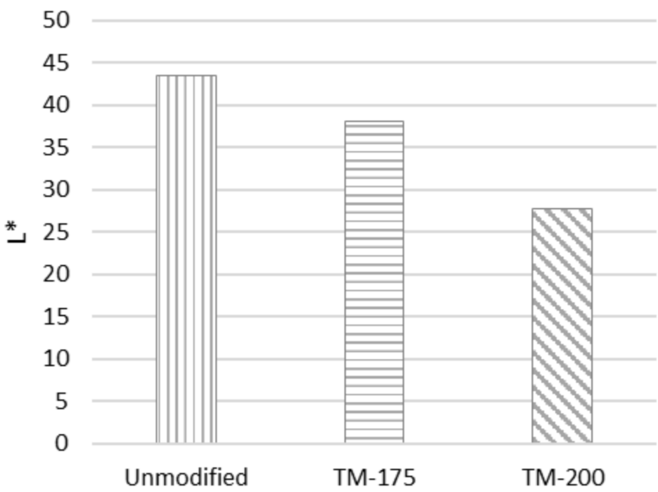

Figure 2 Change in the degree of lightness (L*) of hammermilled thermally modified bolo

Table 3 Optical and physical properties of clear finishes applied on untreated and thermally modified bolo

Finish	Treatment	L*	a* (red/green hue)	b* (yellow/blue hue)	Gloss (GU)	Classification	Film thickness (μ)
NC	Unmodified	47.17a	7.45	32.88	72.04b	Glossy	34.14a
	TM-175	37.78c	13.63	32.38	58.84c	Semi-gloss	39.71b
	TM-200	39.22c	12.14	35.11	21.91de	Semi-matte	53.44c
PU	Unmodified	46.65ab	8.08	32.13	82.53a	Glossy	32.81a
	TM-175	38.23c	12.74	32.61	80.51a	Glossy	59.91d
	TM-200	30.37d	14.35	32.39	24.97d	Semi-matte	88.92e
PUW	Unmodified	45.67ab	8.83	30.03	58.29c	Semi-gloss	32.47a
	TM-175	24.84d	11.37	23.12	15.14f	Semi-matte	32.31a
	TM-200	40.61bc	11.47	32.11	19.09ef	Semi-matte	56.29f

* = Average of 10 replicates, for each property, means with the same letter are not significantly different at a 99% confidence level

were almost carbonised, especially those near the edge of the chamber. These samples were deemed too dark to be applied with the finish. Only uncarbonised bamboo poles were used for the finishing experiment.

Red/green hue (a*) and yellow/blue hue (b*) all showed positive values, which means all sample colors were located in the first quadrant. Unmodified bolo showed the lowest red hue values for all types of finishes. The change in color is associated with the products degraded during oxidation (Herrera et al. 2015, Korkut et al. 2013). Likewise, according to ITWA (2003), the transformation of lignin starts at 120–220 °C, which is responsible for the change in color. The darker and more stable color of thermally modified samples could mean reduced finishing costs as fewer pigmented stains need to be applied.

ANOVA showed that finish, treatment and combination of finish and treatment significantly affected gloss ($p = < 0.0001$). The glossy surface of NC and PU-finished bolo turned semi-matte with TM200, similar to the semi-gloss of PUW with TM175 and TM200. Heat treatment results in lower gloss as it causes substrates to take on a darker color. The darker the substrate, the less light it reflects back resulting in lower gloss. While consumers have different preferences on the gloss level of finished samples, they should be aware that the lower the gloss, the harder it is to hide surface imperfections.

Film thickness tended to expand with samples treated at higher temperatures, except for PUW, which showed a notable increase only at 200 °C. The increasing thickness could be due to the chemical changes in bamboo during thermal modification. The thermally modified wood's cell wall is permanently swollen (Homan and Jorissen 2004). In the present study, the first coat of the finish may already have been enough to fill the longitudinal grains of dimensionally stable bolo, thus resulting in a thicker film with each succeeding coat. For the furniture or handicraft producer, this could mean reduced finishing costs as a thicker film can be achieved using fewer coats.

All the finishes applied on unmodified bolo showed no significant differences, but PU exhibited the highest film thickness at both temperatures. This could be due to the solid content of PU, which, after thinning, was still high at 102.33% (Table1).

Coating adhesion performance

Adhesion is the most important finishing property as it determines how long the coating can protect a substrate. ANOVA showed that the finishing material and heat treatment interaction significantly affected the adhesion property ($p = < 0.0001$). Tukey's grouping of means showed significantly improved adhesion for NC and PU at 175 °C and PUW at 200 °C (Figure 3). Bolo treated at 175 °C and finished with NC gave the best adhesion value.

The thermal modification increases the water contact angle, which makes treated wood hydrophobic (Miklesic & Jirous-Rajcovic 2016, Karlinasari et al. 2018). Hydrophobicity affects wood's coating absorbency and adhesion (Esteves and Pereira 2009). In this case, the adhesion bond of hydrophobic finishes like NC and PU with the hydrophobic surface of TM175 bolo enhanced film adhesion. However, a further increase in temperature reversed this effect. This suggests that the optimum temperature for NC and PU finishes is at 175 °C. This concurs with the findings of Gaff et al. (2019) that optimum improvement of wood properties is achieved below 200 °C.

The PUW, a water-based finish, achieved the same level of improved adhesion as solvent-based PU at TM200 (Figure 3). This was not expected since studies have shown that contact angle is directly proportional to treatment temperature (Herrera et al. 2015, Miklecic & Jirous-Rajkovic 2016, Karlinasari et al. 2018). For a water-based finish, better adhesion is expected on untreated samples. Nevertheless, current results concurred with Herrera et al. (2015) cross-cut test on the water-borne finish, which showed enhanced film adhesion for thermally modified *Fraxinus excelsior.* The improvement is proportional to the increase in treatment temperature.

In the present study, the increase in acidity could have triggered this effect on PUW. At TM200, the bamboo samples underwent substantial pH changes, favoring water-based PU adhesion. In the same way, the chemical changes may also have reinforced PUW penetration, resulting in a thicker finish and better adhesion.

Film hardness

Another important property of applied finishes is durability or the ability of a finishing material

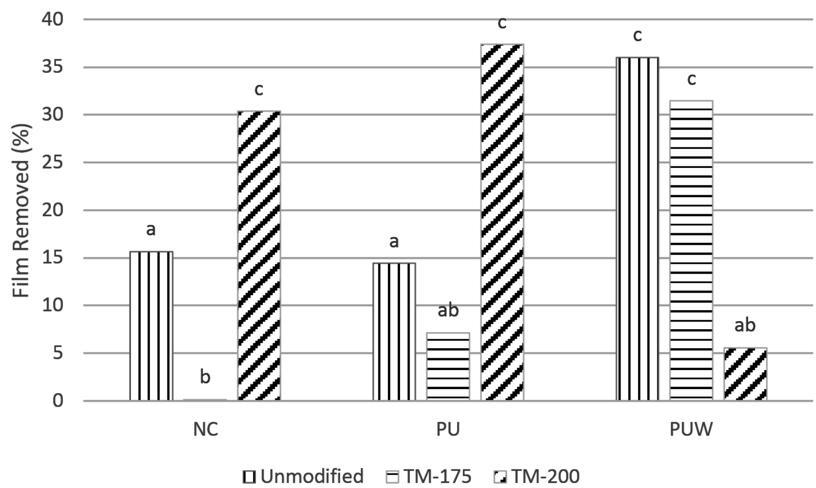

Figure 3 Adhesion performance of applied finishes on unmodified and modified bolo

to resist being damaged. This can be tested via the pencil hardness test, which determines the hardest pencil that leaves the film uncut or unscratched. ANOVA showed that the interaction of finish and temperature significantly affected gouge hardness (p = < 0.0001). For all finishes, the control gave the lowest gouge hardness (Figure 4). For NC and PU-finished TM samples, hardness increased with temperature. The PU applied on TM200 bolo gave the highest hardness.

Resistance to liquids

Neither coating material nor thermal modification significantly affected resistance to ethyl alcohol (Table 4). This was not true, however, with resistance to coffee and acid. The NC-finished TM 200 showed slightly lower resistance to coffee, while PUW samples showed the least resistance to acid, with the mean rating not even reaching 2 for the unmodified in both temperatures. There was, however, a slight improvement with thermal modification.

Results showed that the test with acid had no observable effect on PU (Figure 5). This means that thermal modification did not affect PU, which is already known for its high resistance to acid, alkalis and other solvents. Meanwhile, the solvent-based NC showed a slight increase in acid resistance at TM175.

As the pH of bamboo samples decreased at TM200, the coating's resistance to the acid of an already acidic substrate may have worsened it. The NC at TM175, on the other hand, was still slightly basic, and this contributed to the coating's acid resistance. This further supports the conclusion that TM175 is the optimum treatment for improving the finishing properties of bolo.

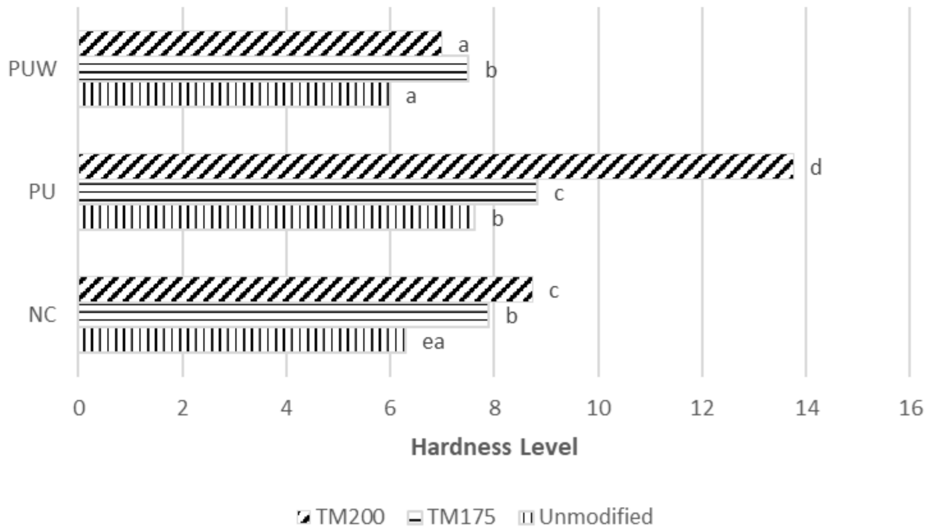

Figure 4 Effect of thermal modification on film hardness, average of 10 replicates

Table 4 Resistance to three types of liquids of different finishes applied on thermally modified bolo

Finish	Treatment	Coffee	Ethyl Alcohol	Acetic Acid
NC	Unmodified	5.0a	5.0	3.2a
	TM-175	5.0a	5.0	3.9b
	TM-200	4.8b	4.9	3.1a
PU	Unmodified	5.0a	5.0	4.9c
	TM-175	5.0a	5.0	5.0c
	TM-200	5.0a	5.0	5.0c
PUW	Unmodified	5.0a	5.0	1.0d
	TM-175	5.0a	5.0	1.8e
	TM-200	5.0a	5.0	1.6e
	Chi-square	35.2111*	9.8314ns	85.717*

* = Average of 10 replicates, for each type of liquid, means with the same letter are not significantly different at a 99% confidence level

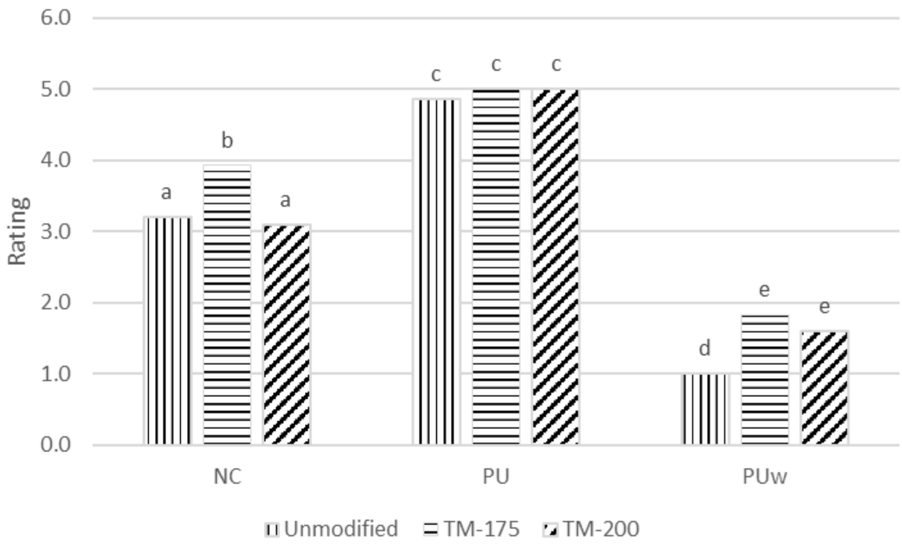

Figure 5 Effect of thermal modification on resistance to acetic acid of applied finishes

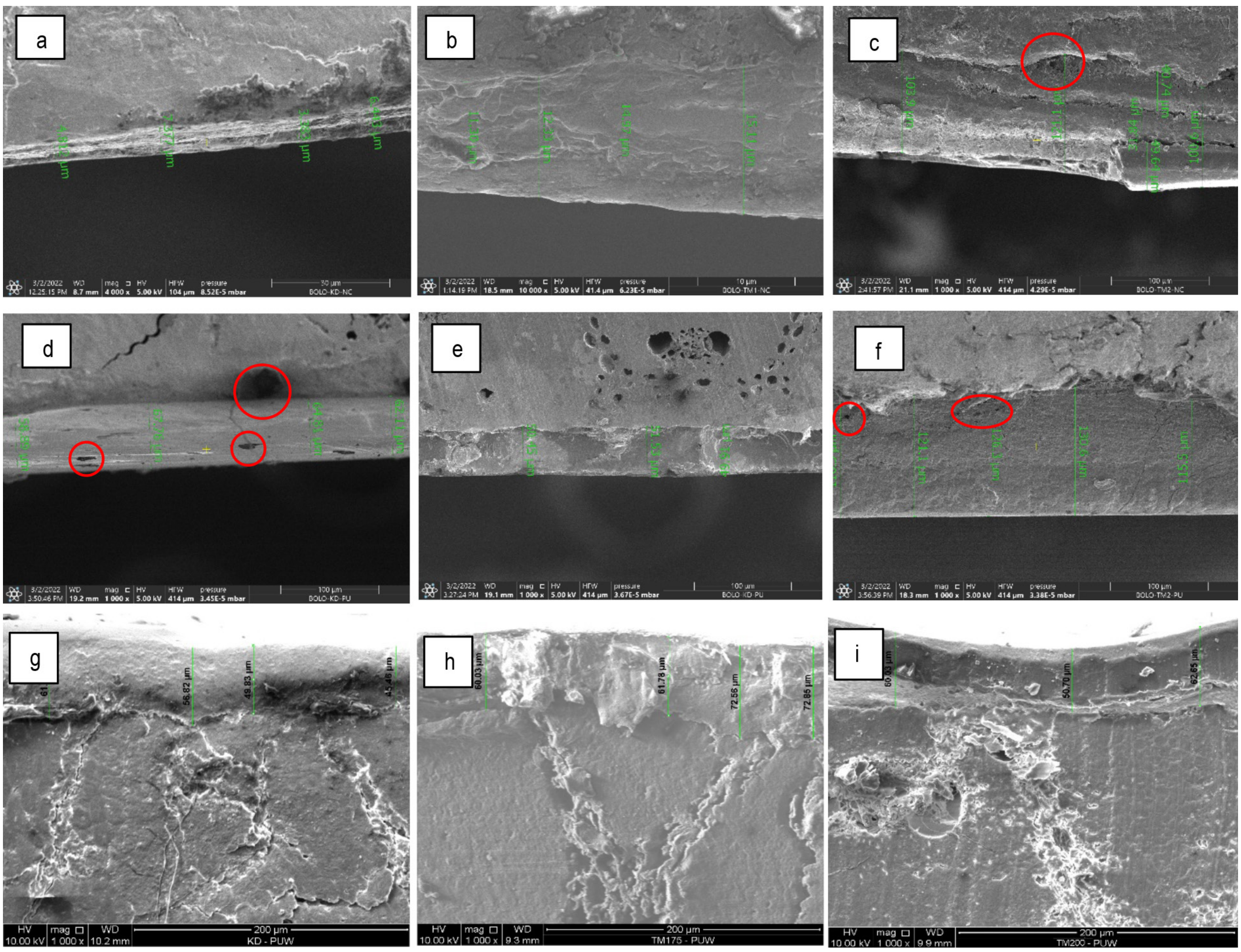

Figure 6 Scanning electron micrograph of various finishes applied on untreated and thermally modified bolo, NC on a) kiln-dried, b) TM175 and c) TM200 bolo, PU on d) kiln-dried, e) TM175 and f) TM200 bolo, PUW on g) kiln-dried, h) TM175 and i) TM200 bolo

Microstructure of finished bolo

Figure 6 shows the micrograph of untreated and thermally modified bolo applied with different finishes. The thickness of NC and PU concurs with the result of ultrasonic measurement, where there is an increase in dry film thickness with thermal modification and with an increase in temperature (Table 3). The NC applied on KD and TM200 showed a weak interfacial bond. The PU applied on KD and TM200 showed air pockets. These further explain the reduction in adhesion property of said finishes. Application of NC on TM175 showed improved mechanical interlocking. While PU and PUW on TM175 and TM200 showed good and smooth bonding. This further suggests that 175 °C is the optimal TM setting for the application of finish on thermally modified bolo.

CONCLUSION

The study determined the performance of three commercial finishes on thermally modified bolo. Thermal modification at 200 °C reduced the pH of heat-treated bolo, indicating chemical changes in the material. Even after the application of different clear finishes, NC, PU and PUW, the thermally modified bolo was still darker than the control.

This dark color eliminates the need to use stains or pigments to enhance the look of the substrate. It also results in a lower gloss, making it easier to detect coating imperfections on the finished surface. As treatment temperature increases, the thickness of applied finishes also increases, thus reducing the amount of coating material needed.

Thermal modification of bolo at 175 °C for 30 min enhanced the performance of NC in adhesion, film hardness and resistance to acid. For PU, only adhesion and film hardness were improved. For both finishes, 200 °C gave the highest gouge hardness. For PUW, 200 °C increased adhesion and resistance to liquids but lessened gouge hardness. The SEM analysis revealed an improved adhesion bonding of NC and PU finish upon thermal modification at 175 °C for 30 mins.

Overall, the thermal modification of bolo enhanced its finishing properties. Further research is recommended to look into the chemical changes responsible for this improvement.

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