UTILIZATION OF CITRIC ACID AS BONDING AGENT IN SEMBILANG BAMBOO (*Dendrocalamus giganteus* Munro) PARTICLEBOARD PRODUCTION

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UTILIZATION OF CITRIC ACID AS BONDING AGENT IN SEMBILANG BAMBOO (*Dendrocalamus giganteus* Munro) PARTICLEBOARD PRODUCTION. Citric acid was utilized as a bonding agent in the production of Sembilang bamboo particleboard. The limitation in using bamboo for particleboard production is that the silica content in bamboo skin can accelerate particleboard processing machines' bluntness and reduce particle adherence in particleboard manufacturing. This research aimed to investigate the influence of bamboo skin and citric acid content on the characteristics of sembilang bamboo particleboard. Particleboards were prepared using bamboo particles (type A) and unskinned bamboo particles (type B). The citric acid solution (59%) was sprayed over the surface of bamboo particles to obtain three different levels of citric acid, i.e., 15, 20, and 25% (based on bamboo particles' dry weight). The sembilang bamboo particleboards were manufactured using a hot-pressing machine at 200°C, 5 MPa for 10 min. The particleboard targeted density was 0.8 g/cm^3. The type B particleboards' internal bond (IB), modulus of rupture (MOR), water absorption (WA), and thickness swelling (TS) were superior compared to the type A particleboards. This was influenced by the lower concentration of silica in type B particleboards, which tend to allow an intimate contact area among particles and citric acid then produced better quality particleboards compared to type A particleboards. The type B particleboards met the obligation of JIS A 5908 for type 18 particleboard in terms of modulus of rupture, modulus of elasticity, and internal bond, however, only fulfilled the type 8 particleboard in terms of screw holding power. The physical properties of Sembilang bamboo particleboard were also improved when using type B bamboo particles and adhered with citric acid at a level of 25%.

Keywords: Sembilang bamboo, particleboards, citric acid, physical properties, mechanical properties, silica

*PEMANFAATAN ASAM SITRAT SEBAGAI AGEN PEREKATAN PADA PEMBUATAN PAPAN PARTIKEL BAMBU SEMBILANG (*Dendrocalamus giganteus* Munro). Asam sitrat digunakan sebagai agen perekatan dalam pembuatan papan partikel bambu Sembilang. Keterbatasan penggunaan bambu untuk pembuatan papan partikel adalah kandungan silika pada kulit bambu dapat mempercepat ketumpulan mesin pengolah papan partikel dan mengurangi kerekatan antar partikel. Penelitian ini bertujuan untuk mengetahui pengaruh kulit bambu terhadap karakteristik papan partikel bambu sembilang. Papan partikel dibuat menggunakan partikel bambu lengkap dengan kulit bambu (tipe A) dan partikel bambu tanpa kulit (tipe B). Larutan asam sitrat (59%) disemprotkan pada permukaan partikel bambu untuk memperoleh tiga kadar asam sitrat yang berbeda, yaitu 15, 20, dan 25% (berdasarkan berat kering partikel bambu). Papan partikel bambu sembilang diproduksi menggunakan mesin kempa panas pada suhu 200°C, 5 MPa selama 10*

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menit. Kepadatan papan partikel yang ditargetkan adalah 0,8 g/cm³. Papan partikel tipe B menunjukkan keteguhan lentur (MOR), keteguhan tarik tegak lurus permukaan (IB), penyerapan air (WA) dan pengembangan tebal (TS) yang lebih baik dibandingkan dengan papan partikel tipe A. Hal ini dipengaruhi oleh konsentrasi silika yang lebih rendah pada papan partikel tipe B, cenderung mempererat area kontak antara partikel dan asam sitrat sehingga menghasilkan kualitas papan partikel yang lebih baik dibandingkan dengan papan partikel tipe A. Papan partikel tipe B memenuhi persyaratan JIS A 5908 untuk papan partikel tipe 18 dalam hal MOR, modulus elastisitas dan IB, namun hanya memenuhi papan partikel tipe 8 dalam hal keteguhan cabut sekrup. Sifat fisik papan partikel bambu Sembilang tipe B dengan asam sitrat pada kadar 25%, lebih baik dibandingkan papan partikel bambu sembilang lainnya dalam penelitian ini.

Kata kunci: Asam sitrat, bambu Sembilang, papan partikel, sifat fisis, sifat mekanis, silika

I. INTRODUCTION

Commercially, particleboards were bonded with formaldehyde-based adhesive. Unfortunately, formaldehyde emission during the use of particleboard, especially for interior furniture purposes, can be harmful to human health. Therefore, the application of formaldehyde-free adhesives for particleboard production has become a critical concern. Some biopolymers have been utilized as the main constituent in formaldehyde-free adhesives. A starch-based adhesive was applied as a binder in particleboard production, although its water resistance property has not met the requirement for application (Amini, Hashim, & Sulaiman, 2019; Salleh et al., 2015) Some lignocellulosic plants contain lignin or tannin that can be utilized as formaldehyde-free adhesives. Lignin is known as the third largest biopolymer. Lignin exists in the structure of lignocelluloses such as wood, agricultural residues, grasses, and other plants (Khalil et al., 2006; Younesi-Kordkheili, 2017). Lignin has a similar structure to phenol. This means lignin can substitute phenol-based adhesive such as phenol-formaldehyde (PF) (Nasir, Zakaria, Sipaut, Sulaiman, & Hashim, 2011).

However, one of the lignin-based adhesive disadvantages is low reactivity due to its complexity and the low number of reactive sites (Pizzi, 2006). Naturally, tannin is present in the skin of several trees, such as mimosa, pine, and quebracho (Fechtal & Riedl, 1993; Kim, 2009). Tannin is a water-soluble compound. Tannin's chemical structure is similar to phenolic compounds and can react with formaldehyde to replace phenolic resins (Faris, Ibrahim, & Rahim, 2016). A tannin-based adhesive has been utilized as a binder in particleboard production (Cui et al., 2015; El-Sayed, El-Sakhawy, Kamel, El-Gendy, & Abou-Zeid, 2019). Nevertheless, the current use of commercial tannin for leather and in the beverages industries limits their availability as an industrial adhesive.

One of the bio-based wood adhesives that attract many researchers' attention is citric acid-based adhesive. Umemura et al. began to study the potential of citric acid as a binder in molded products and as a wood adhesive (Munawar, Umemura, & Kawai, & Kawai, 2009; Umemura et al., 2011, 2012). A citric acid-based adhesive has been used for a binder in the production of particleboard made from *Agave sisalana* (Syamani & Munawar, 2012), bamboo (Widyorini et al., 2013; Widyorini, Umemura, et al., 2016a), oil palm frond (Syamani & Munawar, 2013), sugarcane (Liao et al., 2016; Syamani et al., 2020), sweet sorghum bagasse (Kusumah et al., 2016; Kusumah, Umemura, et al., 2017b), *Imperata cylindrica* (Syamani et al., 2018), corn stalk (Prasetiyo, Octaviana, et al., 2018), and corn husk (Prasetiyo, Gopar, Kurniawati, Syamani, & Kusumah, 2018).

The advantages of using citric acid-based adhesive are renewable, non-toxic, produce particleboards with physical and mechanical properties that meet the standard application. Nevertheless, some disadvantages need to be overcome, such as the required high temperature to set the bonding linkage between particles, which can affect the particleboards processing machine due to adhesive acidity, and produce particleboards with a darker color than particleboards bonded with urea-formaldehyde adhesive. Certain procedures were employed to increase the performance of the citric acid adhesive. Additive agents, such as sucrose, can resolve the particleboard brittleness (Kusumah et al., 2017), increase the number of ester groups to improve the bonding ability of citric acid adhesive (Widyorini, et al., 2016a), increase the hydrogen bond, also the molecular linkage force between particles to produce stronger particleboards (Liao et al., 2016).

There are 1,662 bamboo species in 121 genera spread worldwide (Canavan et al., 2017). Among them, 145 bamboo species belonging to 20 genera are found in Indonesia (Nurdiah, 2016). Bamboo was utilized for furniture, handicrafts, chopsticks, and so on. Moreover, bamboo used as a building material because bamboo culms are strong, tough, straight, easy to bend, and lithe (Widjaja, 2000). Bamboo can also be utilized as particleboard raw materials due to the limitation of wood as particleboard raw material. Sembilang bamboo (*Dendrocalamus giganteus* Munro), has large biomass with a diameter of 20.5 cm and 30.5 m in height, and a thickness of 16.5 mm (Park et al., 2020), that has the potential to be utilized as particleboard raw material. Furthermore, sembilang bamboo can be cultivated after 3 years, providing more sustainability and continuity regarding the availability of particleboard raw materials compared to wood plants.

On the other hand, compared to woods, most bamboo species contain much more silica (0.5-4% w/w) (Ding et al., 2008). The silica is stored as amorphous hydrated silica ($SiO_2.H_2O$) in bamboo with few crystalline phases (Motomura et al., 2006). (Yin et al., 2016) reported that 2-years-old bamboo (*Neosincalamus affinis*) outer skin contains a high concentration of silicon (6.21%). High silica content in some tropical wood species is still a challenge for the woodworking industry due to its abrasive action (Cristóvão, 2013). Moreover, in particleboards production when using urea-formaldehyde resin, the high ash content, primarily silica, contributed to non-uniform resin distribution (Hiziroglu & Suzuki, 2007).

This study aims to examine the properties of particleboards made from bamboo particles (type A) and unskinned bamboo particles (type B) of Sembilang bamboo using citric acid as adhesive. Type A particles were obtained by directly processing bamboo slats with a ring flaker. While the outer skin of Sembilang bamboo was removed and then processed using a ring flaker to obtain type B particles. Using these raw materials, the particleboards were manufactured by hotpressing. The effect of bamboo particles types and citric acid content on particleboards properties were discussed.

II. MATERIALS AND METHODS

Sembilang bamboo (*Dendrocalamus giganteus* Munro) was harvested from the bamboo garden of the Research Center for Biomaterial. We prepared two types of bamboo particles: bamboo particles (type A) and unskinned bamboo particles (type B). Bamboo culms were cut into 40 cm lengths to obtain slats. The bamboo slats were further processed using a planer to remove external layers of bamboo (to obtain unskinned bamboo particles). The bamboo particles were prepared by splitting and chopping bamboo slats, then treated using a ring flaker. Subsequently, bamboo particles were separated with No. 4-mesh and No. 14-mesh screens to obtain bamboo particles with size of 1.41 ~ 4.76 mm. All of the bamboo particles were oven-dried at 60°C to reduce the moisture level of bamboo particles to below 5%. The technical grade of anhydrous citric acid (manufacturer: Weifang Ensign Industry Co., Ltd.) was used without further purification. The citric acid solution with a concentration of 59-60 wt% was obtained by dissolving the citric acid powder in a certain amount of distilled water. This liquid was used as the bonding agent in Sembilang bamboo particleboard manufacturing.

A. Measurement of Bamboo Particles Size Distribution

As many as 3 g bamboo particles were classified using a laboratory vibrating sieve shaker, with sieves of 18 mesh (1000 μm), 14 mesh (1410 μm), 10 mesh (2000 μm), 7 mesh (2830 μm), and 4 mesh (4760 μm). Particles of 5 granulometric classes were used for bamboo particle size distribution: -18 mesh; -18+14 mesh; -14+10 mesh; -10+7 mesh and -7+4 mesh. Symbols (-) and (+) indicate passage and retention of particles, respectively. Afterward, the weight of each particle class was measured and presented.

B. Measurement of Bamboo Particles Geometry

The length, width, and thickness of 100 bamboo particles were measured using a caliper (Mitutoyo Digital Caliper 500-170-30). Based on data of bamboo particle geometry, the slenderness ratio was calculated by dividing particles' length to particles' width. The aspect ratio was calculated by dividing particles' length to particles' thickness of the two types of bamboo particles.

C. Measurement of Bamboo Particles Bulk Density

Particles' bulk density was calculated based on the proportion of the mass to the volume engaged (Cardoso et al., 2013; Omoniyi & Olorunnisola, 2014). Bamboo particles were oven-dried for 24 h before calculation. Firstly, the weight of the 50-mL cylinder was measured. Bamboo particles were placed into the cylinder until they filled the cylinder to reach the 50-mL mark, then the cylinder was re-weighed. The bulk density was calculated based on the difference in weight between the bamboo particles loaded cylinder and the empty cylinder, then was divided by the volume loaded by the bamboo particles.

D. Measurement of Silica Content in Bamboo Particles

The silica content in bamboo particles was measured using a modified method from previous research (Yuan, 2017). Five grams of dried Sembilang bamboo powder was placed in a weighted ceramic crucible, then entirely ashed at 525°C. After cooling, crucible content bamboo ash was weighted to determine the ash content in bamboo. A 10 mL of HCl (6 mol/L) was poured onto the ash. The acid-soluble ash solution was slowly boiled to near dryness using a boiling water bath. HCl treatment was conducted for 10 min and repeated three times. The other 15 mL of HCl (6 mol/L) was added to the solution. After 2 more minutes, the solution was filtered off through No. 42 ashless filter paper (Fisher Scientific, Canada), weighted. The precipitate (silica) was washed 5-6 times with 1 mol/L HCl solution, subsequently 5-6 times with hot deionized water (≈ 50°C). The filter paper with the precipitate and filter paper only (as control) was put in a different ceramic crucible and put in a muffle furnace to be ashed at 600°C to reach a constant weight. The difference in weight of ceramic contains filter paper residue and filter paper + silica residue was calculated to determine silica content.

E. Measurement of Bamboo Particles Wettability

Wettability of bamboo particles was conducted by dropping citric acid solution (0.1 mL) on the outer part (representing type A particles), and on the inner part (representing type B particles) of the bamboo slat. The citric acid solution spread onto the bamboo surface was recorded for 3 minutes with a Digital Microscope (Dino-lite Basic AM2111). The contact angle was measured using Image J software every 10 sec. Shi and Gardner (2001) presented a wettability model with the following equation.

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

Where:

θ = contact angle

θi = initial contact angle

θe = equilibrium contact angle

K = contact angle change rate constant

t = time

F. Production of Particleboard

The citric acid solution was sprayed over the surface of Sembilang bamboo to obtain particles with three different levels of citric acid, which were 15, 20, and 25 wt% (based on dry bamboo particle weight). Afterward, the particles were oven-dried at 80°C for 6 h to reduce water content. Following this, the particles were placed in a wooden forming mold with a dimension of 30 cm × 30 cm to form a mat. The hot-pressing machine pressed the particle mat at 200°C for 10 min. A 9-mm thick steel bar was positioned to control the board thickness during the hot-pressing process to produce particleboard with a target density of 0.8 g/cm^3. Once the upper part of the pressing plate reached the steel bar, the pressing pressure was 25 MPa.

G. Evaluation of Particleboards Surface Roughness Properties

The surface roughness analysis was performed by using a fine stylus profilometer (Mitutoyo SJ-201). Triplicate measurements were conducted for each particleboard. The average roughness (Ra) as roughness parameters were determined. The calculation of surface roughness parameters was based on digital information generated by the equipment. The particleboard surface roughness in this study was measured with a sensitivity of 0.5 µm. Pin diameter, pin top angle, and measuring the tool's speed were 4 µm, 90°and 0.5 mm/sec, respectively. The length of the tracing line (Lt) and cut-off were 12.5 and 2.5 mm (c), respectively. The measuring force of the scanning arm on the samples was 4 mN (0.4 gf). Measurements were conducted at room temperature, and the pin was calibrated before the tests.

H. Particleboards' Mechanical Properties Characterization

The bamboo particleboards were tested to investigate the modulus of elasticity (MOE), modulus of rupture (MOR), internal bond (IB), by the Japanese Industrial Standards for particleboards (JIS A 5908:2003). The particleboard bending strength properties, including MOR and MOE, were measured by a three-point bending test method using Universal Testing Machine (Shimadzu Autograph 50kN). The bending strength analysis was using a board specimen with dimension length x width of 200 mm × 50 and was tested in dry condition. During the bending strength analysis, load cell speed was 10 mm/min, and the effective span was 150 mm. Meanwhile, IB analysis was conducted on sample test dimension of length x width of 50 mm × 50 mm, and with load cell speed of 2 mm/min.

I. Particleboards' Physical Properties Characterization

The bamboo particleboards were subjected to water absorption (WA) and thickness swelling (TS) analysis, based on JIS A 5908:2003. Physical properties testing was conducted following 7 days of conditioning at room temperature. The specimen size of 50 x 50 x 9 mm was applied to evaluate TS and WA. Specimens were immersed in water for 24 h at room temperature. The thickness and weight differences before and after immersion were calculated.

J. Statistical Analysis

The mechanical and physical particleboards properties data were evaluated using a balanced analysis of variance (ANOVA) procedure for a completely randomized design. The experimental design consisted of two parameters (type of particleboards and citric acid content), and their interactions. Tukey's pairwise comparison test was performed to permit the separation of means. Results were considered significant at 95% confidence levels, by using Minitab Software.

K. Particleboard's Durability Analysis by Cyclic Aging Treatment

Particleboards's durability was tested by treating sample specimens under successive severe conditions, based on the method by Kusumah et al. (2016) with slight modification. Cycling aging treatment involves five steps: 1) soaking in water at room temperature for 24 h, 2) oven-dry at 105°C for 8 h, 3) immersion in warm water (70°C) for 10 h, 4) oven-dry at 105◦C for 10 h, 5) immersion in boiling water for 4 h, and 6) oven-dry at 105°C for 16 h. The changes in weight and thickness of the samples during the treatment were recorded. Each experiment was performed with five replications. The standard deviation and the mean values were calculated. The thickness swelling (TS) and water absorption (WA) of the particleboard after each treatment cycle were measured.

L. Bonding adhesion of Bamboo Particles and Citric Acid Analysis by FTIR

The edge of the particleboard was scratched to obtain particles. The particles were ground into a powder, and the powder obtained was dried in a drying oven at 60°C for 16 h. Infrared (IR) spectral data were obtained with an FTIR spectrophotometer (Spectrum Two, Perkin Elmer) using the Universal Attenuated Total Reflectance (UATR) method and were recorded by an average of 16 scans at a resolution of 4 cm^{-1}.

III. RESULT AND DISCUSSION

A. Particleboards Surface Roughness

Bamboo particle size in this study was distributed unevenly, as demonstrated in Figure 1. Most of them have particle size more than 2830 μm (2.83 mm) or passed through No 7-mesh screen, 90.78% for type A bamboo particles and 74.56% for type B bamboo particles.

Type B bamboo particles consist of more fine particles (9.64%) than type A bamboo particles (0.63%). After bamboo slats were peeled off, the hardest parts of bamboo slats no longer existed. So that during the processing in ring-flaker, type B bamboo slats produced extra fine particles (Figure 2). While the width

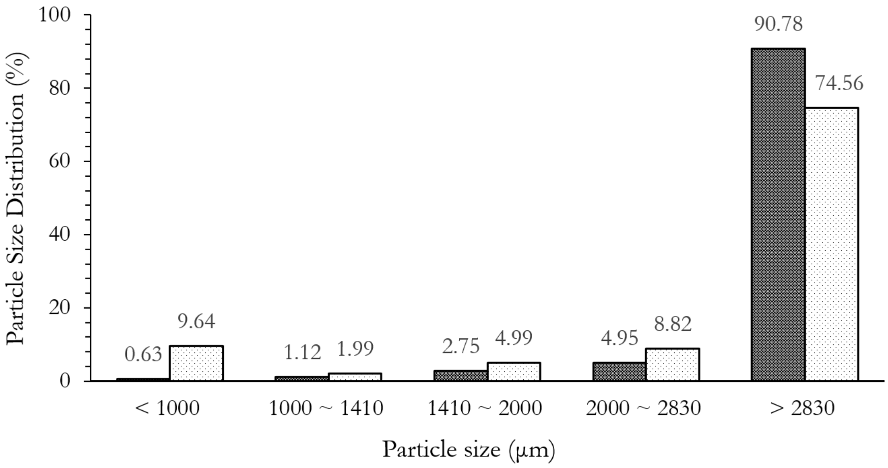

Figure 1. Bamboo particles size distribution

Figure 2. Type A bamboo particles and Type B bamboo particles

and thickness of type A bamboo particles and type B bamboo particles show no significant difference (Figure 3).

The average of particle bamboo geometry from 100 particles of each type is presented in Figure 3. Type B bamboo particles tend to split along the longitudinal direction. The length of type B bamboo particles (41.49 mm average) was longer than that of type A bamboo particles (22.40 mm average). Based on data of particle geometry, we can calculate the slenderness ratio (length/width) and aspect ratio (length/thickness) of the two types of bamboo particles (Table 1).

de Lira Bazzetto et al. (2019) produced bamboo particleboards from bamboo (Dendrocalamus asper) particles with a size of 0.210 mm~0.500 mm and classified them into 4 groups, which were -0.500+0.420 mm, -0.420+0.297 mm; -0.297+0.250mm; -0.250+0.210 mm. Symbols (-) and (+) indicate passage and retention of particles, respectively. Those bamboo particle sizes were relatively smaller than the size of bamboo particles used in this study.

Type B bamboo particles' slenderness ratio and aspect ratio are higher than of type A bamboo particles. Thinner and longer particles yield a higher aspect ratio, larger surface area, and increased contact area in the glue line, contributing to better interaction and thus higher strength (Juliana et al., 2012; Kasim et al., 2018). Nonetheless, the bulk density of both types of bamboo particles shows no significant difference.

Smaller particles produced particleboard with improved surface smoothness nevertheless tend to reduce strength properties and dimensional stability and increase the difficulty in blending resin and mat-forming (Kelly, 1977). The particleboards' characteristics and properties are affected by the type of raw material, the particle geometry, the chemical content in the raw material, and the type and level of adhesive. Particle geometry closely interacts with all these parameters, responsible for the particleboard properties (Maloney, 1993). Furthermore, the particleboard structures are defined by the conditions in which the pressing conditions and the mattress are formed.

Table 1. The geometry ratio and bulk density of bamboo particles

	Slenderness Ratio	Aspect Ratio	Bulk Density (g/cm^3)
Type A bamboo particles	37.06	21.17	0.054
Type B bamboo particles (unskinned)	67.70	31.81	0.053

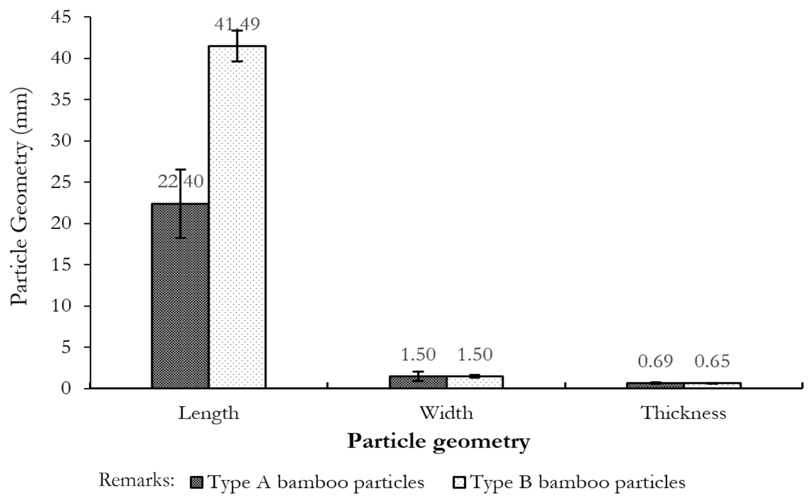

Figure 3. Bamboo particles geometry

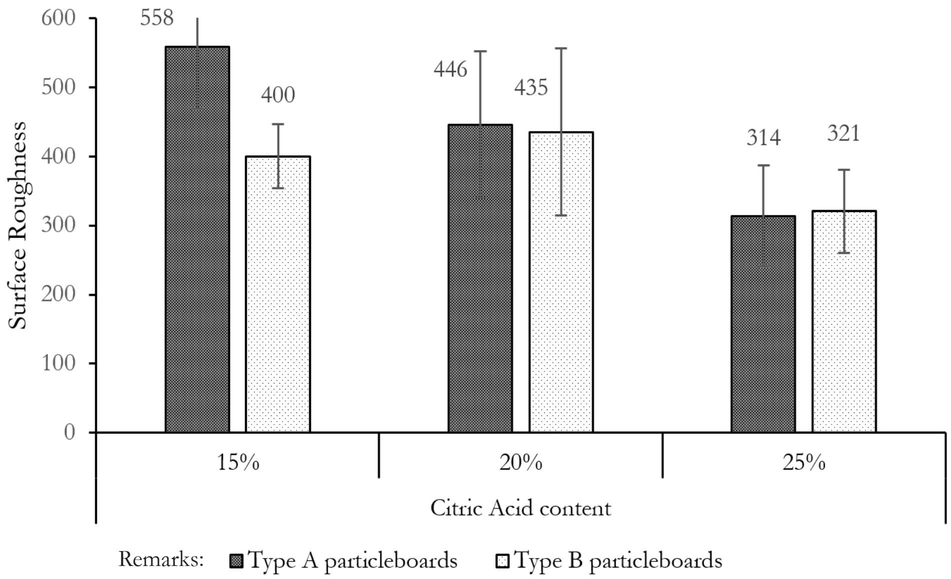

Figure 4. The surface roughness of bamboo particleboards

As explained in the previous section, Type B bamboo particles have a higher slenderness ratio and aspect ratio than type A bamboo particles. With small and thin characteristics, type B bamboo particles could transfer more easily to fill void spaces during hot pressing and create smooth surfaces. Particle geometry has a considerable effect on panel surfaces and edges. The small and thin particles result in smooth board surfaces. The smooth surface is appropriate for different coating types due to their ability and flexibility to fill void spaces.

A standard commercially manufactured particleboard could have average surface roughness (Ra) values of 3 to 6 μm (Hiziroglu, 1996). Type B bamboo particleboards show a smoother surface compared with type A particleboards (Figure 4). The average roughness value was 400 μm for type B bamboo particleboards and 558 μm for type A particleboards when bonded with 15% citric acid. At the same time, the surface roughness of bamboo particleboard bonded with 20% and 25% citric acid when using type A or type B bamboo particles showed no significant differences. The higher amount of citric acid application for adhering bamboo particleboard produced smoother particleboards. The average roughness of type B bamboo particleboards bonded with 20% and 25% citric acid were 435 μm and 321 μm, respectively. Regrettably, it seems that the board surface characteristic of all the particleboards is very rough. So, the sanding process needs to be applied when overlaying application is desired.

Particleboard made from fine particles had a smoother surface compared to that of coarse particles. The usage of citric acid developed a better contact among bamboo particles; therefore, better adhesion, producing a smooth particleboard surface. Rising resin content level would cause the particleboard surface to become smoother or decrease Ra value (Widyorini, Umemura, et al., 2016b).

Other than particle geometry, silica in bamboo skin might also affect the particleboard surface roughness. Silica content in type A bamboo particles was 1.07 %, while in type B bamboo particles was 0.93 %. The effort of peeling off Sembilang bamboo slats can improve particleboard surface smoothness, particularly when applying lower content of citric acid.

B. Particleboards Wettability

In this study, the contact angle between type A bamboo particles and water or citric acid was higher than the contact angle of type B bamboo particles and water or citric acid (Table 2).

Table 2. The contact angle of water and citric acid on the bamboo surface

	Contact angle (°)	
	Water	Citric acid
Type A bamboo particles	69 ± 5	78 ± 6
Type B bamboo particles	35 ± 13	52 ± 11

K values of water and citric acid on the type B bamboo surface were higher than of type A bamboo (Table 3). The higher the K value is, the more the contact angle is; thus, the curve is steeper (Figure 5), indicating the type B bamboo surface has higher wettability than type A bamboo.

Moreover, the bamboo skin's initial contact angle against the water was greater than 83° indicating that water could not spread easily on bamboo skin. Yuan and Lee (2013) said contact angles greater than 90° commonly mean that wetting is critical, so the fluid will minimize its contact with the surface and form a dense liquid droplet.

The surface's wettability indicates the condition that determines the extent to which the fluid will be spread by the surface, affecting the absorption, adsorption, penetration, and spread of adhesive (Marra, 1992). Wettability is an essential property of the wood surface, and it influences bonding properties directly (Tang et al., 2012). In the liquid wetting process, the contact angle change as a function of time is a decreasing function (Shi & Gardner, 2001).

Silica content in bamboo skin was higher than in unskinned bamboo, so bamboo skin was more hydrophobic. The contact angle value is also influenced by material surface macroscopic

Table 3. Bamboo dynamic wettability towards the water and citric acid (R= correlation coefficient, θe = equilibrium contact angle, θi = initial contact angle)

		K ($L.sec^{-1}$)	R	θe (°)	θi (°)
Type A particles	Water	0.0065	0.9124	65.3460	83.0887
	Citric acid	0.0061	0.9133	71.4629	93.3712
Type B particles	Water	0.0479	0.9442	25.9650	75.6353
	Citric acid	0.0203	0.9478	43.0043	82.0578

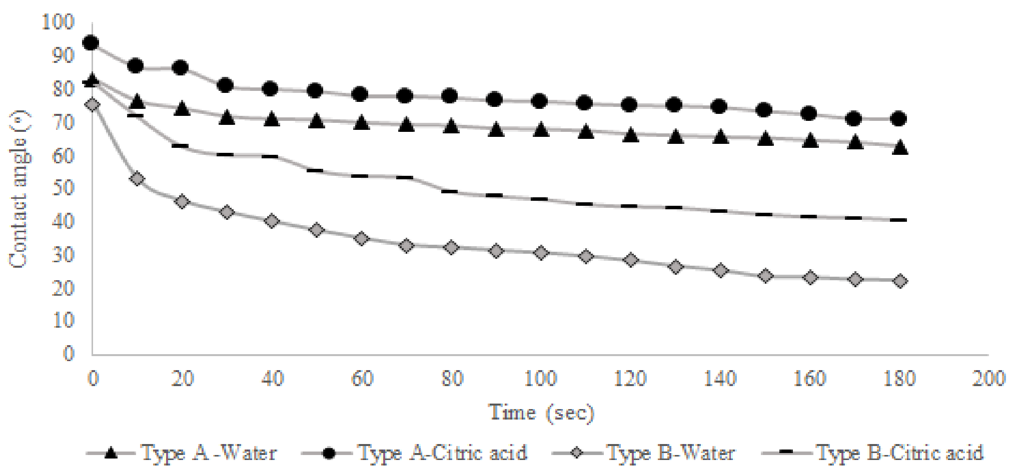

Figure 5. Citric acid and water contact angle change on the surface of type A and type B bamboo particles as a function of time

properties such as porosity, surface smoothness, pH, and chemical compound (Lu & Wu, 2006).

In bamboo culms, silica was considered as a major constituent of the epidermis with values between 1.5 % (*Bambusa vulgaris*) and 6.4 % (*Schizostachyum lumampao*) (Liese, 1998). Van Acker et al. (2000) reported values of 0.066 % in the lower part and 0.082 % in the upper part of *Phyllostachys praecox* culms and values of 0.120 % in the lower part and 0.355 % in the upper part of *Phyllostachys nigra* culms. (Lybeer, 2006) indicated lower values between 0.04 and 0.11 % Si for *Phyllostachys nigra* and *Phyllostachys viridiglaucescens* and 0.08 to 0.11% Si for the tropical species *Gigantochloa levis* and *Dendrocalamus asper*. Compared to another genus of bamboo, as previously stated, the silica content in sembilang bamboo was quite high.

C. Particleboards Mechanical Properties

Figure 6 shows the internal bond (IB) of Sembilang bamboo particleboards. The range IB value of 0.79 – 0.97 N/mm2 was recorded in type B bamboo particleboards, and 0.57-0.78 N/mm^2 was recorded in type A bamboo particleboards. The internal bond of sembilang bamboo particleboards bonded with citric acid was higher than the requirement (0.3 N/mm2) for the Type 18 particleboard internal bond, based on JIS A 5908.

Analysis of variances shows, P-value <0.05 (Table 4), that the type of bamboo particle has a significant effect on the IB value of particleboard.

Further tests using the Tukey comparison method, with 95% confidence level, are presented in Table 5, showing that type B bamboo particles produce particleboards with

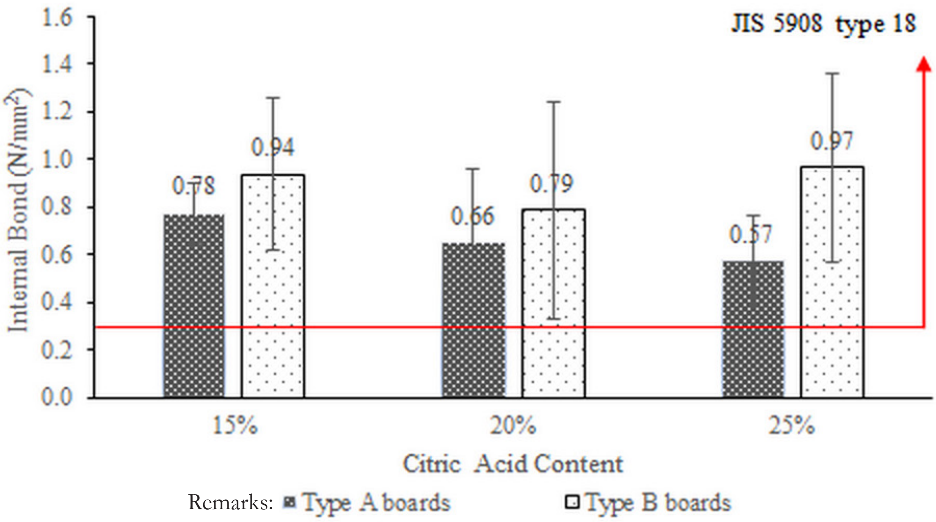

Figure 6. Internal bond of particleboards made from type A and type B bamboo particles

Tabel 4. Analysis of variance for particleboard's internal bond

Source	DF	SS	MS	F	P
Replication	4	0.79150	0.19787	2.38	0.086
Particleboards type	1	0.39496	0.39496	4.74	0.042
CA content	2	0.09468	0.04734	0.57	0.575
Particleboards type*CA content	2	0.10127	0.05064	0.61	0.554
Error	20	1.66525	0.08326		
Total	29	3.04766			

Tabel 5. Grouping information using the Tukey Method and 95% confidence

Particleboards type	N	Mean	Grouping	
Type B	15	0.897777	A	
Type A	15	0.668298		B

Remarks: *Means that do not share a letter are significantly different.*

higher IB values and are significantly different from type A bamboo particleboard.

The results of the wettability analysis were in accordance with the results of the IB analysis, which show that the particleboard's IB values were not influenced by citric acid content but influenced by the type of bamboo particles. IB values of particleboards made from Sembilang bamboo particles demonstrate an interesting value. It shows that 15 % of citric acid was enough to bond type A and type B bamboo particles to produce particleboards that fulfilled the Type 18 particleboard standard, based on JIS A 5908. The citric acid solution can easily cover and fill the void of bamboo particles whether or not the skin bamboo is still attached to the bamboo particles. Due to hot-pressing, citric acid was reacted with hydroxyl groups of bamboo particles, producing a strong internal bond (explained further in the bonding mechanism at the end of the article). The same bonding mechanism was observed in another grass family, such as Imperata cylindrica (Syamani et al., 2018) and sugarcane (Syamani et al., 2020).

Citric acid is expected to act as a bonding agent in bamboo-based particleboard. However, the presence of silica in type A boards drives citric acid to be reacted to destruct silica rather than reacted with hydrogen groups in bamboo particles. Therefore, the internal bond in type A boards was lower than in type B boards.

MOR average values of two kinds of bamboo particleboards at various citric acid contents are presented in Figure 7. It is indicated that the type of bamboo particles affected the particleboards MOR significantly. Analysis of variances shows that the type of particleboard, has a value of P<0.01 (Table 6), indicating that the type of bamboo particles has a very significant effect on the MOR value of particleboard.

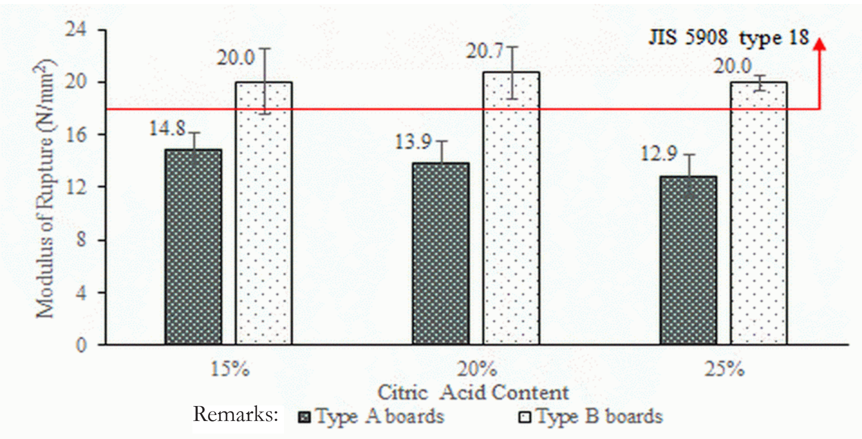

Figure 7. Modulus of rupture of particleboards made from type A and type B bamboo particles

Tabel 6. Analysis of variance for particleboards' modulus of rupture

Source	DF	SS	MS	F	P
Replication	4	21.844	5.461	2/31	0.093
Particleboards type	1	304.388	304.388	128.74	0.000
CA content	2	6.075	3.038	1.28	0.299
Particleboards type*CA content	2	5.169	2.585	1.09	0.354
Error	20	47.288	2.364		
Total	29	384.764			

Further tests using the Tukey comparison method, with 95% confidence level, are presented in Table 7, showing that type B bamboo particleboard produces a higher MOR value and is significantly different from type A bamboo particleboard.

Tabel 7. Grouping information using the Tukey method and 95% confidence

Particleboards type	N	Mean	Grouping	
Type B	15	20.2352	A	
Type A	15	13.8646		B

Remarks: *Means that do not share a letter are significantly different.*

The MOR values of type B bamboo particleboard, bonded with 15, 20, 25 % citric acid, were higher than type A bamboo particleboard, i.e. 20.0, 20.7, and 20.0 N/mm^2, respectively, and fulfilled the requirement for type 18 particleboard by JIS 5908. The high MOR values indicated that the type B bamboo particles developed good bonding strength with citric acid. In contrast, particleboards made from type A bamboo particles showed lower MOR values, considering that the adhesion between bamboo particles could be obstructed by the existence of silica in bamboo skin. However, all particleboards made from type A bamboo particles could fulfill the obligation of Type 13 of JIS A 5908, where MOR of 13.0 N/mm^2 or more is obliged. Regarding the citric acid amount applied to bond bamboo particles, the MOR values were not significantly affected.

In contrast to MOR value, particleboards made from type B bamboo particles showed a lower MOE value (Figure 8) compared to particleboard made from type A bamboo particles when bonded with 15% or 20% citric acid. Nonetheless, the MOE values of particleboards made from type A and type B bamboo particles were high that can fulfill the requirement of type 18 of JIS A 5908, where MOE of 3000 N/mm^2 or more is obliged. Based on the analysis of variances, the interaction of particleboard type and citric acid content gave a significant effect on the MOE value of particleboard (Table 8).

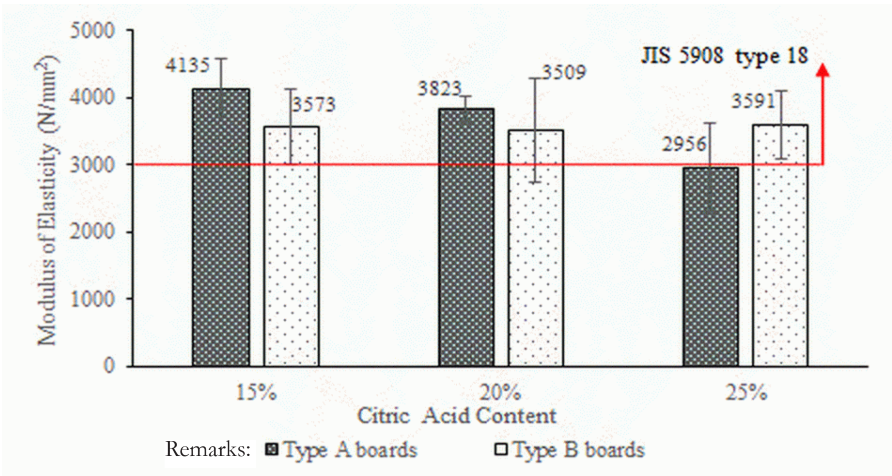

Figure 8. Modulus of elasticity of particleboards made from type A and type B bamboo particles

Tabel 8. Analysis of variance for particleboard's modulus of elasticity

Source	DF	SS	MS	F	P
Replication	4	2209546	552387	2,18	0,109
Particleboards type	1	48710	48710	0,19	0,666
CA content	2	1756876	878438	3,46	0,051
Particleboards type*CA content	2	1997017	998508	3,93	0,036
Error	20	5078729	253936		
Total	29	11090878			

Further testing of the effect of particleboard type used the Tukey comparison method, with 95% confidence level is presented in Table 9 and Table 10, indicating that the type of particleboard or the level of citric acid does not affect the MOE value of the particleboard. As reported by (Li & Beijing, 2004) and (Yuan, 2017), in general, bending properties (MOR and MOE) decreased as the portion of the outer bamboo surface removed increased. The outer bamboo surface shows higher bending properties than the inner part of bamboo. In line with Li's statement, this study result shows that the MOE of particleboards made from type B bamboo particles was lower than that of

Tabel 9. Grouping information using the Tukey method and 95% confidence

Particleboardstype	N	Mean	Grouping
Type A	15	3638.17	A
Type B	15	3557.58	A

Remarks: *Means that do not share a letter are significantly different.*

Tabel 10. Grouping information using the Tukey Method and 95% confidence

CA content	N	Mean	Grouping
CA 15%	10	3854.04	A
CA 20%	10	3666.34	A
CA 25%	10	3273.25	A

Remarks: *Means that do not share a letter are significantly different.*

MOE particleboard made from type A bamboo particles when bonded with 15% and 20% citric acid. Type B particleboard, which no longer has bamboo skin, shows lower stiffness compared to type A particleboard.

Both types of Sembilang bamboo particleboards (type A and type B) demonstrate screw holding power (SHP) value greater than 300 N (fulfilling type 8 particleboards based on JIS A 5908) when bonded with 15% and 20% citric acid. However, the addition of citric acid until 25% causes particleboard SHP value to decrease (Figure 9). Analysis of variances from the treatment of citric acid levels, has a value of $P < 0.01$ (Table 11), indicating that the levels of citric acid have a very significant effect on the value of the screw holding power of the particleboard.

Further testing of the effect of citric acid levels using the Tukey comparison method, with 95% confidence level is presented in Table 12, showing that 15% citric acid content produces particleboard with higher SHP values and is significantly different from bamboo particleboard with a citric acid content of 20% and 25%.

Citric acid affected the power of the screw to hold the particleboard due to its acidity. Based on this data, it is suggested to use not more than 20% citric acid content to bond the particle, especially when the particleboards are intended to be used as furniture. The SHP of type A

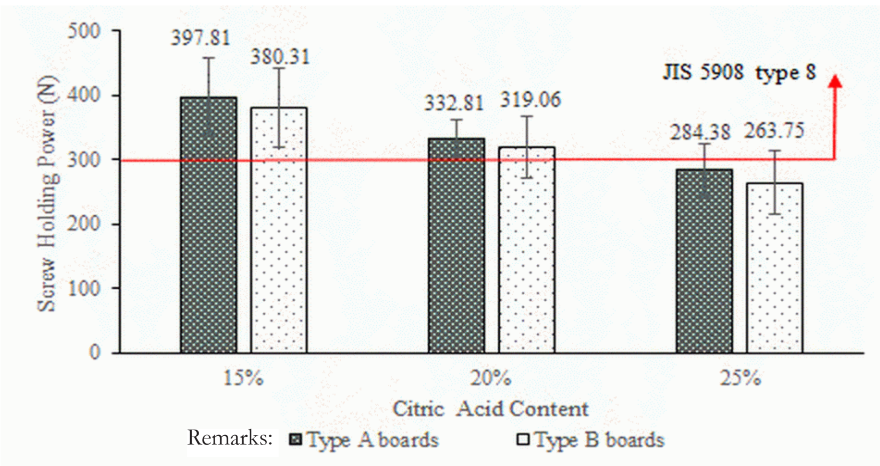

Figure 9. Screw holding power of particleboards made from type A and type B bamboo particles

Tabel 11. Analysis of variance for particleboard's screw holding power

Source	DF	SS	MS	F	P
Replication	4	11383	2845.7	1.18	0.351
Particleboards type	1	2243	2242.5	0.93	0.347
CA content	2	66336	33168.1	13.72	0.000
Particleboards type*CA content	2	59	29.6	0.01	0.988
Error	20	48358	2417.9		
Total	29	128379			

Tabel 12. Grouping information using the Tukey method and 95% confidence

CA content	N	Mean	Grouping	
CA 15%	10	389.063	A	
CA 20%	10	325.938		B
CA 25%	10	274.062		B

Remarks: *Means that do not share a letter are significantly different*

particleboards was higher than that of type B particleboards. Particleboard strength, nail, and screw withdrawal resistance are important characteristics of particleboards and are mainly affected by particle geometry. As stated in Table 1, the slenderness ratio and aspect ratio of type A bamboo particles were lower than of type B bamboo particles. It means that the shape of type A bamboo particles is wider and thicker than of type B bamboo particles. Therefore, particleboards made from type A bamboo particles have more power to hold the screw.

D. Particleboards Physical Properties

Particleboards' water absorption and thickness swelling characteristics were analyzed to explain bamboo particleboards' physical properties. It is obvious from Figure 10 that the resistance of the particleboards to absorb water was improved significantly when using type B bamboo particles. The water absorption (WA) values were 19.38 ~ 23.35 % for type B bamboo particleboards bonded with various citric acid content. On the other hand, the particleboards using type A bamboo particles show WA values in a range of 32.69 ~ 33.53 %. The influence of bamboo particle type was more significant compared to citric acid content. Analysis of variances due to particleboard type has a value of $P<0.01$ (Table 13), indicating that particleboard type has a very significant effect on the water absorption (WA) value of particleboard.

Further testing of the effect of particleboard type using the Tukey comparison method, with

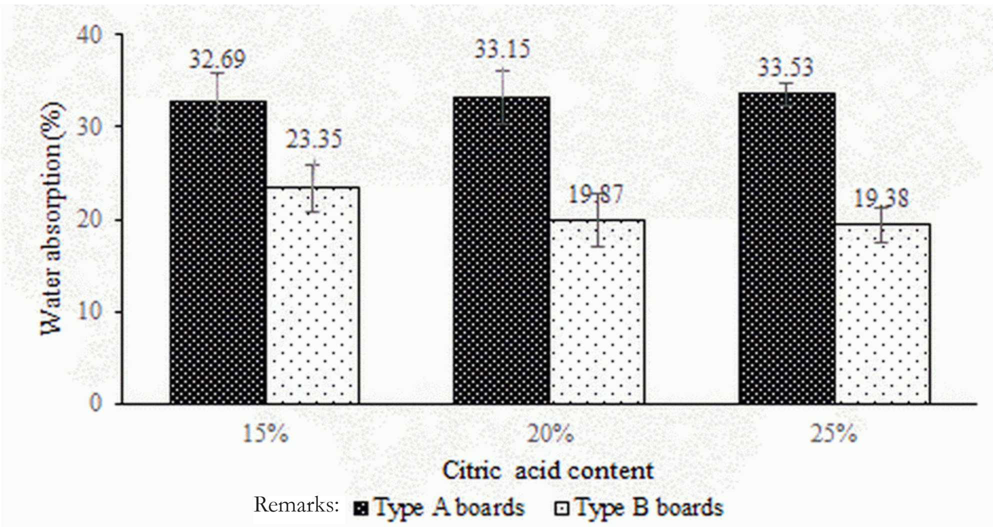

Figure 10. Water absorption of particleboards made from type A and type B bamboo particles

Tabel 13. Analysis of variance for particleboards' water absorption (%)

Source	DF	SS	MS	F	P
Replication	4	23.74	5.94	0.94	0.462
Particleboards type	1	1127.08	1127.08	178.04	0.000
CA content	2	15.79	7.89	1.25	0.309
Particleboards type*CA content	2	32.73	16.37	2.59	0.100
Error	20	126.61	6.33		
Total	29	1325.95			

Tabel 14. Grouping information using the Tukey method and 95% confidence

Particleboards type	N	Mean	Grouping	
Type A	15	33.1238	A	
Type B	15	20.8650		B

Remarks: *Means that do not share a letter are significantly different.*

a 95% confidence level is presented in Table 14, showing that type B bamboo particles produce particleboard with a lower water absorption value than type A particleboard. This indicates the stability of the type B particleboard is better than the type A particleboard.

(Widyorini et al., 2016) applied 15% and 30% citric acid content to bond various bamboo species with different sizes of particles. She explained that citric acid content was more principal on particleboards dimensional stability than bamboo species or particle size. The WA values were 35%, 17%, and 26% for particleboards made from 15% citric acid and fine particles of petung, wulung, and apus bamboo. Then the WA values were improved to 15%, 13%, 12% for particleboards made from 30% citric acid and fine particles of Petung, Wulung, Apus bamboo, respectively. In this study, the citric acid amount varied at 15%, 20%, and 25%, and has not significantly influenced particleboard WA value. The difference of bamboo particles type influenced wettability properties, as mentioned before. Type B bamboo particles which have lower silica content, have superior wettability properties than type A bamboo particles, thus causing the improved bonding then resulted in enhanced dimensional stability.

As demonstrated in Figure 11, the thickness swelling (TS) values of the particleboards bonded with citric acid fulfilled the requirement

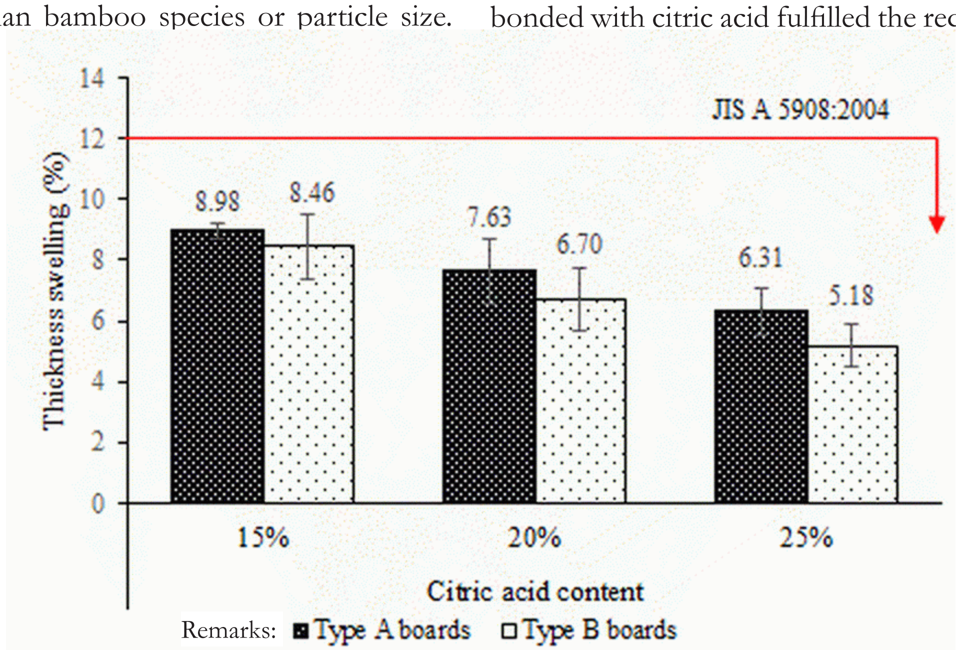

Figure 11. Thickness swelling of particleboards made from type A and type B bamboo particles

Tabel 15. Analysis of variance for particleboard's thickness swelling

Source	DF	SS	MS	F	P
Replication	4	7.987	1.9967	0.90	0.483
Particleboards type	1	5.563	5.5634	2.51	0.129
CA content	2	44.193	22.0965	9.95	0.001
Particleboards type*CA content	2	0.487	0.2436	0.11	0.897
Error	20	44.399	2.2199		
Total	29	102.630			

of JIS A 5908 (max 12 %), demonstrating that bamboo particleboard had good dimensional stability. Analysis of variances due to citric acid content has a value of $P<0.01$ (Table 15), indicating that citric acid content has a very significant effect on the value of thickness expansion (TS) of particleboard.

Further testing of the effect of citric acid levels using the Tukey comparison method, with 95% confidence level is presented in Table 16, showing that particleboard with 25% citric acid content produces particleboard with a lower thickness expansion value than particleboard with 15% and 20% citric acid content. This shows that the stability of the bamboo particleboard bonded with citric acid at a level of 25% is better than that of the bamboo particleboard bonded with citric acid at a concentration of 15% and 20%.

Tabel 16. Grouping information using the Tukey Method and 95% confidence

CA content	N	Mean	Grouping	
CA 15%	10	8.71781	A	
CA 20%	10	7.16632	A	B
CA 25%	10	5.74580		B

Remarks: *Means that do not share a letter are significantly different.*

The TS value was 8.98% for type A particleboard bonded with 15% citric acid. The TS value was improved to 7.63% and 6.31% by adding 20% and 25% citric acid, respectively (Figure 10). Moreover, the TS value of type B particleboard was also improved when applying higher citric acid content, with a range of TS values of 5.18~8.46%. This occurrence is possible because type B bamboo particles might deliver a more intimate contact area among particles, and in contrast with type A bamboo particles due to the lower concentration of silica in type B bamboo particles, resulting in more intense bonding. The same trend was also found by (Widyorini, Nugraha, et al., 2016). They reported that bamboo-citric acid particleboards have TS value in a range of 7 ~ 9% when bonded with 15% citric acid and 2 ~ 4% when bonded with 30% citric acid.

E. Particleboards Durability Analysis by Cyclic Aging Treatment

The thickness change of particleboards made from type A and type B bamboo particles after cyclic aging treatment are depicted in Figure 12. The thickness change of bamboo particleboards at each phase of the cyclic aging treatment decreased with the increase of citric acid content, regardless of the type of bamboo particles.

The particleboard dimensions have remained stable during all steps of cyclic aging treatment. The results demonstrate that boards with a 25% citric acid content performed better dimensional stability than the boards bonded with 15% or 20% citric acid. The percentage of thickness-change of type A and type B bamboo particleboards bonded with 25% citric acid after being boiled in water for 4 h was 12.15% and 12.31%, respectively. This value of particleboard thickness-change was lower than those bonded with 15% or 20% citric acid. The result suggests that 25% of citric acid effectively produced good dimensional stability particleboard.

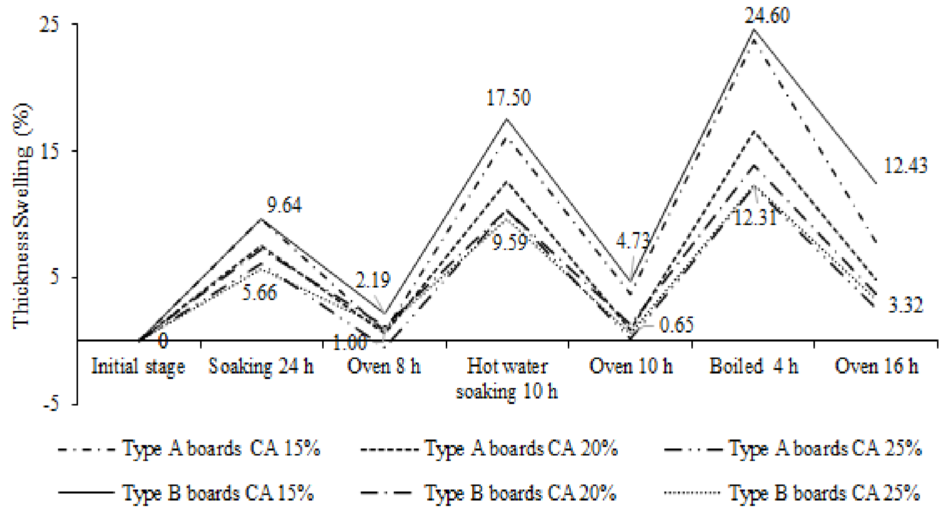

Figure 12. Thickness swelling of bamboo particleboards during cyclic aging treatment

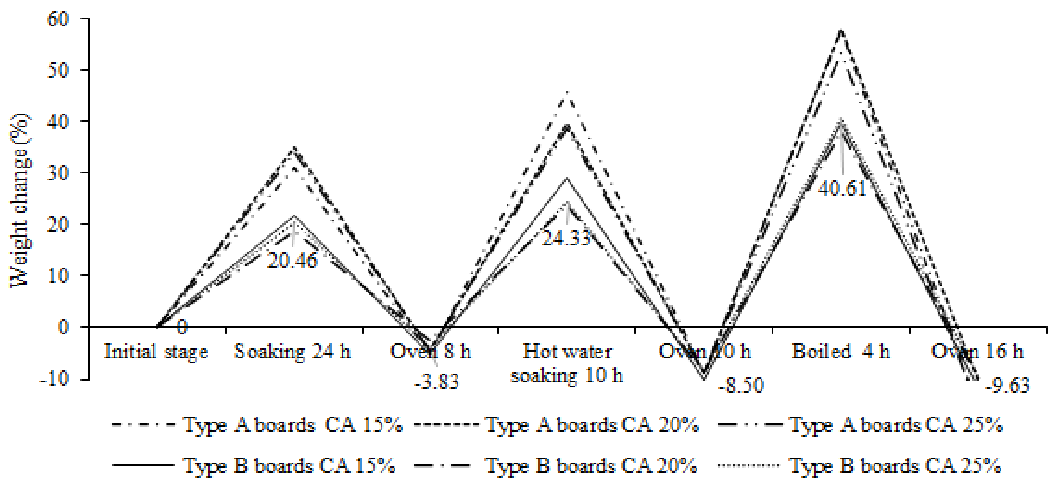

Figure 13. Weight changes of bamboo particleboards during cyclic aging treatment

The change of particleboards weight after cyclic aging treatment is illustrated in Figure 13, indicating that the weight change decreased with the increase of citric acid level.

This outcome demonstrates that the increase of citric acid levels enhances the inhibition of water absorption. The type B bamboo particleboards weight changes due to the following hot-water immersion treatment (24.33%) were higher than those of type A bamboo particleboards after the first phase of water immersion treatment (20.46%). This occurrence is affected by the water resistance of the adhesive decreased due to hot-water immersion treatment, thus enhancing the water infiltration into the boards. The type A bamboo particleboard weight bonded with 25% citric acid changed by approximately −4.61 to −15.74% with the following drying treatment. The scale of type B bamboo particleboard weight change is similar to that of type A bamboo particleboards. Nevertheless, the type B bamboo particleboards weight changes due to each phase of drying treatment were lower, approximately −3.83 to −9.63%. This outcome demonstrates that the bonding between type B bamboo particles and citric acid was superior to type A bamboo particles, thus more dominant to resist water due to a more intimate contact area among particles with citric acid, by the lower concentration of silica in bamboo skin.

F. Bonding Adhesion of Bamboo Particles and Citric Acid Analysis by FTIR

The bonding mechanisms of bamboo particleboards with various citric acid levels were evaluated using FT-IR spectroscopy. The board infrared (IR) spectra are presented in Figure 14 and Figure 15.

In the FTIR analysis, the peak intensity at approximately 1714 cm^{-1} is typically ascribed to C=O stretching due to carboxyl groups and/or C=O ester groups (Yang et al., 1996; Žagar & Grdadolnik, 2003). Kusumah, Arinana, et al., (2017) and Kusumah, Umemura, et al., (2017a) mentioned that carbonyl groups were represented as ester linkage between hydroxyl groups of sweet sorghum bagasse

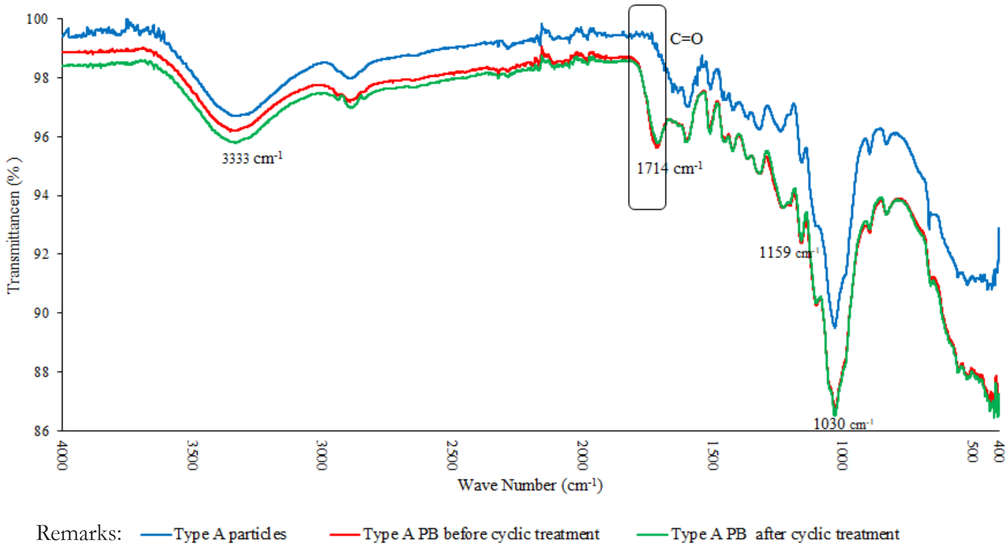

Figure 14. FTIR spectrogram of type A bamboo particles and particleboards made of type A bamboo particles bonded with 25% citric acid before and after cyclic aging treatment

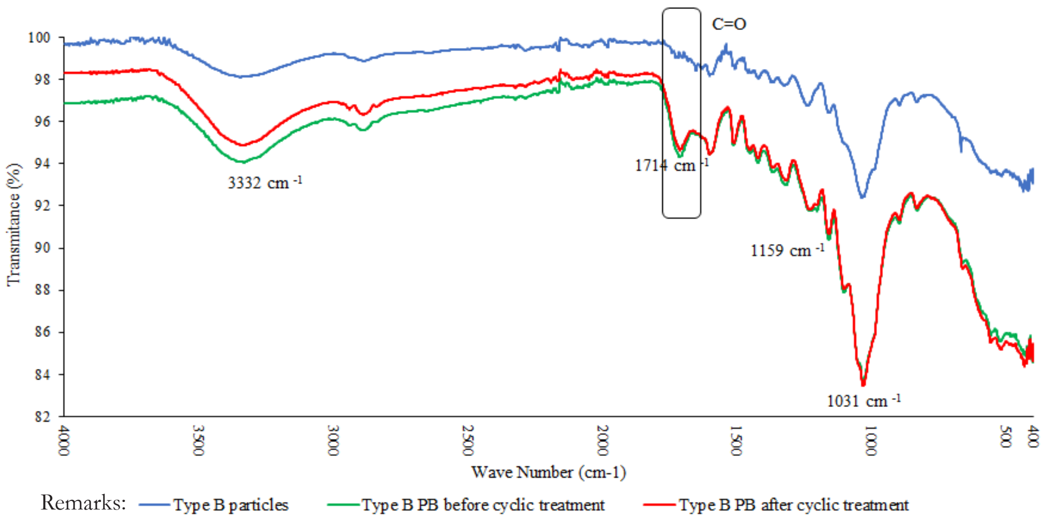

Figure 15. . FTIR spectrogram of type B bamboo particles and particleboards made of type B bamboo particles bonded with 25% citric acid before and after cyclic aging treatment

(lignocellulosic materials) and carboxyl groups of citric acid. As described in Figure 14, FTIR spectra of Type A bamboo particles show no peak at 1713 cm^{-1}. On the other hand, FTIR spectra of particleboard made from type A bamboo particles show a peak at 1713 cm^{-1} before and after cyclic aging treatment. It has proven the forming of ester linkage in particleboards.

According to the results presented in Table 4, band characteristics were changed from type A and type B particleboards before and after cyclic treatment.

The peak at nearly 1714 cm^{-1} was assigned to the C=O stretch in esters. The IR spectra of type B bamboo particleboards exhibit that the intensities of the transmittance peaks at nearly 1714 cm^{-1} representing that carboxyl groups in citric acid reacted with the hydroxyl groups of type B bamboo particles to form ester linkages (Figure 15). The bonding mechanism is very similar to that detected in wood particleboards bonded with citric acid (Umemura et al., 2015). Accordingly, ester linkages formation would cause good adhesiveness and convey superior physical characteristics to the bamboo particleboards.

IV. CONCLUSION

The effects of bamboo particle type and citric acid level on the mechanical and physical properties of Sembilang bamboo particleboards bonded with citric acid were evaluated. The type B particleboards' internal bond (IB), modulus of rupture (MOR), water absorption (WA), and thickness swelling (TS) were superior compared to the type A particleboards. This was influenced by the lower concentration of silica in type B particleboards, which tend to allow an intimate contact area among particles and citric acid then produced better quality particleboards compared to type A particleboards. On the other hand, type A particleboard shows higher quality, in terms of modulus elasticity, due to bamboo outer skin contributing to particleboards' rigidity. Furthermore, the MOR, MOE, and IB values of type B bamboo particleboards satisfied the type 18 requirements of JIS A 5908. Although, the screw holding power of type B bamboo particleboards only satisfied type 8 of JIS A 5908. The physical properties of Sembilang bamboo particleboards were also improved when using type B bamboo particles and bonded with 25% citric acid. Based on the infrared spectra, ester linkages appeared clearly in Sembilang bamboo particleboards manufactured with type B bamboo particles and bonded with 25% citric acid. This means that removing bamboo skin containing silica affected the mechanical and physical properties of Sembilang bamboo particleboards bonded with citric acid.

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