Utilization of Sweet Sorghum Bagasse and Citric Acid in the Manufacturing of Particleboard. III: Influence of Adding Sucrose on the Properties of Particleboard

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Sweet sorghum bagasse (SSB) and citric acid (CA) were used as sustainable raw materials in the development of environmentally friendly particleboard. Sucrose was added to improve the mechanical and physical properties of the particleboard. The effects of the weight ratio between CA and sucrose on the physical properties of the particleboards were investigated. The mechanical properties of particleboards bonded with adhesives at 15/85 and 10/90 wt.% ratios of CA to sucrose were superior to particleboard with other ratios. The thickness swelling of the particleboard increased with an increasing sucrose ratio. Moreover, the physical properties of the particleboard were comparable to those of particleboard bonded using phenol formaldehyde (PF) resin and satisfied the requirements of the type 18 JIS A 5908 (2003) standard. Also, the brittleness of the particleboard was decreased by adding sucrose. Low formaldehyde emission and biological durability against termites and decay were obtained by particleboard under suitable ratios of CA to sucrose. According to the results from thermal analysis and infrared spectra measurement, reactions leading to ester linkages occurred among the CA, sucrose, and SSB components.

Keywords: Citric acid; Particleboard; Sucrose; Sweet sorghum bagasse

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INTRODUCTION

The conservation of forest resources worldwide has become paramount due to the detrimental effects of global deforestation (FAO 2016). Consequently, the utilization of non-wood lignocellulosic resources such as agricultural plants for the raw material of particleboard will certainly increase in the future. Recently, the bagasse of sweet sorghum *(Sorghum bicolor L. Moench)*, typically disposed as a waste, has been used in the development of environmentally friendly particleboard bonded with citric acid (CA) (Kusumah *et al.* 2016, 2017).

In previous reports, particleboard made from sweet sorghum bagasse (SSB) bonded with the CA content of 20 wt.% under a pressing temperature of 200 °C for 10 min had mechanical and physical properties that satisfied the type 18 JIS standard for particleboard (Kusumah *et al.* 2016, 2017). In addition, the mechanical and physical properties of the

SSB-particleboard bonded with CA were better than those of the wood-particleboard (Umemura *et al.* 2013, 2014) and bamboo-particleboard (Widyorini *et al.* 2016) bonded with CA. However, the modulus of rupture (MOR) of the sweet sorghum bagasse (SSB) particleboard was lower than that of the particleboard bonded with phenol formaldehyde (PF) (Kusumah *et al.* 2017). Moreover, Kusumah *et al.* (2017) also showed that the screw holding (SH) of the CA bonded SSB particleboard was lower than the type 18 requirements from JIS A 5908 (2003). These phenomena were due to the brittleness resulting from the high content of CA as an acidic compound. Jonsson and Martin (2016) reported that amorphous polysaccharides of lignocellulose were easily degraded by acidic compounds. The degradation of amorphous polysaccharides is likely a main factor affecting brittleness in wood (Phuong *et al.* 2007). They mentioned also that the brittleness caused the wood to absorb relatively little energy during breakage; hence the wood had low mechanical properties such as MOR and screw holding. By reducing the amount of CA, the brittleness of particleboard can be reduced, and the properties of particleboard will improve.

Previously, Umemura *et al.* (2013, 2014) found that adding sucrose when manufacturing particleboard and CA as a binding agent improved the board mechanical and physical properties. Sucrose is a common disaccharide used as a condiment and food ingredient, and its chemical characteristics are well researched. The heat derivatives of sucrose have a hydroxyl group that could react with a carboxyl group of CA (Umemura *et al.* 2014). Therefore, in this study, sucrose was added in the manufacturing of particleboard from SSB and CA to reduce amount of CA as a main factor affecting brittleness and improve its mechanical and physical properties. In this study, the effects of adding sucrose on the mechanical and physical properties of sweet sorghum bagasse particleboard were investigated. In addition, the formaldehyde emissions, termite and decay resistance, thermal stability of SSB particles sprayed by CA-sucrose-based adhesive, and infrared spectra (IR) of the particleboard were examined.

EXPERIMENTAL

Preparation of Materials

SSB was collected from a research field at the Center for Innovation of the Indonesian Institute of Sciences (LIPI) in Indonesia. SSB particles were prepared using a chipper and knife-ring flaker (Pallmann Maschinenfabrik GmBH & Co. KG, Zweibrucken, Germany). The particles were screened, and those that remained between aperture sizes of 0.9 mm to 5.9 mm were used as a raw material. The particles were dried in an oven at 80 °C for 12 h; at that condition, the moisture content was less than 4%. CA and anhydrous sucrose of extra purity grade were purchased from Nacalai Tesque Inc. (Kyoto, Japan) and used without further purification. Different weight ratio mixtures of CA and sucrose (Table 1) were dissolved in water at a concentration of 59 wt.%, and those mixture solutions were used as adhesives. The pH and viscosity of the adhesive at 20 °C were measured using a pH meter and viscosity meter, respectively.

Manufacturing of Particleboards

Resin content, pressing temperature, and time were decided upon based on the results from Kusumah *et al.* (2016), where 20 wt.%, 200 °C, and 10 min were used, respectively. The SSB particles were sprayed with adhesives made from various CA:sucrose ratios, and the particles were dried at 80 °C for 12 h to reach a moisture content

of approximately 10%. Subsequently, the particles were formed into mats using a 300 mm \times 300 mm forming box. Particleboard size and target density were 300 mm \times 300 mm \times 9 mm and 0.8 g/cm³, respectively. Particleboard thickness was controlled by a 9 mm thick metal bar during the hot-pressing process. Based on the ratios of CA and sucrose, the particleboards formed were classified into 8 types (Table 1).

Type of Particleboard	Mixture Ratio of CA to Sucrose (wt.%)	Concentration (wt.%)	Viscosity at 20 °C (mPa⋅s)	рН
A	100:0		30	0.30
В	75:25		21	0.55
С	50:50		22	1.00
D	25:75	50	25	1.25
E	20:80	59	50	1.26
F	15:85		50	1.36
G	10:90		60	1.55
H	0:100		70	2.77

Table 1. Viscosity and pH of Mixture Solution of CA and Sucrose

Evaluation of Mechanical and Physical Properties of Particleboards

The particleboards were tested after conditioning for 1 week at room temperature (20 °C) and a relative humidity of approximately 60%. Each specimen for mechanical (*i.e.*, bending properties, internal bonding (IB), screw holding (SH), and impact strength) and physical (*i.e.*, thickness swelling (TS) and water absorption (WA)) properties testing was cut from the fabricated particleboard using a circular saw. The bending properties, IB, SH, and TS were evaluated according to JIS A 5908 (2003). In addition, the Charpy impact strength test was evaluated referring to JIS K 7111-1 (2006).

The bending properties of the boards, *i.e.*, the modulus of rupture (MOR) and the modulus of elasticity (MOE), were evaluated by conducting a three-point loading of bending test on a 200 mm \times 30 mm \times 9 mm specimen of each board under dry conditions. The loading speed and effective span were 10 mm/min and 150 mm, respectively. Furthermore, the brittleness of particleboards was investigated using a load-deflection curve from bending test. The method of analyzing brittleness was described by Phuong *et al.* (2007).

The Charpy impact test was completed using a digital impact tester DG-CD (Toyo Seiki Seisaku-sho, Ltd, Tokyo, Japan). A rectangular specimen of 80 mm \times 10 mm \times 9 mm was prepared, and the impact strength in the flatwise direction was measured with an unnotched sample.

The IB strength was investigated using a 50 mm \times 50 mm \times 9 mm specimen of each board with a tension loading speed of approximately 2 mm/min. The TS and water absorption (WA) values of each board were immersed in water at 20 °C for 24 h and measured for specimens with the same size as those used in the IB test.

The SH of specimens 50 mm \times 100 mm were tested with a drill that had a diameter of 2 mm to make holes of approximately 3 mm deep placed in two center positions of the board cross section. The distance of each hole was 25 mm from the edge side as described in JIS A 5908 (2003). Screws used for the SH test were 2.7 mm in diameter and 16 mm in length. The screws were inserted into a 9 mm hole, which left 7 mm of shank free for

loading grip. The depth of the inserting screw into the hole was a modification of the JIS method requiring that the screw depth be approximately 11 mm. The pulling out load speed was about 2 mm/min.

After the TS of 50 mm \times 50 mm specimens were measured, the suitable particleboard specimens were subjected to a cyclic aging treatment in which they were dried at 105 °C for 10 h, immersed in warm water at 70 °C for 24 h, dried at 105 °C for 10 h, immersed in boiling water for 4 h, and dried at 105 °C for 10 h. The change in thickness of the specimens that occurred after each cyclic aging treatment was determined. Each experiment was performed in five replications, and the average values and standard deviations were calculated. The MOR, MOE, and IB values of the boards shown in the figures are corrected for each target density based on regression lines between the actual values of the mechanical properties and the specimen densities.

Formaldehyde Emission

The formaldehyde emission of suitable types of particleboards were measured. Specimens of 150 mm \times 50 mm were measured by the desiccator method outlined in JIS A 1460 (2001). Ten replicates were tested for a total surface board area of approximately 1800 cm². The specimens were placed in a desiccator containing a vessel with water. The formaldehyde released from the specimens at 20 °C over 24 h was absorbed by the water solution. This solution was thoroughly mixed before it was measured. The detailed procedures of the formaldehyde emission measurement are found in JIS A 1460 (2001). The absorbance of the formaldehyde emission in the water solution was measured using a spectrophotometer at 412 nm, and distilled water used as a control. The concentration of the formaldehyde from the test piece absorbed into the water in the glass crystallizing dish inside the desiccator was calculated using the formula found in JIS A 1460 (2001).

Termite and Decay Resistance Test

Termite and decay resistance of suitable types of particleboard as well as type A particleboard were evaluated. A 20 mm \times 20 mm \times 9 mm of the specimens sizes were used for each test of termite and decay resistance. The number of specimens for each test of termite and decay resistance was five.

The procedure of termite resistance test referred to the Japan Wood Protection Association standard (JWPAS-TE) (2011). The specimens of termite resistance test were exposed to the subterranean termite *Coptotermes formosanus* Holmgren. The lower ends of PVC cylinders (80 mm in diameter and 60 mm in height) that used as containers for the termite test were sealed with a 5 mm thick hard dental plaster (New Plastone, GC Corp., Tokyo, Japan). The test specimen was placed at the center part of the container. The 150 workers and 15 soldiers of termites were introduced into each test container. These kind of termites were collected from a laboratory termite colony at the Department of Forest Product, Faculty of Forestry, Bogor Agricultural University (IPB), Bogor, Indonesia. As a control, small wood blocks (20 mm \times 20 mm \times 10 mm) of akamatsu wood (*Pinus densiflora*) were used. The water were supplied to the specimens by putted the assembled containers on damp cotton pads and it was left in the darkness room with 28 °C and 85% relative humidity of room conditions for 3 weeks. The mass loss of the specimens that caused by termite attack was calculated based on the difference between the initial and final oven dry weights of the specimens after cleaning off the debris from the termite attack.

The method of decay test referred to the JWPAS-FE (2011). A monoculture decay test was attempted with brown-rot fungus (*Fomitopsis palustris*) (Berk. Et Curt) Gilbn. &

Ryv. (FFPRI 0507) and white-rot fungus (Trametes versicolor) (L.:Fr.) Pilat. (FFPRI 1030). A stock culture of either T. versicolor or F. palustris was used for inoculation of A 100 mL aliquot of liquid medium containing 1.5% malt extract, 0.3% peptone, and 4% glucose. The incubation of the inoculated liquid medium was carried out by a shaker at 120 rpm at 26 °C for 10 days. For the *T. versicolor* testing, 80 mL to 85 mL of nutrient solution containing 4% glucose, 0.3% peptone, and 1.5% malt extract was used for permeation of 250 g medium of sea sand in a glass jar. 50% of the quantity of each component was used for F. palustris. The volume of these liquid fungal stock cultures that were used in inoculating the jars was about 3 mL to 4 mL. The specimens were sterilized with gaseous ethylene oxide after measuring the oven dry weight of the board specimens. Three specimens were put on top of the growing mycelium when the mycelium had fully covered the medium in the glass jars. A plastic mesh spacer was utilized just for F. palustris. The control specimens that used in the decay test were small wood blocks (20 mm \times 20 mm \times 10 mm) of sugi wood (Cryptomeria japonica). The incubation of the test jars was conducted at 27 °C and for 12 weeks. The decay resistance test of each board type was attempted in nine replicates. The average mass loss (%) was calculated from the oven dry weights of nine specimens before and after the decay procedure. This average mass loss (%) indicates the level of the fungal attack.

Thermal Analysis

The specimens used in the thermal analysis were SSB, CA, sucrose, and CAsucrose mixture solutions with various ratios. As previously mentioned, SSB particles were sprayed with CA-sucrose based adhesive. The SSB particles sprayed with the adhesive were categorized into 8 types based on the ratio of citric acid to sucrose as mentioned in Table 1; *i.e.*, type A to H. All specimens were dried at room temperature for one day and pulverized into a less than 150 μ m mesh. All specimens were freeze-dried for 1 h. Thermogravimetric analysis (TGA) was conducted using a TGA 2050 (TA Instruments, New Castle, USA). The powder was scanned from room temperature to 400 °C at a rate of 10 °C/min under nitrogen purging. The differential scanning calorimetry (DSC) measurement was completed using a DSC2500 (TA Instruments). The powder was encapsulated in an aluminum pan and scanned from room temperature to 400 °C at a rate of 10 °C/min under nitrogen purging.

Fourier Transform Infrared Spectroscopy (FTIR)

The edges of the specimen after the cyclic aging treatment were scraped to obtain particles. The particles were ground into a powder and dried in a vacuum drying oven at 60 °C for 12 h. Infrared (IR) spectral data was obtained with a FTIR spectrophotometer (FT/IR-4200; JASCO Corporation, Tokyo, Japan) using the KBr disk method. Spectra were recorded with an average of 16 scans at a resolution of 4 cm⁻¹.

Statistical Analysis

Data for each test were statistically analyzed. The analysis of variance was used to evaluate the significance in difference between factors and levels. Comparison of the means was done by using Duncan post hoc test to identify which groups were significantly different from other groups at 95% confidence level.

RESULTS AND DISCUSSION

Mechanical Properties of Particleboard

Figure 1 shows the bending properties of particleboard under various weight ratios of CA and sucrose. The MOR and MOE values increased gradually as the sucrose ratio increased but exceedingly decreased for the H type of particleboard. The G type particleboard had the highest MOR and MOE average values of 30.22 MPa and 7.07 MPa, respectively. The MOR value of the G type of particleboard was comparable to the MOR of particleboard bonded with PF (32.9 MPa) (Kusumah *et al.* 2017). These results indicated that the addition of sucrose effectively improved the bending properties of particleboard. Except for the type H particleboard, the MOR value of all the particleboards satisfied the type 18 JIS A 5908 (2003) standard (> 18 MPa).



Fig. 1. Bending properties of particleboard bonded with 20 wt.% CA-sucrose based adhesive. The error bar indicates standard deviation (n = 5).



Fig. 2. Brittleness of particleboard bonded with 20 wt.% CA-sucrose-based adhesive. The error bar indicates standard deviation (n = 5).

The load deflection curve of the static bending of particleboard was used to calculate the brittleness of the particleboard (Phuong *et al.* 2007), as shown in Fig. 2. In addition, the brittleness of particleboard bonded with PF was calculated by using the bending data in Kusumah *et al.* (2017). In Fig. 2, the brittleness of particleboard decreased remarkably with an increasing sucrose ratio. Moreover, the brittleness of type G particleboard (27%) was comparable with that of particleboard bonded with PF (25%). This means that the addition of sucrose effectively reduced the brittleness of SSB particleboard bonded with CA.

Figure 3 shows the Charpy impact strength of SSB particleboard under several weight ratios of CA and sucrose. The Charpy impact strength of the SSB particleboards increased gradually with an increasing sucrose ratio, which is seen in the A to E types of particleboards, where they seemed to hold steady until the H type. Commonly, a high Charpy impact strength value indicates low brittleness (Koehler 1933). Therefore, based on the impact strength of SSB particleboard, the brittleness of SSB particleboard was effectively reduced by adding sucrose. In other words, reducing the CA content in the adhesive by adding sucrose and was an effective method to decrease the brittleness, which deteriorates the bending properties of particleboard (Kusumah *et al.* 2017).



Fig. 3. Charpy impact strength of particleboard bonded with 20 wt.% CA-sucrose-based adhesive. The error bar indicates standard deviation (n = 5).

The IB values of the particleboard are shown in Fig. 4. The IB strengths of particleboards continuously increased with an increasing sucrose ratio until the G type and then decreased dramatically for the H type. The IB strength of F and G types were approximately 33% greater than that of the A type and two times higher than that of the H type. The addition of sucrose provided an improvement in the bond strength between particles. Furthermore, the IB strengths of both F (1.15 MPa) and G (1.17 MPa) types were considerably higher than particleboard bonded with PF (0.78 MPa) (Kusumah *et al.* 2017). The IB strengths of all types of particleboards fulfilled the requirement of the type 18 JIS standard (> 0.3 MPa).

Judging from the results of bending properties, brittleness, impact strength, and IB as shown in Figs. 1 to 4, the F and G types of particleboards had excellent mechanical properties. Therefore, the SH values of the F and G types were investigated and found to

be 502 and 525 N, respectively. Those SH values were about 50% higher than that of the particleboard bonded with CA only (348 N) (Kusumah *et al.* 2017). In addition, the SH of the F and G types satisfied the type 18 requirement of JIS A 5908 (2003) (\geq 500 N). This result indicated that the addition of sucrose effectively improved the SH of the particleboard. This phenomenon occurred because the brittleness of the particleboard was decreased with the increasing sucrose ratio, as shown in Fig. 2. The addition of sucrose resolved the problem of the low SH and high brittleness of the particleboard resulting from the high CA content (Kusumah *et al.* 2017).



Fig. 4. Internal bonding (IB) of particleboard bonded with 20 wt.% CA-sucrose-based adhesive. The error bar indicates standard deviation (n = 5).

Physical Properties of Particleboard

Figure 5 shows the TS and WA values of particleboards with different weight ratios of CA and sucrose. The TS values of the particleboard increased slightly as the sucrose ratio was increased, which is shown in the values of the B to G types of particleboards, and then increased sharply when the particleboard only used sucrose (H type). However, the TS values of the C to G types of particleboards were not significantly different (p > 0.05). The TS values of those types of particleboards were in the range of 11.3% to 12%. This means that adding sucrose from the middle to high ratio of sucrose did not affect the TS increase. Those TS values were slightly higher than those of A (10.2%) and B (10.4%) type particleboards. Moreover, the TS of the H type of particleboard (24.2%) was two times higher than those of the C to G types of particleboards. In other words, the H type of particleboard had low dimensional stability. With the exception of the H type of particleboard, the TS values of the particleboards complied with the type 18 type JIS A 5908 (2003) standard ($\leq 12\%$). In addition, the TS values of those particleboards were 11.3% to 12, and they were lower than particleboards bonded with PF (20.6%) (Kusumah et al. 2017). This result indicated that the particleboard maintained a good level of dimensional stability with the addition of sucrose from B type to the G type.

The WA values of the B through G types of particleboards were significantly lower than A and H types. CA reacts effectively with SSB components containing hydroxyl groups when the CA content is 20 wt.% under a pressing temperature of 200 °C for 10 min (Kusumah *et al.* 2016, 2017).

In addition, Umemura *et al.* (2014) reported that CA possibly reacts with sucrose and its heat derivatives having hydroxyl groups. The formation of those chemical linkages likely inhibited the water absorption of the particleboard during the water-immersion treatment. As a result, the water absorption of sucrose that contained particleboard decreased. In fact, the WA value (41.12%) of the G type of particleboard was lower than that of particleboard bonded with PF (51.24%) (Kusumah *et al.* 2017).

The thickness change of particleboard was measured continuously by a cyclic accelerated aging treatment test to clarify in greater detail the dimensional stability under severe conditions.



Fig. 5. Thickness swelling (TS) and water absorption (WA) of particleboard bonded with 20 wt.% CA-sucrose-based adhesive. The error bar indicates standard deviation (n = 5).

Based on the results shown in Fig. 5, the particleboards of F and G types were used as representative specimens of particleboard in the investigation of the thickness change. The thickness change of the A type of particleboard was also measured as a reference.

Figure 6 shows the thickness change of A, F, and G types of particleboards in the cyclic accelerated aging treatment. The accelerated aging treatment led to a stepwise increase of thickness regardless of the kind of particleboard. The thickness change of the particleboard decreased when there was a decrease in the sucrose ratio. The thickness change of F (9.3%) and G (11.8%) types of particleboards were slightly higher than the A type of particleboard (7.9%).

Based on the statistical analysis, the last thickness change value of the F type particleboard was not significantly different (p > 0.05) from the A type particleboard. Thus, the good dimensional stability of the particleboard was maintained with the addition of sucrose in the adhesion system even after severe treatment.



Fig. 6. Thickness change of A, F, and G types of particleboard bonded with 20 wt.% CA-sucrosebased adhesive in cyclic accelerated aging treatment. The error bar indicates standard deviation (n = 5).

Formaldehyde Emission, Termite, and Decay Resistance

Based on Haworth and Jones (1944), sucrose is converted to its respective monosaccharides and generate aldehyde compounds when it is heated. Formaldehyde is the simplest of the aldehydes and it is a volatile organic compound (VOC) that has short-term and long-term adverse health effects (Gminski *et al.* 2011a, b). Waller and Curtis (2003) mentioned that paper treated with sucrose has low durability against termites and that a high content of sucrose in the cellular lumen of wood resulted in low decay resistance (Severo *et al.* 2016). Therefore, termite and decay resistance and formaldehyde emissions were evaluated.

The results shown in Figs. 1 to 6 show that the most effective particleboard types were F and G. Accordingly, the formaldehyde emissions of the F and G types of particleboards were evaluated. Those particleboards types (0.00 mg/L) did not emit formaldehyde such as the boards bonded with CA (0.00 mg/L) and PF (0.01 mg/L) (Kusumah *et al.* 2017). The formaldehyde emission values were lower than the lowest formaldehyde emission criterion ($F^{\star\star\star\star}$) (< 0.4 mg/L) of JIS A 5908 (2003).

As with formaldehyde emission, the biological durability against termites and decay of F and G types of particleboard were investigated. The results are shown in Table 2. The termite mortality in the F and G types of particleboards were comparable with that of the A type and of particleboard bonded with PF (46.40%) (Kusumah *et al.* 2017). Additionally, the mass losses of the F and G type particleboards were relatively the same as the A type of particleboard and particleboard bonded with PF (3.92%) (Kusumah *et al.* 2017). Therefore, the particleboards had good termite resistance even when there was a high concentration of sucrose in the particleboard.

Biological-Durability	Specimen Type						
Testing	A type	F type	G type	Control			
Termite resistance							
Termite mortality (%)	45.87 (1.10)	43.20 (2.33)	43.07 (2.14)	23.60 (4.46)			
Mass loss (%)	4.33 (1.72)	5.27 (1.07)	5.45 (1.26)	13.43 (4.04)			
Decay resistance							
Mass loss of the specimen exposed to:							
White-rot fungus (%)	21.63 (11.7)	23.27 (7.9)	23.42 (6.9)	30.15 (5.12)			
Brown-rot fungus (%)	5,43 (1,36)	6.12 (1.2)	6.40 (0.9)	10.16 (2.05)			

Table 2. Termite and Decay Resistance of A, F, G Types of Particleboard

Values in parentheses are the standard deviation. A, F, and G types are particleboard bonded with 20 wt.% resin content of CA-sucrose-based adhesive under A (100/0), F (15/85), and G (10/90) ratio of citric acid to sucrose; control is sugi (*Cryptomeria japonica*) wood specimen for decay test and akamatsu (*Pinus densiflora*) for termite test.

The mass losses of F and G types after 3 months of exposure to white-rot fungus were relatively similar with the A type. Moreover, those mass losses were higher than particleboard bonded with PF (9.09%) (Kusumah *et al.* 2017). Hoareau *et al.* (2006) and Yalinkilic *et al.* (1998) found that particleboard bonded with PF has good durability against decay due to phenolic compounds and the formaldehyde being resistant to decay fungi. Furthermore, the mass losses of the F and G types after exposure to brown-rot fungus were comparable to the A type of particleboard and particleboard bonded with PF (4.54%) (Kusumah *et al.* 2017). This indicates that the heat treated mixture of CA and sucrose showed an effective inhibition against decay. Despot *et al.* (2008) mentioned that the improvement in decay resistance in the wood modified by CA was clearly the result of the cross-linking of CA and hydroxyl groups of the wood components. In this study the CA could have cross-linked the hydroxyl components of SSB and sucrose, which would result in the particleboard having good decay resistance.

Thermal and FTIR Analyses

Investigation of the adhesion mechanism was attempted by thermal and FTIR analyses. The A and B type specimens showed similar behavior in the DSC and TG curves. Moreover, the C through G type specimens showed similar DSC and TG curves. Thus, Fig. 7 (I) and (III) shows just the results of A, F, and H types as representative of SSB particles sprayed with a CA-sucrose mix solution. Figure 7 shows the DSC of the SSB, A, F, and H types of particle specimens (I); the DSC of the CA, sucrose, and CA-sucrose mix solution with a weight ratio of 15/85 (F) as references (II); and the TG of the particle specimens of A, F, and H types (III).

In Fig. 7 (I), the DSC curves of SSB particles show an endothermic peak at around 90 °C, while the A, F, and H types show an endothermic peak at approximately 75 °C. This endothermic peak did not appear in the CA-sucrose mix solutions or in the CA and sucrose only formulations. Hardly any weight loss of those particle specimens was recognized at that temperature, as shown in Fig. 7 (III). Mehrotra *et al.* (2010) mentioned that the endothermic peak at approximately 90 °C was the consequence of the weakening of the hydrogen bonds between carbohydrates. Therefore, the endothermic peaks of SSB, A, F, and G types of particle specimens were due to the weakening of the hydrogen bonds between carbohydrates.

In Fig. 7 (I), two endothermic peaks appeared clearly at approximately 150 °C to 200 °C in the A type particle specimen. The first endothermic peak at approximately 150 °C shows the melting point of CA (Barbooti and Al-Sammerrai 1986), and the second endothermic peak with a temperature at approximately 180 °C indicated the decomposition of CA (Barbooti and Al-Sammerrai 1986). These endothermic peaks appeared unclear in the F type particle specimen. Also, the endothermic peak was at approximately 225 °C in the H type particle specimens, indicating the decomposition of sucrose (Gintner *et al.* 1989; Enggleston *et al.* 1996) did not appear in the F type. These results suggested that some interactions or reactions occurred between CA, sucrose, and SSB components in the F type particle specimen.

In the DSC curve of the CA-sucrose mix compound shown in Fig. 7 (II), the same phenomena occurred that was seen in the F type. The DSC curve of CA-sucrose mix compound has two endothermic peaks at around 160 and 175 °C. Those endothermic peaks appeared at different temperatures from those of CA only (157 °C) and sucrose only. In addition, the endothermic peak at around 225 °C appeared in CA and sucrose as these compounds decomposed, but the endothermic peak did not appear in the CA-Su mix compound, which was the F type of particleboard. It was expected that some of the interactions or reactions between CA and sucrose occurred. Therefore, in Fig. 7 (III), the weight of the F type particle specimen especially at temperatures of more than 200 °C was more moderately decreased than the A and H type particle specimens due to an interaction or reactions between CA, sucrose, and SSB components, which occurred at a low temperature of approximately 150 °C to 200 °C as shown in the DSC curve of the F type.

The infrared (IR) spectra of the F type of particleboard was measured to clarify the effect of the sucrose addition on the chemical change of SSB particleboard. Figure 8 shows the general results of IR spectra of SSB particles and F types of particleboard after cyclic aging treatment. An absorption peak at approximately 1727 cm⁻¹ appeared clearly in the F specimens. The peak at 1727 cm⁻¹ was typically ascribed to C=O stretching due to carboxyl groups and/or the C=O ester groups (Yang *et al.* 1996 and Zagar *et al.* 2003). An additional absorbance intensity peak at 1245 cm⁻¹ was clearly observed in the F type. This peak was related to the C=O stretching vibration band of ester groups (Aflori and Drobota 2015).

The appearance of ester groups in the IR spectra of F specimen indicates that the carboxyl groups of CA reacted with hydroxyl groups of SSB and sucrose to form ester linkages (Umemura *et al.* 2012; Liao *et al.* 2016). Kwok *et al.* (2010) reported that sucrose was hydrolyzed to its respective monosaccharides, glucose, and fructose in the mixtures of sucrose-CA solution.

Generally, those monosaccharides exist as carbohydrates having hydroxyl groups. Essentially, the lignocellulose materials have abundant hydroxyl groups derived from lignocellulose components such as cellulose, hemicellulose, and lignin (Zhang *et al.* 2015). Therefore, the particleboard bonded with the adhesive under a suitable weight ratio of CA to sucrose and appeared to have more ester linkage branches than particleboard bonded with CA only. This was due to the carboxyl groups of CA reacting with hydroxyl groups of sucrose and SSB. The consequent formation of ester linkages would improve the adhesiveness. As a result, the mechanical and physical properties of the particleboard was improved by adding sucrose.

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Fig. 7. Differential scanning calorimetry (DSC) of powder of original sweet sorghum bagasse (SSB) of A, F, and H types of particles specimen (I); DSC of citric acid (CA), sucrose, and CA-sucrose mix solution in 15/85 wt.% (II); Thermogravimetric (TG) of A, F, and H types of particles specimen (III).



Fig. 8. IR spectra of SSB (Kusumah *et al.* 2016) and F type of particleboard after cyclic aging treatment.

CONCLUSIONS

- 1. The particleboards bonded with adhesives at 15/85 and 10/90 wt.% ratios of CA to sucrose had higher mechanical properties, lower brittleness, and comparable dimensional stability compared with those bonded only with CA and PF resin. The bending properties, IB strength, SH, and TS values of those boards satisfied the requirements for the type 18 JIS A 5908 (2003) standard.
- 2. The particleboards bonded with the adhesives at 15/85 and 10/90 wt.% CA:sucrose Su ratios had low formaldehyde emission and satisfied the type 18 JIS A 5908 (2003) standard.
- 3. The particleboards bonded with adhesives at 15/85 and 10/90 wt.% CA:sucrose ratios had good termite and decay resistance, which was similar to those of particleboard bonded with citric acid only.
- 4. Thermal and FTIR analyses demonstrated that citric acid reacted with the sucrose and sweet sorghum bagasse particles to form ester linkages. Ultimately, the mechanical properties of particleboard were enhanced and the particleboard still has good dimensional stability.

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