Bonding Ability of a New Adhesive Composed of Citric Acid-Sucrose for Particleboard

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Citric acid is a potential binding agent for composite products that has three carboxyl groups that can be ester linked with the hydroxyl groups found in wood. The addition of sucrose provides hydroxyl groups and increases the amount of ester groups. This research investigated the bonding ability of a new adhesive composed of citric acid-sucrose for teak particleboard. Citric acid and sucrose were dissolved in water under various ratios, and the concentration of the solution was adjusted to 59 to 60 wt%.This adhesive solution was sprayed onto the particles at 10% resin content based on the weight of air-dried particles. Each mixture was then hot pressed at 180 and 200 °C for 10 min. The physical and mechanical properties of the particleboards were tested, and the results showed that increasing the pressing temperature affected the dimensional stability. However, increasing of citric acid in adhesive composition improved the dimensional stability and mechanical properties of the particleboards. The optimum properties of the board were achieved at a pressing temperature of 200 °C and addition of only 10% citric acid. The results also indicated that the peak intensity of C=O group increased with the addition of citric acid and increasing pressing temperature, indicating that ester linkage occurred. However, the addition of sucrose did not greatly affect the peak intensity of C=O group.

Keywords: Citric acid; Sucrose; Teak; Particleboard; Bonding mechanism; Mechanical properties; Physical properties

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INTRODUCTION

The utilization of adhesives in wood composite manufacturing is important to improve the function of the product. Composite products typically use formaldehyde-based adhesives, such as urea formaldehyde or phenol formaldehyde. Research (Axis Research Mind 2014) suggests that urea formaldehyde consumption will increase to 18.7 million tons by the end of 2016, with 71.9% resulting from fiberboard and plywood production. These adhesives usually exhibit good properties and excellent performance. However, the International Agency for Research on Cancer/IARC monographs volume 88 (IARC 2006) and 100F (IARC 2012) state that formaldehyde is carcinogenic to humans. Therefore, the acceptable levels of formaldehyde emission from wood panel products are becoming more stringent.

Because of formaldehyde's detrimental qualities, the development of eco-friendly biocomposites, which use natural adhesives, is quickly becoming a very lucrative and attractive option. In contrast to fossil fuels, the use of renewable resources (such as biomass) for adhesives provides a remarkable number of environmental advantages. Many studies have focused on finding new adhesives based on renewable materials, such as citric acid (Umemura *et al*. 2011; Widyorini *et al*. 2016), modified lignin (Nasir *et al.* 2013), citric acid with sucrose (Umemura *et al*. 2013, 2015), tannin with sucrose (Zhao and Umemura 2014), and glucose with sucrose (Tondi *et al*. 2012; Lamaming *et al*. 2013). Citric acid, which is a natural organic polycarboxylic acid containing three carboxyl groups, could act as a binding agent for boards made from wood and bark (Umemura *et al*. 2011; Umemura *et al*. 2012), various bamboo species (Widyorini *et al*. 2016), and oil palm frond (Widyorini *et al*. 2012). The results show that citric acid-bonded boards exhibit high mechanical properties and good dimensional stability. In addition, citric acid has also been studied as a cross-linking agent for wood (Vukusic *et al*. 2006; Hasan *et al*. 2007), starch (Reddy and Yang 2010), and as an absorber for heavy metal ions (Thanh and Nhung 2009). Studies have shown that when citric acid is used as a cross-linking chemical, it reacts with the hydroxyl groups and reduces the hygroscopicity of wood as well as the tendency of wood to swell or shrink (Rowell 1991; Vukusic *et al*. 2006). Umemura *et al*. (2012) pointed out that ester linkages can be detected by Fourier transform infrared spectroscopy (FTIR), which indicates that the carboxyl groups from citric acid can react with hydroxyl groups from wood, improving the performance of boards.

Other researchers have focused on utilizing sugar materials, such as glucose and sucrose. Lamaming *et al*. (2013) found that the addition of glucose and sucrose enhanced the modulus of rupture, internal bond strength, thickness swelling, and water absorption of oil palm particleboards. Based on the xylose/arabinose ratio, the boards made with the addition of sucrose consisted of short chain polymers that possessed a large amount of branching with other monosaccahrides. Tondi *et al.* (2012) proposed the possible bonding mechanism of synergy between starch and sucrose in wood adhesion technology. The addition of sucrose into a cementious matrix increased the mechanical properties of concrete, decreasing the setting time and improving concrete behavior in water (Khazma *et al.* 2008). Zhao and Umemura (2014) investigated a new wood adhesive composed of tannin and sucrose. Other studies have reported that the addition of sucrose to softwood particleboards provided higher performance compared with boards bonded with only citric acid (Umemura *et al*. 2013, 2015).

Sucrose is expected to provide additional hydroxyl groups and formed ester-linked bonds with citric acid (Umemura *et al*. 2013). However, a clear bonding mechanism has remained unknown until now. This problem is very important for application in the wood products industry. Therefore, this paper was designed to investigate the characteristics of particleboard bonded using citric acid and sucrose with various compositions. Considering that the melting temperatures of these chemicals are different, the effect of pressing temperature was also studied in this research.

EXPERIMENTAL

Materials

Teak wood particles were collected from the Yogyakarta province area and were used as the board material for these experiments. The particles were screened, and those able to pass through a 10-mesh screen were used as board materials. All particles were air-dried to a moisture content of approximately 12%.

Preparation of Adhesive Solution

Citric acid (anhydrous) and sucrose, both from Weifang Ensign Industry Co. Ltd., China, were used without further purification. Citric acid and sucrose were dissolved in water under a certain ratio, and the solution concentration was adjusted to 59 ~ 60 wt%. The mixture ratios of citric acid/sucrose were 100/0, 75/25, 50/50, 25/75, and 0/100. The solutions were then used as an adhesive. The viscosity and pH of the solutions are shown in Table 1.

Table 1. Viscosity and pH of Mixture Solution of Citric Acid and Sucrose

Mixture ratio of citric acid and sucrose (wt%)	Concentration (wt%)	Viscosity at 25°C (mPa.s)	pH
100:0		15.8	0.85
75:25		17.4	0.95
50:50	59 ~ 60	23.5	1.14
25:75		27.2	1.31
0:100		37.5	6.32

Manufacturing of Particleboard

The solution was used as an adhesive and sprayed onto teak wood particles at 10 wt% resin content, based on the weight of air-dried particles. The sprayed particles were then oven-dried over night at 80 °C to reduce the moisture content. The moisture content of the mat was 4% to 6%. The particles were hand-formed into a mat using a forming box of 250 mm x 250 mm, followed by hot pressing into particleboards with a distance bar of 7 mm to control the board thickness. The boards were pressed at 180 and 200 °C for 10 min under a pressure of 3 MPa. Binderless particleboards were also produced using the same condition, as a control. The target board density of 0.9 g/cm^3, and three replications were applied during this study. Prior to the evaluation of the mechanical and physical properties, all boards were conditioned at ambient conditions for approximately 10 days.

Evaluation of Board Properties

After conditioning, the particleboards were evaluated according to the Japanese Industrial Standard for Particleboards (JIS A 5908 (2003)). Tests were carried out to determine modulus of rupture (MoR), modulus of elasticity (MoE), internal bond strength (IB), thickness swelling (TS), and water absorption (WA). Static three-point bending tests (for both dry and wet conditions) were conducted on a 200 mm x 50 mm x 7 mm specimen. The effective span and loading speed were 150 mm and 10 mm/min, respectively. The wet bending strength test was conducted on specimens after immersion in boiling water for 2 h and in water at 20 °C for 1 h, respectively. The IB test was performed on a 50 mm x 50 mm x 7 mm specimen cut from each board. The same specimen dimensions were also prepared for TS-WA tests after water immersion for 24 h at 20°C. Each experiment was performed in triplicate, and the average value and standard deviation were calculated.

Fourier Transform Infrared Spectroscopy (FTIR)

For citric acid and sucrose bonded particleboard, the FTIR (Shimadzu IRPrestige-21, Japan) analysis was performed. For this purpose, the wet bending strength test samples were used. The material was first dried at 40 °C overnight and powdered using a mill machine. All infrared spectra were obtained using the KBr disk method and recorded by means of an average of 10 scans at a resolution of 16 cm^{-1}.

RESULTS AND DISCUSSION

Physical Properties

The densities of citric acid-sucrose bonded particleboards ranged from 0.85 to 0.94 g/cm^3, irrespective of the condition of manufacture. All of the particleboards could be manufactured without any delamination. However, the color of the boards became darker with increasing pressing temperature, as shown in the research of Widyorini *et al.* (2005), indicating a high degree of hydrolysis or other modification of chemical components during treatment.

Figure 1 shows the thickness swelling and water absorption of the particleboards with various adhesive compositions and pressing temperatures. The results clearly showed that all of the particleboards pressed at 180 °C met the requirements of JIS A 5908 (TS max 12%), except for sucrose-bonded particleboards (62%) and binderless board (66%). The same trend was also found for the water absorption value of the particleboards, with WA values of sucrose-bonded particleboard of 135% and 161% for binderless board. The properties of binderless boards were not good, considering that the adhesive mechanism depends only on the chemical characteristic of the materials which are reacted during steam or heat pressing (Widyorini *et al*. 2005). It is interesting that the TS and WA values of sucrose-bonded particleboards were not very different from that of binderless particleboard pressed at the same pressing temperature (180 °C). This can be attributed to the fact that sucrose more easily dissolves in water than citric acid, as shown by its solubility. After the addition of citric acid, the TS and WA were decreased significantly, indicating that the dimensional stability of the boards improved. This is supposedly due to the ester-linked bonds that were formed. Umemura *et al.* (2013) also deduced the same results for particleboard made from softwood.

Increasing pressing temperature greatly reduced the TS and WA values with the same composition. The pressing temperature was an important component of the reaction between carboxyl groups and hydroxyl groups to form ester groups (McSweeny *et al*. 2006). It was concluded that a press temperature of 200 °C was needed to obtain good dimensional stability of the particleboards, as also found by Umemura *et al.* (2015).

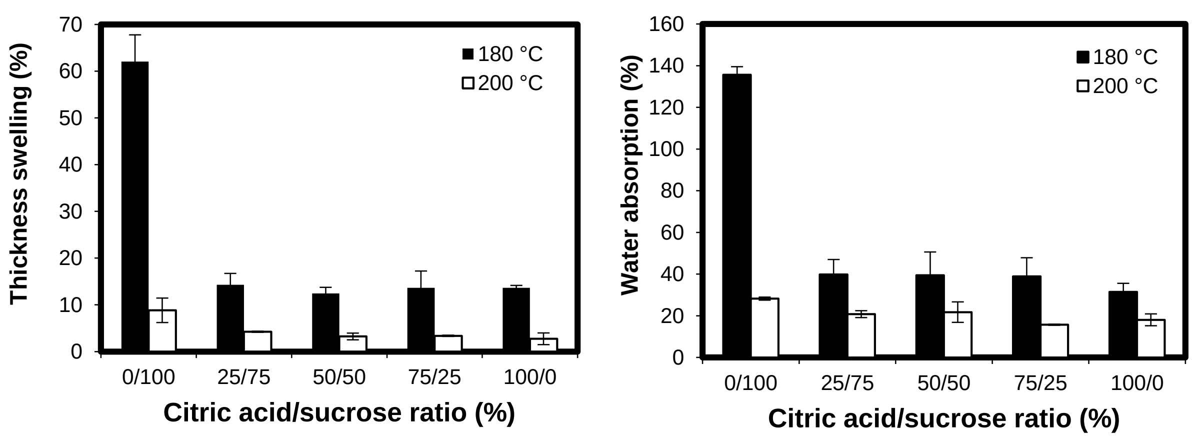

Fig. 1. Thickness swelling and water absorption of particleboard at various adhesive compositions and pressing temperatures. The vertical bar represents standard deviation.

Mechanical Properties

Figure 2 shows the modulus of rupture and modulus of elasticity of teak wood particleboard. Excluding sucrose-bonded particleboard that was pressed at 180 °C and binderless particleboards, all of the particleboards met the requirements of JIS A 5908 8 type for particleboards, *i.e.*, greater than 8 MPa for MoR and more than 2 GPa for MoE. This result was consistent with the dimensional stability of the particleboards. All of particleboards bonded with various composition of sucrose-citric acid had higher bending strength compared to the binderless particleboard. The MoR and MoE of binderless particleboard were 1.4 MPa and 0.6 GPa at 180 °C pressing temperature and 3.1 MPa and 1.1 GPa at 200 °C pressing temperature, respectively. McSweeny *et al*. (2006) stated that pressing temperature was a necessary component of the reaction between carboxyl groups and hydroxyl groups to form ester groups. The highest modulus of rupture and modulus of elastictity could be achieved at a pressing temperature of 200 °C, obtaining values of 12.3 MPa and 3.8 GPa, respectively.

The data show that the adhesive (citric acid/sucrose) composition did not greatly affect the modulus of rupture and modulus elasticity. However, particleboard bonded only with citric acid had the highest bending strength, while sucrose-bonded particleboard had the lowest bending strength. It seemed that carboxyl groups from citric acid reacted well with hydroxyl groups from teak wood to form ester linked groups, providing strong bonding properties. Umemura *et al.* (2013) found different results, where the optimal properties of softwood particleboard were obtained at a citric acid/sucrose ratio of 25/75.

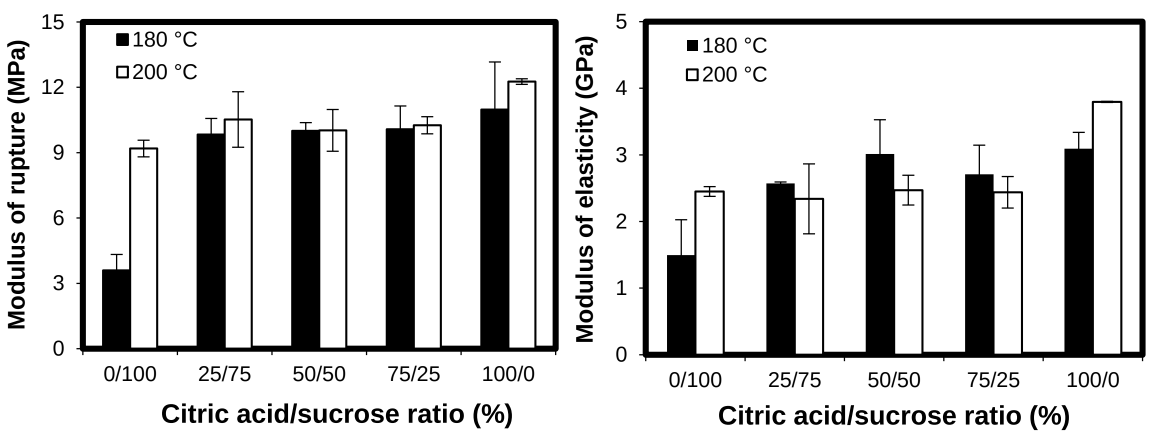

Fig. 2. Modulus of rupture and modulus of elasticity of particleboard at various adhesive compositions and pressing temperatures. Vertical bar represent standard deviation.

The water resistance of the particleboards is shown in Fig. 3. The wet bending strength test was conducted on specimens after immersion in boiling water for 2 h and in water at 20 °C for 1 h, respectively. All of the particleboards bonded with sucrose and pressed at 180 °C were broken into pieces after immersion in boiling treatment for 2 h; therefore, no results were gathered for the wet bending strength. Sucrose adhesive did not provide any water resistance at a pressing temperature of 180 °C; however, the water resistance could be improved by increasing the pressing temperature to 200 °C. The percentage of both the wet/dry modulus of rupture and the modulus of elasticity ranged from 0 to 56%. These values indicated that adhesives composed of citric acid and sucrose had relatively high resistance. Figure 3 shows that the wet/dry ratio for the MoR of particleboards was noticeably affected by the adhesive composition when pressed at 180 °C; however, it was not affected when the particleboard was pressed at 200 °C.

The IB at various adhesive compositions and pressing temperatures is shown in Fig. 4. All of the particleboards had an IB greater than 0.15 MPa (as required by the standard JIS A 5908 (2003) type 8), except for particleboard pressed at 180 °C and bonded using 100% sucrose. The highest IB was achieved after the addition of 100% citric acid, with an IB of 0.37 MPa, which met the requirement of JIS A 5908 (2003) type 18. Umemura *et al.* (2013) reported that the optimum condition was achieved at a citric acid/sucrose ratio of 75/25 for softwood particleboard. It was thought that sucrose would be hydrolyzed and become glucose and fructose, providing hydroxyl groups that could react with the carboxyl groups from citric acid. These different results showed that the chemical components of raw materials significantly influenced the bonding mechanism of the adhesives.

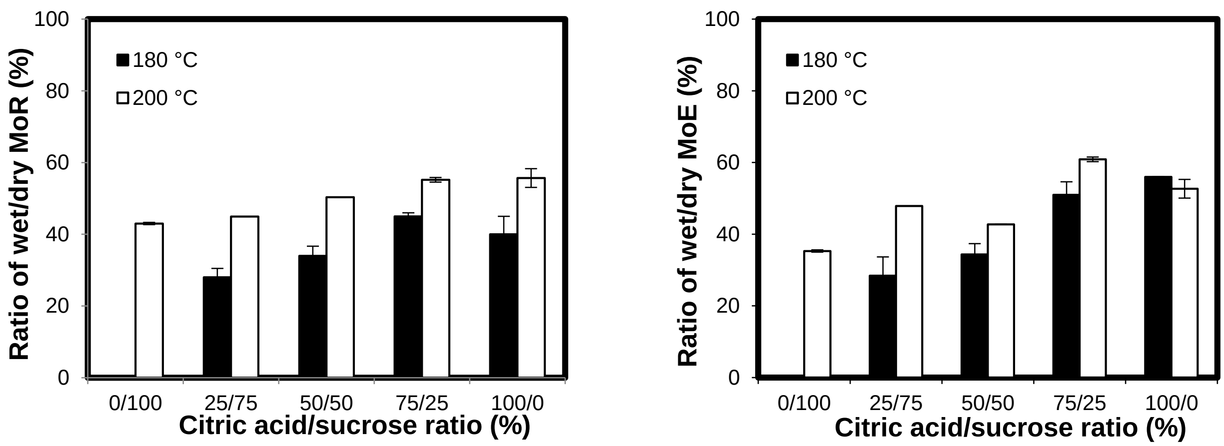

Fig. 3. The ratio of wet/dry bending strength of particleboard at various adhesive compositions and pressing temperatures. The vertical bar represents the standard deviation.

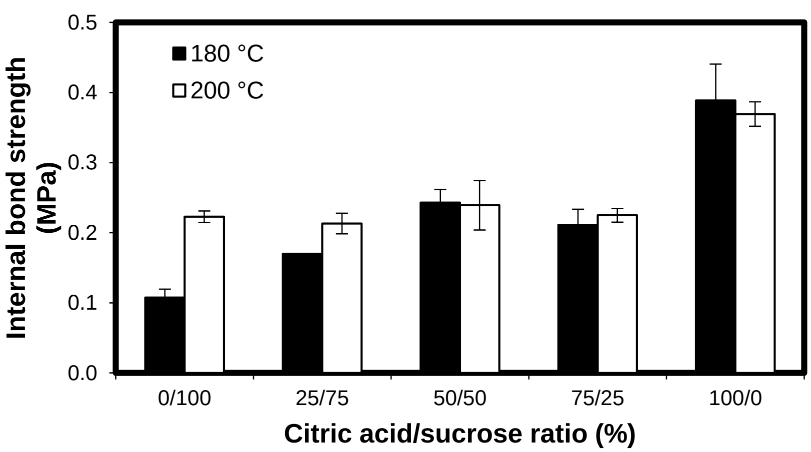

Fig. 4. Internal bond strength of particleboard at various adhesive compositions and pressing temperatures. The vertical bar represents the standard deviation.

The sucrose-only bonded particleboard pressed at 180 °C had the lowest IB value of 0.1 MPa, although it was higher than binderless particleboard pressed at the same pressing temperature (0.06 MPa). Lamaming *et al.* (2013) also showed that the addition of sucrose could improve the IB of oil palm particleboard. However, when the pressing temperature increased to 200 °C, the IB of sucrose bonded particleboard improved by almost two times. This experimental results showed that sucrose-bonded particleboard could provide good performance board properties when it was pressed at 200 °C. Zhao and Umemura (2014) indicated that sucrose could yield 5-hydroxymethyl furfural under heat treatment.

FTIR Analysis

Figure 5a shows an FTIR analysis of binderless board and citric acid bonded particleboard at various pressing temperatures. A carbonyl group (C=O) is indicated at approximately1720 cm^{-1} for the raw materials and binderless particleboard. According to Widyorini *et al*. (2005), these groups resulted from the degradation of hemicellulose during pressing. The intensity increased as the amount of citric acid and pressing temperature increased. A similar trend was found for bamboo particleboard bonded using citric acid at concentrations ranging from 10% to 40% (Widyorini *et al*. 2014). The carbonyl group was present because of the reaction of the hydroxyl groups from the lignocellulosic materials and carboxyl groups from citric acid (Umemura *et al*. 2011). Compared to citric acid, Fig. 5b shows that the addition of sucrose did not provide a strong intensity of carbonyl groups. However, increasing the pressing temperature increased the intensity of the carbonyl groups. The ester groups caused the particleboard to exhibit hydrophobic tendencies, as indicated in Fig. 1.

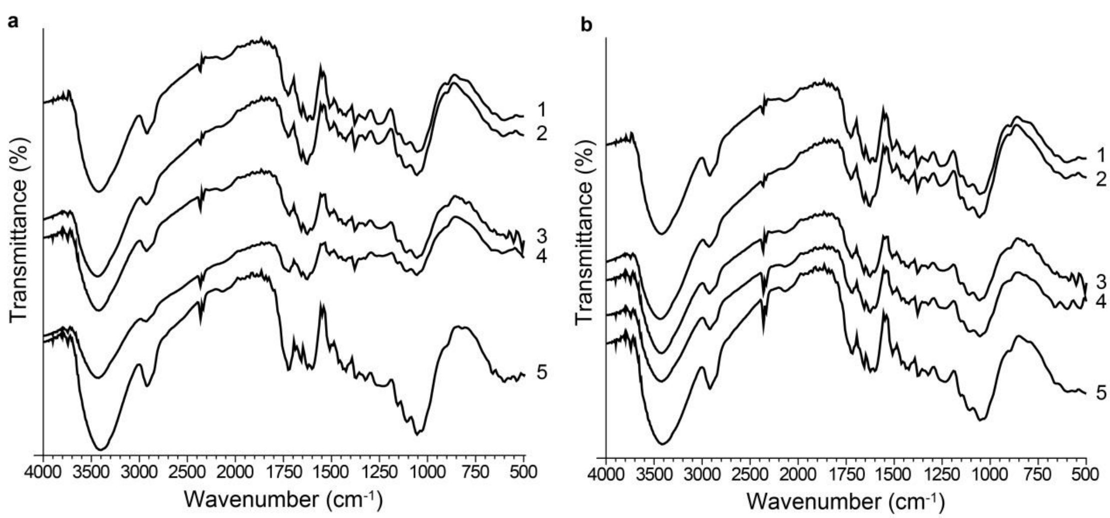

Fig. 5. FTIR analysis of teak wood particle and its particleboard for a) citric acid and b) sucrose adhesives: 1. teak wood particle; 2. binderless particleboard 180 °C; 3. binderless particleboard 200 °C; 4. 10% adhesive and 180 °C; 5. 10% adhesive and 200°C

Figure 6 shows the FTIR analysis of citric acid-sucrose bonded particleboard at 200 °C pressing temperature. The figure shows that an addition of 50% sucrose in the adhesive mixture provided a higher intensity of carbonyl groups than the addition of 100% sucrose. Umemura *et al.* (2013) predicted that sucrose was an important component in the increase in hydroxyl groups, which can react with the carboxyl groups from citric acid. Based on this research, it was assumed that the addition of 50% sucrose in adhesive mixture decreased the carboxyl group amount because the citric acid was also decreased. Therefore, the ester groups did not noticeably increase.

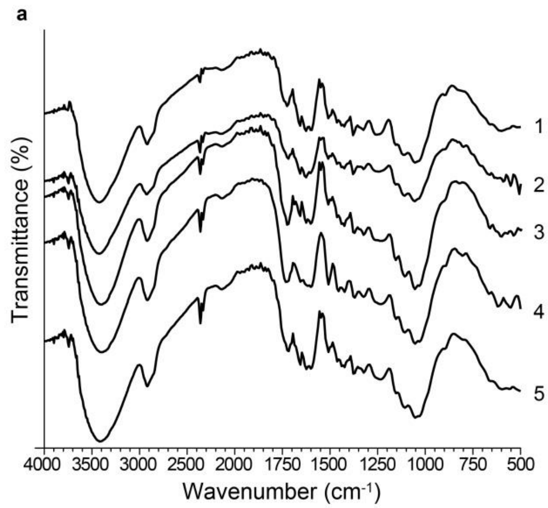

Fig. 6. FTIR analysis of teak wood particle and its particleboard at a pressing temperature of 200 °C:1. teak wood particle; 2. Binderless particleboard; 3. 100% citric acid; 4. 50/50 ratio of citric acid/sucrose; 5. 100% sucrose

The groups that were present at 1512 cm^{-1} were assumed to come from the aromatic unit of lignin. According to Okuda *et al*. (2006), lignin was recorded at C═C groups at approximately 1505 to 1510 cm^{-1}. However, Figs. 5 and 6 show that the addition of citric acid or sucrose did not greatly affect the intensity of the C=C groups. The addition of the citric acid/sucrose mixture (50/50) provided higher intensity C=C groups compared to other compositions. It is thought that lignin also contributed to the formation of the bonding mechanisms. This phenomenon should be further investigated in future work, considering that the role of lignin in the bonding mechanism of the natural adhesive with citric acid and sucrose is still unknown.

CONCLUSIONS

1. Citric acid-bonded particleboard exhibited the highest physical and mechanical properties. Sucrose-bonded particleboard, which was pressed at 180 °C, had the lowest properties. The adhesive composition did not significantly influence the properties of the teak particleboard.
2. Increasing the pressing temperature greatly improved the dimensional stability of the boards. Sucrose did not act as a good adhesive when pressed at 180 °C; however, its bonding properties could be improved when it was mixed with citric acid.
3. In this research, optimal conditions were achieved at 200 °C of pressing temperature with a citric acid/sucrose ratio of 100/0. The properties of the particleboard at the condition were as follows: the thickness swelling, water absorption, modulus of rupture, modulus of elasticity, ratio of wet/dry MoR, ratio of wet/dry MoE,and internal bond strength were 3%, 18%, 12.3 MPa, 3.8 GPa, 56%, 51%, and 0.37 MPa, respectively.
4. FTIR analysis showed that the ester groups increased with increasing pressing temperature and citric acid amount. It was supposed that an addition of 50% sucrose in

the adhesive mixture decreased the carboxyl group amount because the citric acid also decreased. Therefore, the ester groups did not noticeably increase.

5. Based on the FTIR analysis, it was supposed that lignin also contributed in the formation of bonding mechanism. More detailed clarification needs to be investigated in future papers.

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