Effect of Starch Addition on Properties of Citric Acid-bonded Particleboard Made from Bamboo

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Citric acid has been investigated as a good adhesive for particleboard. This research studied the effect of starch addition on the properties of citric acid-bonded particleboard. Starch provides hydroxyl groups that can react with the carboxyl group in citric acid. Three kinds of starches were used in this research, *i.e.* corn, ganyong (*Canna edulis* Ker-Gawl), and garut (*Maranta arundinacea* L.) starches. Petung (*Dendrocalamus* sp.) bamboo particles were used as raw material. The mixture ratios of citric acid/starch were set at 100/0, 87.5/12.5, and 75/25 (w/w), while the resin content was set at 30 wt.% based on air-dried particles. The boards were then manufactured under pressing conditions of 180 °C for 10 min. Based on the physical and mechanical properties of the particleboards, it was concluded that the addition of starch tended to enhance the mechanical properties, while decreasing the physical properties of the boards. An addition of 12.5 wt.% starch in citric acid was the optimum resin ratio for manufacturing bamboo particleboard. Maranta and canna starches provided higher mechanical properties compared to corn starch. A FTIR analysis clearly showed that the intensity of carbonyl groups increased with increasing of starch content, which indicated that crosslinking between starch and citric acid occurred.

Keywords: Citric acid; Starch; Bamboo; Starch types; Adhesive composition; Bonding mechanism; Particleboard

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INTRODUCTION

Composite products typically use a formaldehyde-based adhesive, such as urea formaldehyde or phenol formaldehyde. However, it is well known that those adhesives can release formaldehyde emissions that are carcinogenic to humans. Therefore, the development of eco-friendly biocomposites has become an attractive option. Research has focused on finding new adhesives based on renewable materials, such as corn starch-tannin (Moubarik *et al*. 2010), glucose and sucrose (Tondi *et al*. 2012; Lamaming *et al.* 2013), citric acid (Umemura *et al*. 2011, 2012; Widyorini *et al*. 2016a), citric acid and sucrose (Widyorini *et al*. 2016b), as well as tannin and sucrose (Zhao and Umemura 2014).

Citric acid has been known as a good bonding agent for wood composites (Umemura *et al*. 2012; Widyorini *et al*. 2016b) and non-wood composites (Kusumah *et al*. 2016; Widyorini *et al.* 2016a). This is due to the ester linkages between carboxyl groups from citric acid, and hydroxyl groups from wood or non-wood, which support good adhesion between both materials, as pointed out by Umemura *et al*. (2012) and Widyorini *et al*. (2016a). In addition, citric acid has been researched as a crosslinking agent for plant fiber (Ghosh *et al*. 1995), paper (Yang *et al*. 1996), starch (Yu *et al*. 2005), and solid wood (Vukusic *et al*. 2006; Hasan *et al*. 2007), as well as an absorber for heavy metal ions (Thanh and Nhung 2009).

Reddy and Yang (2010) pointed out that crosslinking starch films could use citric acid to improve their strength and stability. The citric acid acted both as a crosslinking agent and a plasticizer in the starch films (Ghanbarzadeh *et al*. 2011). It has also been reported by Yu *et al*. (2005) that citric acid can form strong hydrogen bond interactions with starch and improve its thermal and water stability.

Starch is the major carbohydrate reserve in plants. It is a biodegradable polysaccharide, produced in abundance at low cost, and exhibits thermoplastic behavior (Moubarik *et al*. 2010). Currently, starch has been proposed as a co-agent for synthetic resins for particleboards, such as isocyanates and urea formaldehyde (Moubarik *et al*. 2010). Recent studies on corn starch-tannin adhesives for plywood applications have demonstrated that high-performance natural wood-to-wood gluing is possible (Moubarik *et al*. 2013). The main sources of starch material in the food industry are cassava, maize, potato, wheat, and sweet potato. Because these crops function as food staple sources, recently many studies have explored the potential utilization of other food crops, which would then avoid world food security problems. Therefore, in this research, three types of starch are used in combination with citric acid: corn starch, and starches from tubers of ganyong (*Canna edulis* Ker-Gawl), and garut (*Maranta arundinacea* L.). Corn starch has already been well applied in the field; however, maranta starch and canna starch have not yet been exploited. The three starches used in this study have different compositions of amylopectin and amylose; therefore, their bonding mechanisms with citric acid require investigation. This paper was designed to investigate the bonding properties of petung bamboo particleboard that bonded using citric acid and starch with various ratio compositions and starch types.

EXPERIMENTAL

Materials

Petung (*Dendrocalamus asper* (Schult.) Backer) bamboo particles were collected from the Yogyakarta province area (Indonesia). All of the bamboo particles were screened, and the particles that passed through a 10-mesh were used as test materials. The particles were then air-dried to a moisture content of around 12%.

Preparation of adhesive solution

Citric acid (anhydrous), made from Weifang Ensign Industry Co. Ltd. (Shandong, China), was used without further purification. Three kinds of starches were used in this research, *i.e*. corn starch (Handayani, Surabaya, Indonesia), maranta, and canna starches (Warung Panganku 3B, Jakarta Timur, Indonesia). The composition of the amylose and amylopectin of three starches is shown in Table 1.

Table 1. Composition of Amylose and Amylopectin

Starch	Amylose (%)	Amylopectin (%)
Corn starch	26.66	54.73
Maranta starch	27.91	54.04
Canna starch	22.69	58.98

Citric acid and starch were dissolved in hot water (70 °C ± 2 °C) with the concentration of the solution adjusted in the range 59 wt.% to 60 wt.%. The mixture ratios of citric acid/starch were set at 100/0, 87.5/12.5, and 75/25 (w/w). The viscosity and pH of the solutions are shown in Table 2.

Table 2. Viscosity and pH of Citric Acid and Starch Solution

Mixture Ratio of Citric Acid:Starch	Starch Types	Concentration (%)	Viscosity at 25 °C (mPa.s)	pH
100:0	-		15.8	0.85
87.5:12.5	Corn starch		11.44*	1.19
75:25	Corn starch		13.23*	1.53
87.5:12.5	Maranta starch		12.75*	1.48
75:25	Maranta starch		13.86*	1.54
87.5:12.5	Canna starch		12.66*	1.26
75:25	Canna starch		13.74*	1.33

*Viscosity at (45 °C ± 2 °C)

Manufacturing of particleboard

The warm mixed solution (45 °C ± 2 °C) was then used as an adhesive and mixed onto bamboo particles at 30 wt.% resin content based on the weight of the dried particles. Citric acid solution was sprayed onto the particles at room temperature. The sprayed particles were then oven-dried overnight at 80 °C to reduce the moisture content to around 4% to 6%.

The particles were then hand-formed into a mat using a forming box of 250 mm × 250 mm. The mat was hot-pressed (Riken Seiki Co., Ltd., Kyoto, Japan) at 180 °C for 10 min under a pressure of 3 MPa. A 7-mm thick steel bar was used to control the board thickness during the hot-pressing process, while the target board density was set at 0.9 g/cm^3. Three replications were applied in this study. All of the boards were conditioned at ambient conditions for approximately 10 days, reaching a moisture content of 5% to 7%.

Methods

Evaluation of board properties

After conditioning, the properties of the particleboards were evaluated according to the Japanese Industrial Standard A 5908 (2003) for Particleboard. Tests were conducted for the physical and mechnical properties, *i.e.* thickness swelling (TS), water absorption (WA), surface roughness (R_a), modulus of rupture (MOR), modulus of elasticity (MOE), internal bond (IB) strength, and the screw holding strength.

The TS and WA values of each board after water immersion at 20 °C for 24 h were measured for specimens of a 50 mm × 50 mm × 7 mm specimen of each board. The static three point bending test in dry condition was conducted on a 200 mm × 50 mm × 7 mm specimen from each board. The effective span and loading speed were 150 mm and 10 mm/min, respectively. Before the bending strength test, the specimens were tested for their surface roughness using a surface roughness SRG 4000 (Bosworth Instrument, Cleveland, OH, USA). Six measurements were randomly taken from both surfaces of each sample, where the average roughness (R_a) was used to evaluate the roughness characteritics of the particleboards. The internal bonding (IB) strength was investigated using the same size as those used for the TS test. The specimen of 100 mm × 50 mm × 7 mm was used for the screw holding strength test. Two positions in the tested pieces were vertically screwed, and the screws were then pulled out with a speed of 2 mm/min. The MOR, MOE, IB, and screw holding strength of the boards were values corrected for each target density based on specimen densities.

Fourier transform infrared (FTIR) spectroscopy

To remove the excess and unreacted citric acid-starch, the specimen tests of 50 mm × 50 mm × 7 mm were immersed in boiling water for 2 h and in water at 20 °C for 1 h, respectively. The materials were then dried at 40 °C overnight and then powdered using a mill machine. A FTIR analysis of those samples was performed with a FTIR spectrophotometer (Shimadzu IRPrestige-21, Kyoto, Japan) using the KBr disk method and were recorded by means of an average of 10 scans at a resolution of 16 cm^{-1}.

RESULTS AND DISCUSSION

Physical and Mechanical Properties

The board densities of particleboards ranged from 0.89 g/cm^3 to 0.96 g/cm^3, and their moisture contents were in the range of 6.3% to 8.0%. The citric acid bonded particleboards took on a dark color. There was no remarkable difference in color between the different kind of starches and composition ratio of citric acid/starch. All bamboo particleboards were manufactured without any delamination. However, in the preliminary study, it was confirmed that starch-bonded particleboard has very low properties, or even becomes delaminated. It was found that starch is more reactive toward the citric acid than bamboo. Based on Reddy and Yang (2010), the possibility of cross-linking starch films using citric acid to improve their strength and stability was examined.

Figure 1 shows the relation between starch types and composition ratios of citric acid/starch on the TS and WA of particleboards.

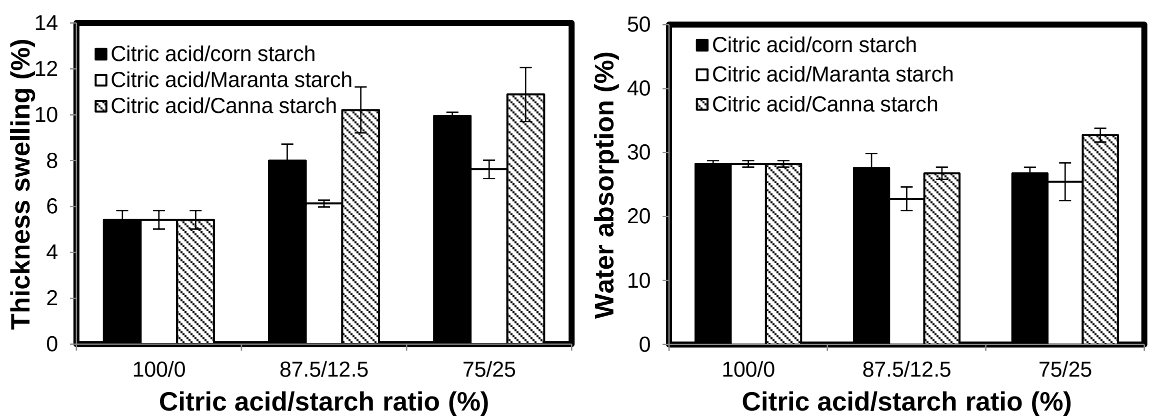

Fig. 1. Effect of ratio composition of citric acid/starch and starch types on the thickness swelling and water absorption values; Vertical bars indicate the standard deviation

It is apparent from Fig. 1 that all of the particleboards met the requirements of JIS A 5908 (2003), *i.e.* a ratio of less than 12%, which indicated that citric acid/starch-bonded bamboo particleboards had good dimensional stability. The average TS values of 100% citric acid-bonded particleboard were 5.4%. However, it increased to 10%, 8%, and 11%, when the composition ratio of citric acid/starch was 75/25 for corn starch, maranta starch, and canna starch, respectively. Increased starch content in the composition resulted in an increase in TS values, because the starches were more soluble in the water. However, all of the boards met the requirement of the JIS A 5908 (2003). It was possible that starch cross-linked citric acid, as mentioned by Reddy and Yang (2010), and produced good dimensional stability of the boards.

There were no remarkable differences in water absorption values of particleboards bonded using citric acid and citric acid/starch with the different starch types. The water absorption values for particleboard with the composition ratio of citric acid/starch (75/25) were 27%, 25%, and 33%, for corn starch, maranta starch, and canna starch, respectively. Widyorini *et al*. (2016a) stated that citric acid content is the more dominant factor in increasing dimensional stability. It was mentioned that carboxyl groups from citric acid were ester-linked with hydroxyl groups from wood or non-wood, therefore reducing the hygroscopicity of wood and the tendency to swell or shrink (Rowell 1991; Vukusic *et al.* 2006; Umemura *et al*. 2012; Widyorini *et al*. 2016a). Canna starch was lowest in amylose compared to other starch types, where the particleboards made from citric acid/canna starch had the lowest dimensional stability. Amylose, which consists of linear chains, is an important characteristics for the formation of strong linkages that responsible for its water resistance.

Figure 2 shows the surface roughness (R_a) value of bamboo particleboards at various composition ratios and starch types. Nemli *et al*. (2005) pointed out that the surface quality of the particleboard is a function of raw material type, pressure, board density, as well as shelling ratio. The average R_a values for all particleboards ranged from 5.8 µm to 6.9 µm. Hiziroglu *et al*. (2004) stated that the average R_a values ranged from 3.67 µm to 5.46 µm for commercially manufactured particleboard. Compared to those studies, the R_a values of bamboo particleboard were slightly higher. The citric acid-bonded particleboard provided the lowest R_a or the smoothest surface. The addition of starch in the adhesive mixture resulted in an increase in the R_a value. However, the starch types had relatively the same R_a value.

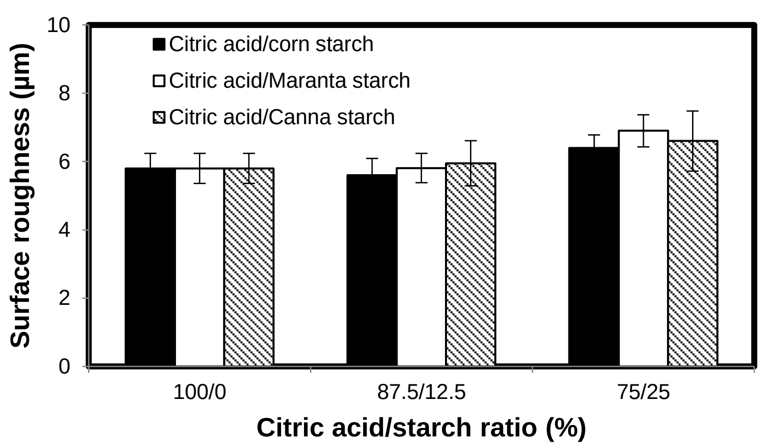

Fig. 2. Effect of ratio composition of citric acid/starch and starch types on surface roughness of particleboards; Vertical bars indicate the standard deviation.

Figure 3 shows the average MOR and MOE values of bamboo particleboards at various resin composition ratios and starch types. It is clearly shown that the starch types and composition ratios of citric acid/starch had a remarkable effect on the mechanical properties of the boards. All bamboo particleboards met the requirement of the 13 types of JIS A 5908 (2003), where a MOR of 13 MPa or more is required. The MOR value of the citric acid-bonded particleboard was 13.8 MPa, while citric acid/starch-bonded boards with the composition of 75/25 were 16, 21, and 16 MPa for corn starch, maranta starch, and canna starch, respectively. This clearly showed that starch types and composition ratios affected the modulus of rupture. The addition of starch until a ratio of 25%, in the adhesive composition, increased the MOR value. A considerable amount of hydroxyl groups in starch is supposed to be reacted with carboxyl groups from citric acid, as mentioned by Reddy and Yang (2010). The highest MOR was achieved by the citric acid/maranta starch at the 75/25 composition ratio, *i.e.* 21 MPa. According to the standard of the 18 types of JIS A 5908 (2003), a MOR of 18.0 MPa or more is required. Therefore, it was clearly clarified that the bending strength of the board bonded using a ratio of citric acid/maranta starch at the 75/25 composition ratio was comparable to the standard.

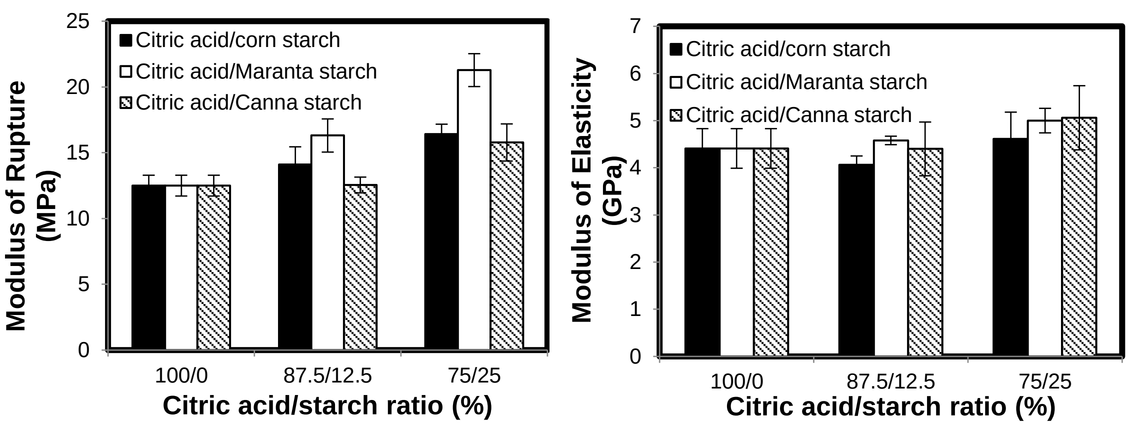

Fig. 3. Effect of ratio composition of citric acid/starch and starch types on modulus of elasticity of particleboards; Vertical bars indicate the standard deviation.

Figure 3 also shows that the values of MOE of citric acid-starches bonded particleboards were much higher than the requirement (3 GPa) for the 18 types of JIS A 5908 (003). The MOE of these particleboards varied between 4.1 GPa to 5.1 GPa. Starch types and composition ratios did not affect the MOE values of the boards. However, a slight increase of MOE was shown to increase the amount of starch in adhesive compositions.

Figure 4 shows the IB strength of the particleboard in various starch types and composition ratios. The average IB strength of citric acid-bonded particleboards was 0.86 MPa, while for citric acid/starch particleboards the strength ranged from 0.96 MPa to 1.36 MPa. All of the IB strengths of the boards bonded in various starch types and composition ratios were much higher than the requirement (0.3 MPa) for the 18 type of JIS A 5908 (2003). The addition of 12.5% starch in the composition yielded a remarkable effect in increasing the IB strength, especially for maranta and canna starches. However, the addition of 25% starch reduced the IB value of the boards, though the value was still higher compared to the citric acid-bonded particleboard. Maranta and corn starches have higher amylose content compared to canna starch. It was mentioned before that amylose was responsible for the strength of the film. However, this research showed that starch types did not affect on the internal bond strength. Judging from the results, it was clarified that the mechanical properties of the boards bonded with citric acid were greatly affected by the resin composition ratios and the optimum ratio citric acid/starch was 87.5/12.5, when the starch types were maranta and canna starches.

The effect of the ratio composition of citric acid/starch and starch types on the screw holding strength of particleboards are shown in Fig. 5. The value of the screw holding strength of citric acid-bonded particleboard was 338 N, as required by the JIS A 5908 (2003) standard (minimum strength of 300 N required). It was interesting that the addition of starch reduced the screw holding strength of the boards, while it increased the internal bond of the the boards. The values of screw holding and IB strengths are properties of wood-based panels of considerable importance for their use in furniture and cabinet manufacturing.

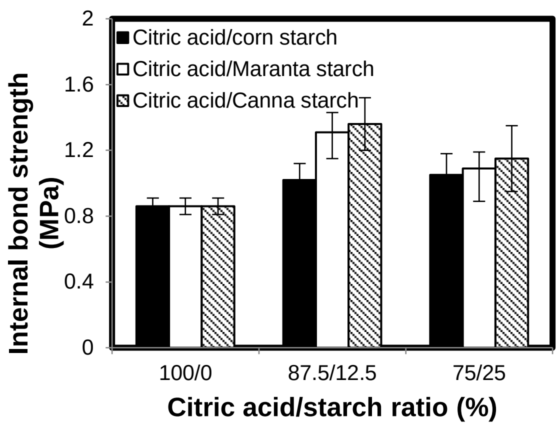

Fig. 4. Effect of ratio composition of citric acid/starch and starch types on internal bond strength of particleboards; Vertical bars indicate the standard deviation

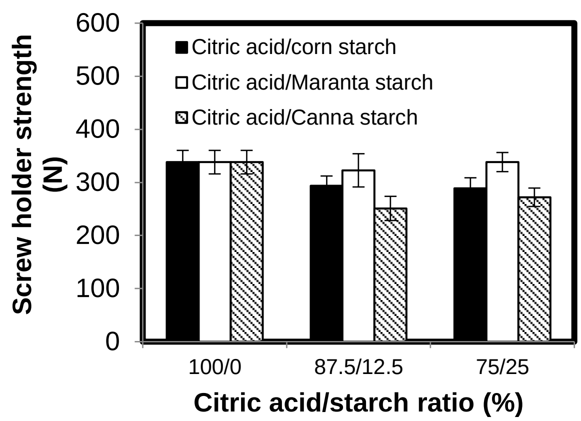

Fig. 5. Effect of ratio composition of citric acid/starch and starch types on screw holding strength of particleboards; Vertical bars indicate the standard deviation

FTIR Analysis

Figure 6 shows an FTIR analysis of raw materials (bamboo particles) and particleboards with different adhesive compositions. The stretching of carbonyl groups (C=O), as indicated at approximately 1720 cm^{-1}, could be derived from the carboxyl group and/or C=O ester groups. As mentioned by Umemura *et al.* (2011), the carbonyl groups were represented as ester linked between hydroxyl groups from the lignocellulosic materials and carboxyl groups from citric acid. Starch also contains a considerable amount of hydroxyl groups that can be expected to cross-link with citric acid (Reddy and Yang 2010). It was clearly shown that the intensity of carbonyl groups increased with increased corn starch content in the adhesive composition, which was consistent with the IB results (Fig. 4).

Figure 6 also shows the FTIR analysis of citric acid starch-bonded particleboard with the composition ratio of 75/25 at different starch types. It shows that the intensity of carbonyl groups of citric acid-corn starch were higher compared to citric acid-canna starch and citric acid-maranta–starch. However, based on the IB result, there was no difference in the IB result for the composition ratio of citric acid/starch (75/25) for all starch types. Based on Table 1, canna starch had the highest composition of amylopectin compared to other starch types. More bonding mechanisms should be investigated in future work, considering that citric acid-starch could be cross-linked to produce better properties in particleboards.

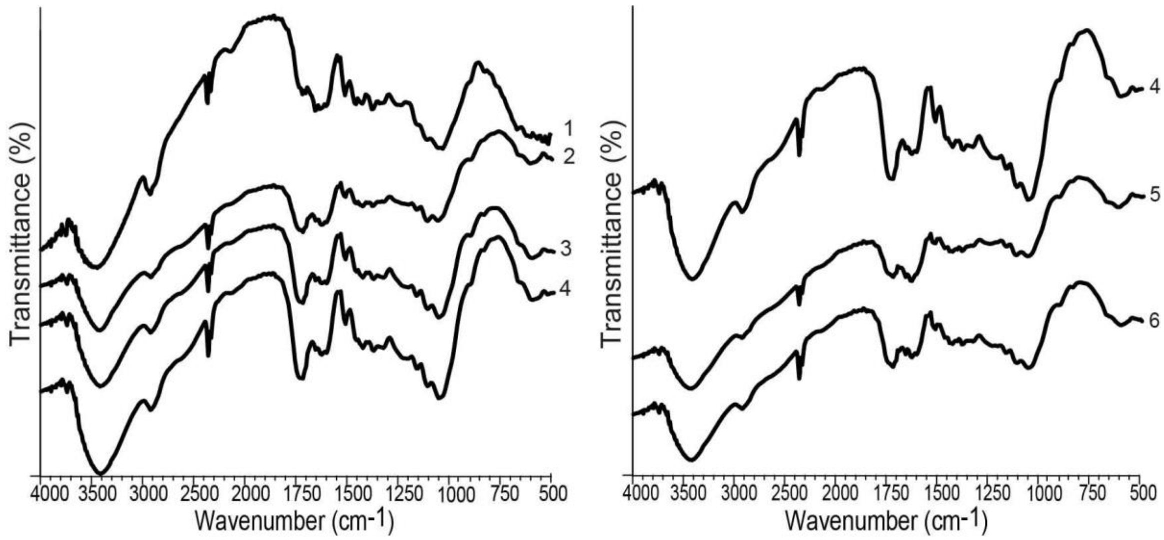

Fig. 6. FTIR analysis of bamboo particle and its particleboard: 1. Bamboo particle; 2. Citric Acid/Corn Starch (100/0) based particleboard; 3. Citric Acid/Corn Starch (87.5/12.5) based particleboard; 4. Citric Acid/Corn Starch (75/25) based particleboard; 5. Citric Acid/Maranta Starch (75/25) based particleboard; 6. Citric Acid/Canna Starch (75/25) based particleboard

CONCLUSIONS

1. All of the boards had physical and mechanical properties that satisfied the requirement of the JIS A 5908 (2003) standard. The citric acid/starch-bonded particleboards had higher mechanical properties compared to the citric acid only bonded particleboard. This was because the hydroxyl groups in starch could be cross-linked with citric acid, thus providing good bonding properties.
2. Maranta and canna starches in resin composition provided higher mechanical properties compared to corn starch. The addition of 12.5 wt.% maranta starch in the composition was an optimum condition for manufacturing bamboo particleboard. The properties of the particleboard at the condition were as follows: the thickness swelling, water absorption, surface roughness, modulus of rupture, modulus of elasticity, internal bonding strength, and screw holding test were 5.9%, 23%, 5.4µm, 16.9 MPa, 4.6 GPa, 1.21 MPa, and 323 N, respectively.
3. The FTIR analysis showed that the intensity of carbonyl groups increased with increased corn starch content, which indicated the cross-link between starch and citric acid occurred, as well as it consistent with the IB results. This research showed that starch types did not give a remarkable difference on the internal bond strength.

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