

# Effect of Citric Acid Content and Extractives Treatment on the Manufacturing Process and Properties of Citric Acid-bonded *Salacca* Frond Particleboard

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This study focused on the effect of an extractive treatment and the application of citric acid on the properties of particleboard made from *Salacca* frond. In general, extractives have a negative effect on the bondability of synthetic resin. However, the effect of extractives on the bonding mechanism of citric acid as the biobased adhesive is unclear. Unextracted and extracted *Salacca* frond were used as the raw materials. A hot water extractive treatment was conducted by boiling the particles for 2 h. The boards were manufactured under the following conditions: citric acid content of 0%; 10%; 20% weight percent (wt%), pressing temperature of 180 °C, and pressing time of 10 min. The target density was set at 0.8 g cm<sup>-3</sup>. The results showed that the addition of citric acid resulted in an increase in the physical and mechanical properties of the particleboard. Interestingly, when a 20% citric acid content was applied, there were no significant differences in the physical or mechanical properties of the particleboards made from unextracted and extracted particles. Based on these results, it was concluded that when citric acid is used as adhesive, the hot-water extractive treatment of *Salacca* frond is not needed.

*Keywords:* *Salacca frond*; *Citric acid*; *Extractives treatment*; *Physical properties*; *Mechanical properties*

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## INTRODUCTION

The increasing demand and the different uses of forest resources have led to the shortage of wood supply. In addition, the need for wood-based materials will increase due to population increase and economic development. As a result, the wood based-industry has seriously considered agricultural residues as raw material for particleboard products.

*Salacca* sp. frond is one potential material that is not optimally utilized. *Salacca* is native to South Sumatra and Southwest Java, and it is widely cultivated in Thailand. Lim (2012) stated that it has also been introduced into New Guinea, the Philippines, Queensland, the northern territory of Australia, the Ponape Island (Caroline Archipelago), China, Surinam, Spain, and the Fiji Islands. *Salacca* frond has potential for new applications due to the large quantities of these fronds that are disposed of and not utilized. *Salacca* is usually harvested for the fruit, and the residues remaining from the frond are left over. But the frond has some characteristics, such as solidity and stiffness, that suggest it may be suitable as material for making board.

Darmanto *et al.* (2017) tried to fibrillize the *Salacca* frond into fibers and found that the fiber had a tensile strength of 160 MPa. The strength increased to 275 MPa,

220 MPa, and 225.75 MPa after alkali, alkali-steaming, and alkali-steam explosion treatments, respectively. Previous studies showed that *Salacca* frond particleboard bonded with citric acid-maltodextrin could be produced, and its properties met the JIS A 5908 standard (Widyorini *et al.* 2018).

Citric acid is a good binding agent in particleboard made from wood and/or non-wood materials due to the ester linkages between the hydroxyl groups of lignocellulosic materials and carboxyl groups of citric acid (Umemura *et al.* 2011; Kusumah *et al.* 2016; Widyorini *et al.* 2016). The hydroxyl groups are present in holocellulose. However, the effect of extractive on bonding mechanism of citric acid is still unclear.

Extractives usually can have adverse effects on the curing of synthetic resins, such as lowering the particle-particle bond strength and reducing the internal bond strength. Removing hot water extractives (by steaming at 160 °C), results in a decrease in thickness swelling values, but also exhibits lower internal bond strength values of *polymeric diphenylmethane diisocyanate (p-MDI)* bonded oriented strand board (Paredes *et al.* 2008), as well as that of *phenol formaldehyde (PF)* bonded hardwood and softwood particleboard (Pelaez-Samaniego *et al.* 2014). It seemed that the high temperature of the water treatment caused a reduction of hemicellulose content by hydrolysis, resulting in the reduction of thickness swelling values of the particleboard (Pelaez-Samaniego *et al.* 2013).

In addition, the mechanical properties of PF bonded particleboard made from hot water (at 100 °C) treated black spruce bark are not as good as the boards made from untreated particles (Yemele *et al.* 2008). This may be due to the decreased amount of PF resin absorbed by the treated bark, as well as the increase of bark particle porosity following the hot water treatment (Yemele *et al.* 2008). Water extractives usually contain starch and other polysaccharides. However, based on Umemura *et al.* (2013) and Widyorini *et al.* (2017), sucrose and starch can react with citric acid in certain ratios and affect the properties of the particleboard. Therefore, it is predicted that extractives will affect the bondability of citric acid bonded particleboard.

*Salacca* frond has extractives and contains hemicellulose, lignin, and cellulose (Widyorini *et al.* 2018). In this research, particleboard was manufactured using *Salacca* frond with citric acid as the adhesive. The effects of extractives on the bonding performance of citric acid bonded particleboard were investigated in this study.

## EXPERIMENTAL

### Preparations of Materials

The *Salacca* fronds were obtained from Yogyakarta province, Indonesia. After they were cleaned, the fronds were cut into samples with a length of 1 m. *Salacca* frond particles were prepared using a chipper and knife ring flaker. The particles that passed through the 10 mesh-screen were used as the raw material. Half of the *Salacca* frond particles were then extracted with boiling water over a 2 h period. All particles (hot water extracted and unextracted particles) were then air-dried at room temperature (26 to 29 °C) and a relative humidity of approximately 77% for about 7 to 10 days to a moisture content of around 12%.

Anhydrous citric acid (Weifang Ensign Industry Co. Ltd., Weifang, China) was used as adhesive. Citric acid was dissolved in water to obtain a concentration of 59 weight percent (wt%).

## Chemical Composition Analyses of Salacca Frond

Ethanol-toluene extractives, hot water extractives, holocellulose, cellulose, and lignin content were tested according to American Society for Testing and Materials (ASTM) standard methods, which were also used in Widyorini *et al.* (2018). The ash content was determined according to ASTM D1102-84 (2001).

The hot water extracted liquor was used to determine the sugar content using high-performance liquid chromatography (HPLC) (Shimadzu, Kyoto, Japan). Samples were prepared for HPLC analysis by taking a 1 mL sample and filtering it through a 0.45  $\mu\text{m}$  filter. The filtrates were analyzed under the following materials and conditions: a MetaCarb 87C column (Varian Inc., Palo Alto, CA, USA), distilled water eluent, flow rate of 0.5 mL  $\text{min}^{-1}$ , column temperature of 85  $^{\circ}\text{C}$ , with an refractive index detector (RID).

## Manufacture of Particleboards

The citric acid was sprayed onto the unextracted and extracted *Salacca* fronds at two levels of resin content (10 wt% and 20 wt%), where the amount of citric acid was based on the weight of the dried fibers. The sprayed particles were then oven-dried for 18 h at 80  $^{\circ}\text{C}$  to reduce the moisture content. The moisture content of the mat was around 3 to 5%.

The particles were hand-formed into a mat by using a forming box, and then they were hot-pressed into the particleboard. The target dimensions of the board were 25 cm x 25 cm, with the target board density set at 0.8 g  $\text{cm}^{-3}$ . The thickness of the boards was controlled by a 1 cm thick metal bar during the hot-pressing process. The pressing time was 10 min, while the hot pressing temperature was 180  $^{\circ}\text{C}$ . Particleboards without any adhesive (0 wt%) were also produced using the same conditions. To obtain the binderlessboard, dried *Salacca* frond particles were directly hand-formed into a mat, according to Widyorini *et al.* (2005).

Three replications of each manufacturing condition were performed in this study. Prior to the evaluation of the mechanical and physical properties, all board samples were conditioned at room temperature (26 to 29  $^{\circ}\text{C}$ ) and a relative humidity of approximately 77% for about 7 to 10 days.

## Evaluation of Board Properties

The boards were evaluated according to the Japanese Industrial Standard for Particleboards (JIS A 5908 2003). The physical and mechanical properties tested were thickness swelling (TS), water absorption (WA), modulus of rupture (MOR), modulus of elasticity (MOE), internal bond strength (IB), and screw holding strength. The TS and WA tests were performed on a 5 cm x 5 cm x 1 cm specimen from each board after they underwent water immersion for 24 h at room temperature. The weight and thickness of the specimens were recorded for each specimen before and after immersion. The same size of the specimens of those used for the internal bond strength (IB) test. The specimen size for the screw holding power test of the boards was 10 cm x 5 cm x 1 cm. The pulling out load speed was approximately 0.2 cm  $\text{min}^{-1}$ . The bending properties of the boards were evaluated by conducting a static three-point bending test on a 20 cm x 5 cm x 1 cm specimen for each board in dry conditions. The support span for bending was 15 cm, while the speed of testing was 1 cm/min. The MOR, MOE, IB, and screw holding strength of the boards were corrected for each target density based on the regression line between the actual values of the mechanical properties and the specimen densities. Data of each test were statistically analyzed by a two-way analysis of variance, followed by a Tukey Honestly Significant Difference (HSD) test. Each experiment was performed in triplicate. The standard deviations were calculated from the data and were shown as error bars in each corresponding figure.

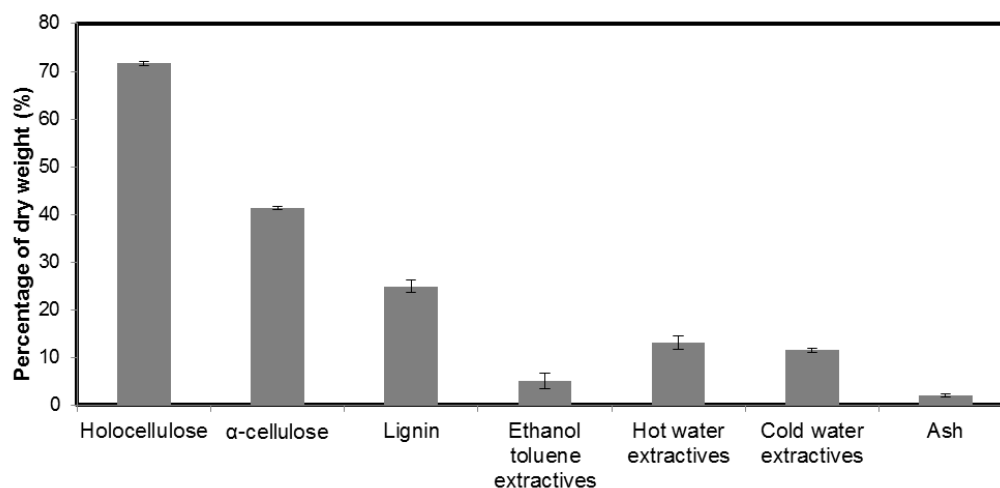
## Fourier Transform Infrared (FTIR) spectroscopy

The samples for FTIR analysis were the *Salacca* frond powder and its boards. The samples (*Salacca* frond and its boards) were boiled for 2 h and immersed in the water at room temperature for 1 h to remove unreacted citric acid. The samples were then dried at 80 °C for 12 h and ground into a powder. All infrared spectra were obtained with a FTIR spectrophotometer (FTIR-4200, JASCO, Tokyo, Japan) using the KBr disk method. The results were recorded by means of an average of 10 scans at a resolution of 16 cm<sup>-1</sup>.

## RESULTS AND DISCUSSION

Figure 1 shows the chemical composition of *Salacca* frond particles. The ash content of *Salacca* frond was 2.03%, which was lower than that of black spruce and trembling aspen barks (Yemele *et al.* 2008). The ethanol-toluene extractives of *Salacca* frond (5.15%) was slightly higher than that of oil palm frond (3.5%) (Hashim *et al.* 2011). The hot water extractives content of *Salacca* frond was 13.13 wt%, which was higher than black spruce bark and lower than trembling aspen bark (Yemele *et al.* 2008).

Removing hot water extractives lowers the physical and mechanical properties of the PF bonded boards (Yemele *et al.* 2008). Essentially, hot water extracts tannins, gums, sugars, and starches. Based on Table 1, the main component of the extractives was glucose, which may be derived from sugars or starches. The hydroxyl groups from glucose are predicted to react with carboxyl groups from citric acid to make ester linkages, which results in stronger bonding within the boards.



**Fig. 1.** Chemical composition of *Salacca* frond. Vertical lines through the bars represent the standard deviation.

**Table 1.** Neutral Sugar Composition of *Salacca* Frond Hot Water Extractives

Sugar Component	Composition (µg mL <sup>-1</sup> )
Glucose	45.0
Arabinose	35.0
Xylose + Lactose	18.9
Galactose + Fructose	22.0

Table 2 shows the TS and WA values of *Salacca* frond particleboard in various citric acid contents. The dimensional stability of the particleboard improved with the addition of citric acid. The TS values of the binderless board (0 wt%) were 117% and 222% for non-extraction and extraction treatment, respectively. These values were decreased to 18% and 20% by the addition of 20 wt% citric acid, respectively. After the extraction treatment, the TS values of the board increased. The hot water treatment released some extractives (Fig. 1) and increased the porosity, which was also mentioned by Yemele *et al.* (2008). The hot water extractives of *Salacca* frond was relatively high (around 13.13%). Pelaez-Samaniego *et al.* (2014) found a different trend than in this study, where the hot water extracted particleboard had a lower TS value compared to the unextracted particleboard. This may be due to the extractive treatment of the boards being done at a higher temperature (steaming at 160 °C) than this study. The high temperature not only removed the extractives, but it also caused the hemicellulose to depolymerize into monomers by hydrolysis (Widyorini *et al.* 2005), resulting in the reduction of thickness swelling values of the particleboard (Pelaez-Samaniego *et al.* 2013).

**Table 2.** Physical Properties of *Salacca* Frond Particleboard

Properties (%)	0 wt% Citric Acid		10 wt% Citric Acid		20 wt% Citric Acid	
	Unextracted	Extracted	Unextracted	Extracted	Unextracted	Extracted
Thickness Swelling (%)	117 ± 19b	222 ± 6c	23 ± 0.1a	37 ± 5a	18 ± 4a	20 ± 1a
Water Absorption (%)	153 ± 18b	328 ± 10c	62 ± 1a	77 ± 1a	58 ± 2a	58 ± 2a

Different letters within rows indicate significant differences between treatments at  $P < 0.01$ .

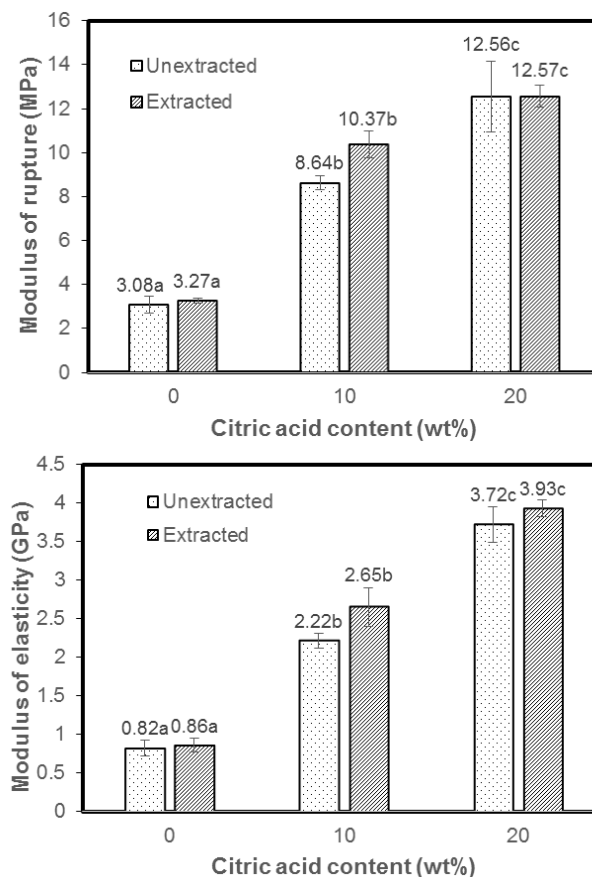
Interestingly, the differences in the TS values between the unextracted and extracted boards in this study were decreased with the addition of citric acid. This indicated that the addition of citric acid was a dominant factor in decreasing the TS value. Citric acid reacted and formed to make ester linkages with lignocellulosic materials (Umamura *et al.* 2011), resulting in the lower TS values.

The same trend observed for the TS was also seen for the WA values, as shown in Table 2. Binderless particleboard made from *Salacca* frond had a very low dimensional stability. The WA values of binderless particleboards were 153% and 328% for non-extraction and extraction treatment, respectively, whereas it decreased significantly by the addition of citric acid. This study showed that the extractives treatment had a negative effect on the WA values of binderless board, indicating that the extractives help to develop self-bonding during the manufacturing of the boards (Widyorini *et al.* 2005). It also showed that the addition of citric acid effectively decreased the WA value during the water immersion treatment. Rowell (1991) and Vukusic *et al.* (2006) mentioned that cross-linking chemicals reacting with hydroxyl groups reduced the hygroscopicity of wood and the tendency to swell or shrink.

Interestingly, when the citric acid content increased to 20 wt%, the extractives treatment did not affect the TS and WA values, indicating that citric acid played an important role in increasing the dimensional stability. The decrease in swelling observed in the particleboards using citric acid as an adhesive was strongly related to the effect of the increase in the amount of cross-linking between hydroxyl groups of lignocellulosic materials and carboxyl groups of citric acid. This study showed that extractives did not significantly affect the dimensional stability of the citric acid bonded boards.

Figure 2 shows that the MOR values of binderless particleboard (0 wt%) made from unextracted and treated particles were relatively the same, *i.e.*, 3.08 MPa and 3.27 MPa, respectively, indicating that the *Salacca* frond particles developed a low bending strength under this condition. These values were relatively the same as bamboo binderless particleboards (Widyorini *et al.* 2016). However, the value drastically increased by approximately 4 times the original amount when the citric acid content was increased to 20 wt%. All citric acid bonded *Salacca* frond (untreated and treated) particleboards met the JIS A 5908 type-8, where an MOR of 8.0 MPa or more is required. Interestingly, extractive treatments did not affect the MOR values of binderless and citric acid bonded particleboard made from *Salacca* frond.

The trend seen for the MOR values was also seen in the MOE values of the particleboards, as shown in Fig. 2. The MOE value of binderless particleboard (0 wt%) made from unextracted and extracted particles were 0.82 and 0.86 GPa, respectively. After 20 wt% citric acid was applied, the MOE value increased by around 4.5 times for both particleboards. The same trend was also found by Lo and Liew (2011) when bark, sapwood, and heartwood were tested, and urea formaldehyde was used as the resin. The MOE values of citric acid-bonded particleboards in this study were more than 2 GPa, exceeding the JIS A 5908 type-8. There were no significant differences between the MOE values of the particleboards made from unextracted and extracted particles during the addition of citric acid, which is consistent with the trend seen in the MOR values.

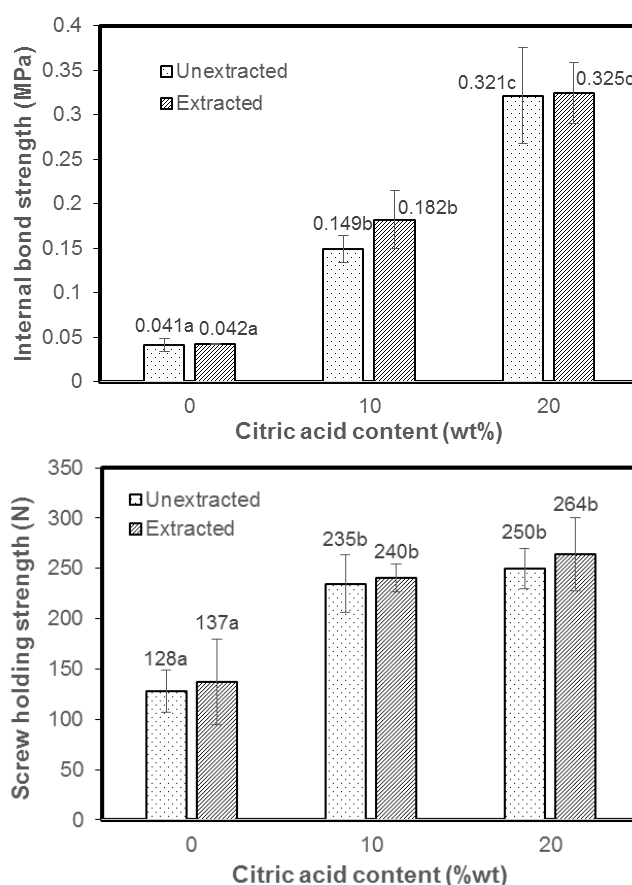


**Fig. 2.** Effect of extractives treatment and citric acid content on MOR and MOE of particleboards. Vertical lines through the bars represent the standard deviations. Different letters indicate significant differences between treatments at  $P < 0.01$ .

Figure 3 shows the increase in IB strength with increasing citric acid content. The IB of both particleboards made from untreated and treated particles binderless particleboards were low, *i.e.*, 0.041 and 0.042 MPa, respectively. This result showed that the extractives treatment did not affect the IB strength of the binderless

particleboard. In addition, the IB strength of the boards made from *Salacca* frond bonded with citric acid was greatly affected by the citric acid content, while it clearly showed that the extraction treatment did not significantly affect the IB strength of the citric acid bonded boards. The IB values of the extracted particleboards were slightly higher compared to the unextracted board. This indicated that some of the hydroxyl groups of sugar components in hot water extractives of *Salacca* frond may have contributed to the formation of ester linkages with carboxyl groups from citric acid. Yemele *et al.* (2008) observed a different result when a PF resin was used as the adhesive in bark particleboard. The difference in the effects of the hot water treatment in this study might be caused by the chemical component of the *Salacca* frond and the citric acid as the binding agent. The strength of the board bonded using citric acid (20 wt%) met the requirement (more than 0.3 MPa) of the JIS A 5908 type-18.

An improvement of the screw holding strength due to citric acid addition can be seen in Fig. 3. However, the extractives treatment did not significantly affect the screw holding strength or the IB strength. The averages of the screw holding strength of the binderless particleboard were initially 128 and 137 N for the citric acid-bonded particleboard (20 wt%) made from untreated and treated particles, respectively, and increased drastically to 250 and 264 N, respectively.

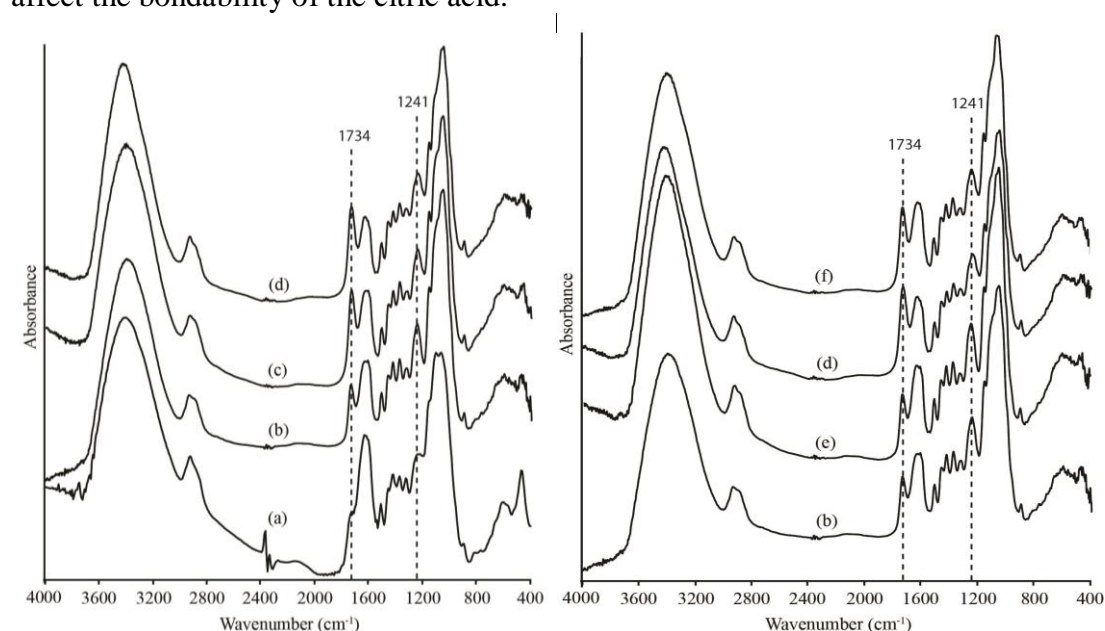


**Fig. 3.** Effect of extractives treatment and citric acid content on IB and screw holding strength of particleboards. Vertical lines through the bars represent the standard deviation. Different letters indicate significant differences between treatments at  $P < 0.01$ .

Figure 4 shows infrared spectra of *Salacca* frond and its corresponding particleboard. In Fig. 4, the peaks around  $1734\text{ cm}^{-1}$  and  $1241\text{ cm}^{-1}$  were hardly perceptible on the *Salacca* frond particles (a). However, the peaks were found on the *Salacca* frond binderlessboard (b and e), with the relatively same intensity, which was consistent with the mechanical properties of the particleboards. The peak at  $1734\text{ cm}^{-1}$  is typically ascribed to the C=O stretching due to the carbonyl groups and/or the C=O

ester groups (Yang *et al.* 1996), and the peak at  $1241\text{ cm}^{-1}$  was related to the C-O stretching vibration band of the ester groups (Aflori and Drobota 2015). The band around  $1740\text{ cm}^{-1}$  was assigned to hemicellulose (Kobayashi *et al.* 2009). It seemed that the peak at  $1734\text{ cm}^{-1}$  in binderlessboard (b and e) is derived from the degradation of hemicellulose during hot pressing, as mentioned by Widyorini *et al.* (2005).

The increase of the peak intensity at  $1734\text{ cm}^{-1}$  occurred by adding the citric acid, which is linear with the increasing mechanical properties of the board. This indicated the formation of ester linkages (Umemura *et al.* 2011), resulting from the reaction between the carboxyl groups of citric acid and the hydroxyl groups of *Salacca* frond. At the addition of 20 wt% of citric acid, the peak's intensity at  $1734\text{ cm}^{-1}$  of particleboard made from untreated (d) particles was higher compared to that of the particleboard made from the treated particles (f). As mentioned before, this indicated that some of the hydroxyl groups of sugar components in hot water extractives of *Salacca* frond reacted with carboxyl groups from citric acid. However, the mechanical properties of both particleboards were not remarkably different. In this study, the important finding was that hot water extractives in *Salacca* frond did not significantly affect the bondability of the citric acid.



**Fig. 4.** Fourier transform infrared (FTIR) spectra of (a) *Salacca* frond (b) non-extracted binderless particleboard (0 wt%), (c) non-extracted particleboard (10 wt%), (d) non-extracted particleboard (20 wt%), (e) extracted binderless particleboard (0 wt%), (f) extracted particleboard (20 wt%)

## CONCLUSIONS

1. The addition of citric acid resulted in an increase of dimensional stability and mechanical properties of the particleboard. Interestingly, when citric acid was applied, there were no significant difference in the physical and mechanical properties of the particleboards made from unextracted and extracted particles.
2. FTIR analyses demonstrated the appearance of peaks at  $1734\text{ cm}^{-1}$  and  $1241\text{ cm}^{-1}$  in binderlessboard and became stronger with the addition of citric acid on both untreated and treated particleboards. The peak's intensity at  $1734\text{ cm}^{-1}$  of the citric acid bonded particleboard made from untreated particles was higher compared to that of the board made from treated particles. However, the properties of both particleboards were not remarkably different.



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