

CHARACTERISATION OF *RHIZOPHORA* PARTICLEBOARD USING BIO-OIL-BASED PHENOL FORMALDEHYDE (PF) RESIN

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ABSTRACT

Phenol formaldehyde (PF) resin has been extensively used in various branches of industry as adhesive especially in the production of wood-based panels. However, due to the use of expensive and limited petroleum-based phenol in its formulation, there is a strong interest to explore renewable biomass material to partially substitute the phenol. In this work, slow pyrolysis was used to convert oil palm frond into bio-oil. From there, the phenol-rich fraction of the bio-oil was separated and added into the formulation of PF resin to produce an economical and environmental-friendly type of PF resin, known as bio-oil-phenol-formaldehyde (BPF) resin. *Rhizophora* particleboard was then fabricated with the BPF resin as adhesive. The particleboard was found to display excellent mechanical and physical properties with satisfactory formaldehyde emission. A morphological study of the particleboard also supported previous findings. The corresponding atomic number of the particleboard obtained from the morphological study was compared with those of water phantom and a fascinatingly favourable similarity was observed. This finding, hence, proposed a novel higher value-added application of the *Rhizophora* particleboard which has been largely researched as a potential phantom material in diagnostic radiography.

Keywords: oil palm frond, bio-oil, phenol formaldehyde resin, *Rhizophora* particleboard, phantom.

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INTRODUCTION

Since 1993, a favourable composition has been found between *Rhizophora* hardwood and water (Sudin, 1993, Tajuddin *et al.*, 1996; Banjade *et al.*, 2001). When water has been publicly recognised as a phantom material, the favourable composition raised a possibility for *Rhizophora* hardwood to be used as phantom as well. At first, untreated natural *Rhizophora* hardwood was employed and it was found that phantom-making from that type

of hardwood was quite unfavourable since it had poor endurance with time especially after being cut into the desired dimension (Shakhreet *et al.*, 2009). Therefore, the natural *Rhizophora* hardwood was improvised into a binderless *Rhizophora* particleboard. As this type of particleboard had no addition of adhesive that would help in binding mechanism, the particleboard was reported to have poor mechanical and physical properties hardly meeting the standard requirement of particleboard to be used in the healthcare industry (Marashdeh *et al.*, 2012).

The use of bio-adhesives such as Arabic gum and *Serishoom* (traditional animal-based adhesive) were then employed to fabricate the particleboard. Still, the mechanical and physical properties of those two did not provide satisfactory results

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(Abuarra *et al.*, 2014; Tousi *et al.*, 2015). Therefore, the incorporation of widely used synthetic adhesive such as phenol formaldehyde (PF) resin is considered. The selection of this type of resin is mainly due to its ability in providing good moisture resistance, exterior strength and durability as well as excellent temperature stability. However, the main drawback of conventional PF resin is that it can be very expensive due to the phenol price. Hence, the use of more natural and economical product such as bio-oil has been suggested. Bio-oil is a very suitable option because it is rich in phenols which are mainly found within the bio-oil in the form of pyrolytic lignin (Kim *et al.*, 2010).

Several attempts have been made to utilise bio-oil as phenol substitute in producing the bio-oil-phenol-formaldehyde (BPF) resin. These attempts include the incorporation of bio-oil obtained from the fast pyrolysis of pine wood (Sukhbaatar *et al.*, 2009), direct liquefaction of white pine sawdust (Cheng *et al.*, 2011) as well as the fast pyrolysis of white spruce and trembling aspen (Chaouch *et al.*, 2014).

Therefore, instead of using the conventional PF resin that incorporated the addition of petroleum-based phenol, BPF resin that incorporated the addition of natural phenol-rich bio-oil was introduced. This bio-oil was obtained from the slow pyrolysis of oil palm frond, chosen due to its abundant availability across the world. Oil palm frond was usually harvested annually at about 10.9 t ha⁻¹ from more than 13.5 million hectares of oil palm plantation around the world. Since oil palm frond had a very limited utility, an initiative was taken by this work to optimally exploit the enormous amount of oil palm frond (Kelly-Yong *et al.*, 2007). From there, the BPF resin was added during the fabrication of *Rhizophora* particleboard. The mechanical, physical and morphological properties of the particleboard were also determined.

EXPERIMENTAL

Materials

Oil palm frond was harvested in August 2014 in a plantation of Universiti Sains Malaysia (USM), Nibong Tebal, Pulau Pinang, Malaysia (5°08'48.2'N 100°29'32.0'E). Oil palm fronds that were left behind from the harvesting process were collected and the leaves attached to them were removed using machete. Immediately after retrieval, the oil palm fronds were dried in a Venticell oven at 105°C until their moisture content reduced to less than 10 mf wt% to avoid the growth of fungus or microorganism (Abdullah *et al.*, 2014). This moisture content was measured using A&D MX-

50 moisture analyser and the result was presented in moisture-free weight percentage (mf wt%). Then, a Hitachi band saw machine was used to cross cut the sample to an appropriate length. This was important to ensure that the sample can be milled by a Riken grinder with screen size of 1.5 mm. For that small size of sample, it was hoped that other properties such as particle shape, regularity, surface area and volume ratio which may have an influence on the experimental results will be negligible. The properties of oil palm frond were later determined and presented as in *Table 1*.

TABLE 1. PROPERTIES OF OIL PALM FROND

Properties (wt%)	Oil palm frond	Method
Structural analysis		
Cellulose	47.3 ± 1.1	ASTM D1103
Hemicellulose	27.3 ± 1.9	ASTM D1104
Lignin	20.1 ± 2.4	ASTM D1106
Extractive	3.6 ± 0.1	ASTM D1107
Elemental analysis		
C	42.9 ± 1.2	Perkin Elmer Series II CHNS/O Analyser
H	6.9 ± 1.4	
N	0.5 ± 0.1	
S	0.03 ± 0.02	
O	46.3 ± 2.4	
Proximate analysis		
Moisture content	7.5 ± 0.3	A&D MX-50 moisture analyser
Volatile matter	82.4 ± 1.4	ASTM E872
Ash	3.4 ± 0.3	ASTM D1102
Fixed carbon	14.1 ± 1.6	By difference

Following that, slow pyrolysis was carried out according to the experimental set-up shown in *Figure 1*. In a typical run, approximately 200 g of milled oil palm frond was introduced into a stainless steel pyrolyser and placed inside the reactor. Nitrogen gas was purged into the pyrolyser at 100 ml min⁻¹ to facilitate the removal of pyrolysis vapours into the liquid collection system (Ertas and Alma, 2010). The reactor was then heated to 375°C at a steady rate of 100°C min⁻¹. These parameters correspond to the optimised yield of phenols obtained in the bio-oil (unpublished data). After 1 hr of holding time, the reactor was turned off and allowed to stabilise to room temperature. The bio-oil, as obtained from the condensation of pyrolysis vapours was collected, separated using dichloromethane and analysed as in *Table 2* before being used in the synthesis of BPF resin.

Synthesis of BPF Resin

BPF resin was synthesised according to the previously described method with a slight modification (Chaouch *et al.*, 2014). In brief,

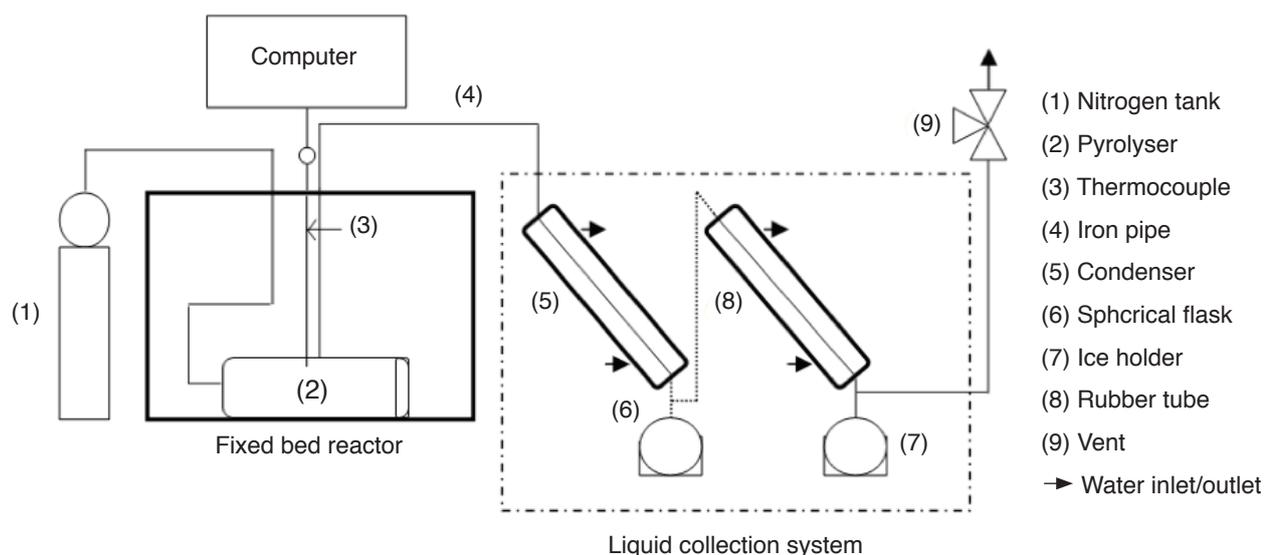


Figure 1. Experimental set-up for slow pyrolysis

liquefied phenol (99%), bio-oil, anhydrous ethanol (99%) and sodium hydroxide (50%) were loaded into a resin kettle equipped with a condenser, dropping funnel and thermometer. The mixture was heated to 65°C and maintained at that temperature for 30 min to ensure homogenous alkaline medium

TABLE 2. PROPERTIES OF BIO-OIL

Properties (wt%)	Oil palm frond	Method
Element		
C	66.23 ± 1.14	Perkin Elmer Series II CHNS/O Analyser
H	9.35 ± 1.16	
N	0.05 ± 0.02	
S	0.00 ± 0.00	
O	23.77 ± 2.30	
Ash (wt%)	0.61 ± 0.03	ASTM D1102
Char (wt%)	0.32 ± 0.13	Filtration method
pH	3.01 ± 0.03	Accumet AB15 pH meter
Total phenols (%)	74.39	
Phenol	40.60	Agilent Technologies 7890A / 5975C GCMS
Phenol, 2-methyl-	1.69	
Phenol, 3-methyl-	2.31	
Phenol, 2-methoxy-	8.47	
4-Mercaptophenol	1.43	
Phenol, 2,5-dimethyl-	1.36	
Phenol, 4-ethyl-2-methoxy-	4.35	
Phenol, 2,6-dimethoxy-	14.18	

was introduced (Chaouch *et al.*, 2014). After homogenisation has occurred, the temperature was raised to 80°C and formaldehyde solution (37%) was added step-wise over a period of 10 min. Finally, the reaction mixture was heated to 95°C and kept at that temperature for a period of time to allow condensation reaction and polymerisation to occur. After the the required viscosity as seemingly reached the reaction was stopped and allowed to stabilise to room temperature. The BPF resin was then refrigerated in a sealed glass bottle to prolong its pot life by minimising any additional slow polymerisation of phenolic rings (Sukhbaatar *et al.*, 2009). The optimum formulation used to prepare the resin was summarised in Table 3.

For easy reference, abbreviation of PF, BPF(OP25) and BPF(OP75) were used throughout this work denoting conventional PF resin, BPF resin synthesised with 25% of bio-oil and BPF resin synthesised with 75% of bio-oil respectively.

The properties of BPF resin were then compared with the properties of conventional PF resin purchased from Asta Chemicals Sdn Bhd. Viscosity measurement was conducted using Visco Basic Plus viscometer with L1 spindle, according to ASTM D1084. The pH value of the resin was determined using Accumet AB15 pH meter. Meanwhile, the non-volatile content of the BPF resin was evaluated at 105 °C in reference to ASTM D4426. Free formaldehyde level was measured according to ISO 11402.

TABLE 3. OPTIMUM FORMULATION OF BIO-PHENOL-FORMALDEHYDE (BPF) RESIN

Type of adhesive	Bio-oil substituted (%)	F/P	Molar ratio NaOH/P	EtOH/P	Time to reach 200 cP (hr)
BPF(OP25)	25	1.3	0.3	0.4	5.0
BPF(OP75)	75	1.3	0.5	0.4	3.5

On the other hand, differential scanning calorimetry (DSC) analysis was conducted to evaluate the thermal curing properties of BPF resin using Perkin Elmer DSC Pyris 6. Approximately 10 mg of resin was sealed in the given DSC aluminium pan, placed onto the sample holder and heated from 30°C to 250°C at 10°C min⁻¹. A flow of nitrogen gas at 20 ml min⁻¹ was maintained over the sample to create a dry and reproducible atmosphere.

Fabrication of *Rhizophora* Particleboard

Rhizophora hardwood was also collected in August 2014 from a charcoal factory in Kuala Sepetang, Perak, Malaysia (4°50'12.1"N 100°38'13.9"E). During the collection, bark of the *Rhizophora* was removed using bark spud and a total of two bark-free hardwoods were randomly chosen. Immediately after retrieval, the hardwood was dried, cut and milled into less than 200 µm using Retsch grinder so that the resulted particleboard had considerable similarity to water phantom (Rabaiee *et al.*, 2015). Then, 10 wt% of the BPF resin was mixed with the pre-weighed *Rhizophora* particles. The thickness and density of particleboard were fixed at 0.5 cm and 1 g cm⁻³ respectively, in reference to the required dimension of analysis and density of water.

To compress the mixture, a 23 x 23 cm² pressing mould was prepared. This pressing mould was placed on top of a metal plate, covered with aluminum foil to prevent the wood particles from sticking on the metal plate after compression. Then, the mixture was laid evenly within the mould forming a mat. A mould cover was also prepared. The mat was pre-pressed using Bluepoint 001 Heating and Pressing Machine manufactured by Milestone Technology Enterprise at room temperature for 2 to 3 min before hot-pressed using Fabricate Molding Test Press by GT Instrument Sdn Bhd at 170°C for 6 min. After the compression, the particleboard was left to cool down to room temperature before being trimmed and cut for further analysis.

The particleboard was analysed according to JIS A 5908 to determine the mechanical properties such as internal bonding (IB) strength and modulus of rupture (MOR) value as well as the physical properties such as thickness swelling and water absorption. Formaldehyde emission of the particleboard was also measured according to the desiccator method as in JIS A 1460. To analyse the morphological properties of the particleboard, FEI Nova NanoSEM 450 field emission scanning electron microscopy (FE-SEM) was employed at 500X and 3000X magnification. Following that, the qualitative elemental composition of the particleboard was calculated.

RESULTS AND DISCUSSION

Properties of Conventional PF and BPF Resins

The properties of the conventional PF and BPF resins were determined and presented as in Table 4. All BPF resins successfully reached the desired viscosity around 200 cP, as in conventional PF resin. The BPF resins also had comparable pH value to those of conventional PF resin. These two findings were expected since the final viscosity and pH value of the resins were carefully controlled by varying the condensation time during synthesis procedure and the molar ratio of sodium hydroxide to phenol, respectively.

Furthermore, the non-volatile content of all BPF resins was close and slightly higher than that of conventional PF resin. The high non-volatile content was associated to the small presence of volatile component such as free formaldehyde that was not consumed during synthesis reaction and hence, evaporated during heating period. The amount of free formaldehyde in the resins was indeed small enough to comply with the above deduction.

Free formaldehyde level of BPF resins increased significantly with an increase of substitution level of phenol with bio-oil. This was mostly due to the lower reactivity of bio-oil over phenol or lower

TABLE 4. PROPERTIES OF RESINS

Properties	PF (other work)		PF (this work)	BPF(OP25)	BPF(OP75)
	Value	Reference			
Viscosity (cP)	250-500 200 233	(Ayrilmis <i>et al.</i> ,	209 ± 3	220 ± 4	204 ± 2
pH	11.75,	2008; Zhao <i>et al.</i> ,	9.4 ± 0.0	11.6 ± 0.0	11.8 ± 0.1
	11.16, 10.5	2010; Chaouch <i>et al.</i> ,	42 ± 0	57 ± 1	43 ± 0
		2014)	Not detectable	0.17 ± 0.05	0.41 ± 0.04
Non-volatile content (wt%)	47, 64, 49	Chaouch <i>et al.</i> (2014)	166	154	156
Free formaldehyde level (wt%)	Not detectable	Zhao <i>et al.</i> (2010)			
Curing temperature (°C)	150	Cheng <i>et al.</i> (2011)			

number of active sites especially in –ortho and –para position to the phenolic hydroxyl group of the bio-oil which caused poor interaction between phenol and formaldehyde, hence leaving an amount of free formaldehyde at the end of synthesis (Cheng *et al.*, 2011).

Meanwhile, the curing temperature of the resins was represented by the single exothermic peak obtained from DSC result. This curing reaction was important to determine the hot pressing temperature during particleboard fabrication as further condensation and polymerisation would occur, producing a more stable cross-linked resin structure. From the table, it was observed that the curing reaction of BPF resins was obtained at an approximately equivalent temperature. Therefore, with regards to the high value of curing temperature of conventional PF resin, it was right to assume that by 170°C, all of the resins would have been cured and this temperature would be the most suitable temperature to be used as the pressing temperature of particleboard.

Physical and Mechanical Properties of *Rhizophora* Particleboard

Table 5 shows that both the conventional PF and BPF resins indeed lived up to expectation, as they provided superior IB strength and MOR value when compared to the standard limit of particleboard or when compared to the previous literature; fabrication of *Rhizophora* particleboard using Arabic gum and *Serishoom* adhesive (Abuarra *et al.*, 2014; Tousi *et al.*, 2015).

The exceptional strength of the formaldehyde-based resins used in this work was mostly due to the interaction of formaldehyde with phenol that created a strong and durable polymer upon condensation and polymerisation. Once cured, this polymer became more stable by the formation of a rigid cross-linked structure, hence further enhancing the mechanical properties of the particleboards.

When different types of resins were employed, no significant change on the IB strength and MOR value was observed, except for a slight increase or decrease according to the viscosity of resins which somewhat affected the mechanical properties (Pizzi and Stephanou, 1994). No significant impact was also seen on the thickness swelling and water absorption, except for a slight variation following the mechanical properties. Mechanical properties of a particleboard affected its physical properties in a way that better bonding contact provided greater resistant to any foreign materials including moisture (Pizzi and Stephanou, 1994).

Furthermore, the significant low value of thickness swelling and water absorption obtained in this work was mostly possible due to the nature of polymer produced from the interaction between formaldehyde and phenol. Apart from providing strong and durable bond, this polymer was known to be less soluble in water. Therefore, the phenolic resin that cured with this type of polymer had a low tendency to interact with water when immersed (Pilato, 2010). Due to that, the thickness swelling and water absorption of the particleboards were lower than those reported in any other work (Abuarra *et al.*, 2014, Tousi *et al.*, 2015).

In addition, all particleboards notably had lower formaldehyde emission than the standard limit with a value ranging from 0.1 mg litre⁻¹ to less than 0.5 mg litre⁻¹. The change in formaldehyde emission when different types of resins were used varied according to the free formaldehyde level of resins since the two were inter-connected. Formaldehyde emission of a particleboard was influenced by two different factors. The first factor was the evaporation of unreacted free formaldehyde from the particleboard over time, especially in a high temperature environment. For this reason, any decrease in the free formaldehyde level of the resins caused a reduction in the formaldehyde emission and *vice versa*. The second factor was hydrolysis reaction that occurred in a high moisture environment. During

TABLE 5. PROPERTIES OF RHIZOPHORA PARTICLEBOARD

Type of resin	IB (MPa)	MOR (MPa)	Thickness swelling (%)	Water absorption (%)	Formaldehyde emission (mg litre ⁻¹)
PF	1.61 ± 0.13	16.2 ± 1.9	12.0 ± 2.5	36.6 ± 5.3	0.15 ± 0.08
BPF(OP25)	1.46 ± 0.03	15.5 ± 2.2	12.7 ± 3.2	42.8 ± 5.0	0.27 ± 0.14
BPF(OP75)	1.65 ± 0.10	16.4 ± 3.3	11.7 ± 2.8	36.4 ± 6.5	0.45 ± 0.14
Arabic gum (Abuarra <i>et al.</i> , 2014)	0.7 – 1.2	-	80 – 110	90 – 100	-
<i>Serishoom</i> (Tousi <i>et al.</i> , 2015)	0.6	-	15	50	-
Standard limit*	> 0.5	> 8	< 12	NA	< 0.5

Note: IB - internal bonding.
MOR – modulus of rupture.

* Standard limit of particleboard for general purpose as issued by Japanese Industrial Standard (JIS).

this hydrolysis reaction, chemical bond created from the condensation and polymerisation process was urged to break down. However, since the polymer produced from chemical interaction between phenol and formaldehyde was less soluble in water, the resins provided little susceptibility to hydrolysis reaction. This property made the PF and BPF resins chemically stable and caused lower emission of formaldehyde, than other types of formaldehyde-based adhesive, such as urea formaldehyde (UF) resin (Kibert, 2008). A previous work has reported the formaldehyde emission of UF resin by investigating the effect of different compression time of particleboard on the formaldehyde emission. In that work, the particleboard was produced by mixing wood particles with 10% of UF resin and the resulting formaldehyde emission was shown to decrease from $0.9 \text{ mg litre}^{-1}$ to $0.8 \text{ mg litre}^{-1}$ with an increase of compression time, ranging from 3 min to 5 min (Eom *et al.*, 2006). Another work has also been conducted to study the formaldehyde emission of $(40 \times 40 \times 6) \text{ cm}^3$ plywood bonded with PF resin that had less than 0.1 wt% of free formaldehyde level. The plywood was prepared using single poplar veneer in the middle and two Eucalyptus veneers on top and bottom of the panel. The veneer was then coated with 0.013 g cm^{-2} PF resin on each side. The formaldehyde emission of the plywood was analysed using desiccator method and the result was obtained at $0.13 \text{ mg litre}^{-1}$ (Zhang *et al.*, 2013).

Morphological Properties of *Rhizophora* Particleboard

The micrographs of all particleboards, as obtained from FE-SEM analysis were displayed in Figure 2 until Figure 4. At the magnification of 500X, neither the wood structure of *Rhizophora* nor the resin network of PF (or BPF) can be identified. Instead, these micrographs showed a rather homogenous blend of network with small amount of void spaces equivalently scattered throughout the surface, hence, indicating a compact distribution within. This compact distribution was produced probably due to the satisfactory mixing procedure as well as efficient compression procedure of the particleboard. Nevertheless, the result was certainly expected since all particleboards produced in this work had a relatively high IB strength and MOR value. The value of the two parameters was greatly influenced by the compactness and homogeneity of the network holding the particleboard together (Abuarra *et al.*, 2014). Furthermore, the presence of adhesive within the particleboards was seen to have a good contact with the wood particles, as observed at 3000X magnification. The balanced interaction between the adhesive and the wood particles consequently enhanced the mechanical properties of the particleboards.

Meanwhile, Table 6 shows the elemental composition of each type of particleboards. Carbon and oxygen were mainly found in the fabricated *Rhizophora* particleboards, with a trace content of sodium. This finding contradicted with previous work that studied the morphological properties of binderless *Rhizophora* particleboard which reported that high weight percentage of carbon and oxygen and low weight percentage of nitrogen were found in the particleboard (Marashdeh *et al.*, 2012). This small contradiction was mostly possible due to the addition of the adhesive.

From there, the average atomic number of all particleboards was calculated and compared with those of water (7.42) (Singh and Badiger, 2014). The average atomic number of natural *Rhizophora* hardwood was previously reported at 7.28 (Marashdeh *et al.*, 2012). In this work, the average atomic number of all particleboards was near to those of water and did not deviate much from the natural *Rhizophora* hardwood, hence raising its possibility to be used as a phantom material.

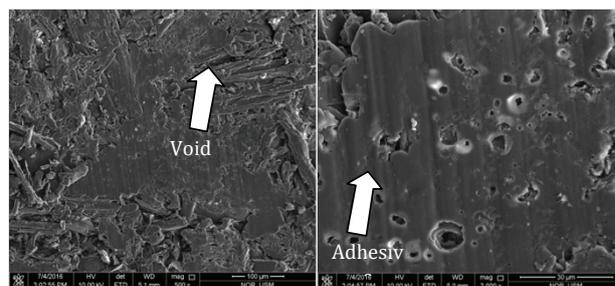


Figure 2. Micrograph of particleboard bonded with 10% phenol formaldehyde (PF) resin (left: 500X, right: 3000X).

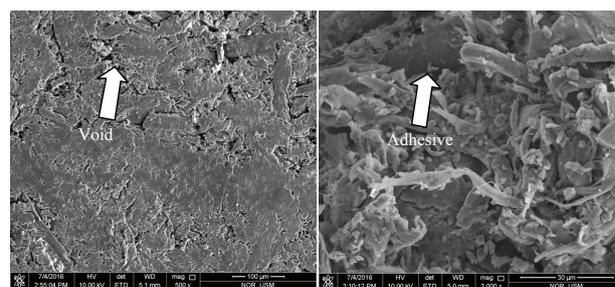


Figure 3. Micrograph of particleboard bonded with 10% bio-oil-phenol-formaldehyde (BPF) (OP25) resin (left: 500X, right: 3000X).

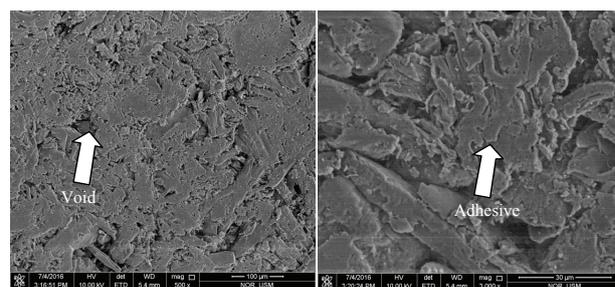


Figure 4. Micrograph of particleboard bonded with 10% bio-oil-phenol-formaldehyde (BPF) (OP75) resin (left: 500X, right: 3000X).

TABLE 6. ELEMENTAL COMPOSITION OF RHIZOPHORA PARTICLEBOARD

Type of adhesive	Carbon	Oxygen	Sodium	Aluminium	Average atomic number
PF	59.60	32.41	4.16	3.83	7.53
BPF(OP25)	51.59	40.21	8.20	-	7.50
BPF(OP75)	50.21	44.95	4.84	-	7.37

Note: PF - phenol formaldehyde.

BPF – bio-oil-phenol-formaldehyde.

CONCLUSION

Due to the high similarity and some superiority of the BPF resin, the possibility to fabricate *Rhizophora* particleboard bonded with this BPF resin was enhanced. The fabrication procedure of this type of particleboard was standardised with those bonded with conventional PF resin to equivalently compare the physical, mechanical and morphological properties. It was found that all the particleboards fabricated in this work showed excellent mechanical and physical properties surpassing the required standard limit issued by the Japanese Industrial Standard (JIS) with satisfactory formaldehyde emission. The morphological properties of the particleboard also supported the previous finding. In addition, it was seen that the average atomic number of the particleboard closely predicted the average atomic number of water. Therefore, further research on the application of the particleboard as a phantom material would be beneficial.

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