

Journal of Engineering and Science Research 7 (1): 05-11, 2023 e-ISSN: 2289-7127 © RMP Publications, 2023 DOI: 10.26666/rmp.jesr.2023.1.2



# Performance Evaluation of Oil Palm Trunk Starch as Binder, Modified by Sodium Trimetaphosphate

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Received: 08 November 2022; Revised: 15 November 2022; Accepted: 30 December 2022; Published: 10 February 2023

**Abstract:** The objective of this study was to evaluate the properties of particleboard prepared from starch-based adhesive, modified with sodium trimetaphosphate as a binder. Three-layer experimental panels were manufactured from a homogenous mixture of Acacia mangium and rubberwood. Panels were prepared from two different target densities of 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> and four different combinations of adhesive. The four combinations of adhesives were sequentially urea-formaldehyde (UF), native oil palm trunk starch (OPT-starch), sodium trimetaphosphate (STMP), modified starch, and STMP-starch mixed with 2% UF. Adhesive properties were analyzed by thermogravimetric (TGA), differential calorimetry (DSC), X-ray diffractometer (XRD), and scanning electron microscopy (SEM). The prepared panels were evaluated for their physical and mechanical properties. An experimental panel with 0.80 g/cm<sup>3</sup> density showed better mechanical and physical properties compared to 0.60 g/cm<sup>3</sup>. Oil palm trunk starch modified with sodium trimetaphosphate exhibited an improvement in physical and mechanical properties when it was used in combination with UF resin.

**Keywords:** *oil palm trunk starch, modified starch, sodium trimetaphosphate, particleboard* 

# 1. Introduction

Formaldehyde-based adhesives are the most common adhesives in wood composite manufacture, used for their excellent physical and mechanical properties at lower cost. However, the formaldehyde emissions from these composites are a major problem. These synthetic adhesives cause not only significant health problem but also pollute the environment after dumping [1]. Therefore, an extensive research is going on to develop a low-cost particleboard without having formaldehyde emission with acceptable properties [2-3]. Various natural products like protein, starch, lignin, and tannin are under investigation to prepare a bio-adhesive which will be eco-friendly and renewable. Starch is one of the bio-adhesives which is derived from various commercial crops such as corn, rice, sago, and oil palm and is readily available. Malaysia is one of the biggest producers of palm oil, but the palm trunk leftover after crop rotation is considered waste. These oil palm tree residue, especially oil palm trunk (OPT) is left to decay or burned in the field which is a loss of natural resources [4]. It has been reported that 25-37 years old trees, covering approximately 365,000 ha of oil palm plantation, will produce nearly 126,000 ha of old trees per year over the next ten years [5]. For Malaysia, it is an advantageous to utilize oil palm starch to produce starchbased products. Since starch has the potential to be used as a green binder, oil palm trunk starch can also be modified to prepare starch adhesive. Oil palm trunk (OPT) contain the highest percentage of starch among all of the parts of the oil palm tree [6-7]. Normal starches extracted from the OPT are used in the food industry due to their high content of

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glucose. Recent studies on modified starch based adhesive showed that it has the potential to be used as a green binder in particleboard manufacturing [7-8].

Chemical modification such as crosslinking and substitution are common methods used for the starch modification [9]. Crosslinking of starch causes delayed viscosity development and slows down the granules' swelling. It improves the starch's tolerance to heat, acid and shear and produces a stable product [10]. In this study sodium trimetaphosphate (STMP) was introduced as a crosslinking agent and produced a modified sodium trimetaphosphate starch (STMP-Starch). STMP is a colorless solid with low toxicity and is widely used in the food industry as additives [9]. The crosslink process of starch with STMP is usually carried out in an aqueous slurry at a warm temperature [11]. STMP has been chosen due to its low toxicity and is ideal for producing good green binder.

# 2. Materials and Method

#### 2.1 Raw material and starch extraction

Starch was extracted from the oil palm trunk (OPT) supplied by a local oil palm plantation in Malaysia. Harvested oil palm trees were cut into discs with an approximate thickness of 3 cm. Starch from the samples was extracted according to method describe by Noor et al. [12]. The discs were cut into small cubes and ground into fine particles before being immersed in 1000 mL of a 0.5% (w/v) aqueous sodium metabisulphite solution for 48 h. The particles were squeezed and filtered using a nylon bag. The filtered solution was centrifuged at 3700 rpm for 10 min using a Beckman Coulter Allegra X-15R centrifuge. Starch collected from the centrifuge bottle was dried in an oven at  $50\pm 2^{\circ}$ C for 3 days before it was ground and stored.

#### 2.2 Starch modification

The modification of starch was carried out according to previous studies by Chatakanonda et al. [9]. Sodium trimetaphosphate (STMP) was used as cross linking agent for this modification. Sodium tripolyphosphate 0.3 g, sodium carbonate, 9 g, and variable amounts (18, 27 and 39 g) of STMP were dissolved in distilled water (300 mL). Variable amounts of STMP were used to diversify the degree of crosslinking. At pH 11, 100 g of oil palm starch was introduced to the solution and the mixtures were stirred, at temperature of 50 °C. The starch suspensions were adjusted to pH 6.5 and then centrifuged at 1000 rpm. A clear solution was deposed and the residue was retained and washed with 5000 mL of distilled water. The starch sediment was dried at 50 °C for 48 h in an air dryer oven before being ground and stored for further use as adhesive for particle board making.

#### 2.3 Particleboard manufacturing

A homogeneous mix of 80% rubberwood and 20% Acacia mangium particles was used for the making of three-layer particleboard. Particles used in this study were obtained from Heveaboard Sdn Bhd, located in Seremban, Negeri Sembilan, Malaysia. The particles were dried in an oven to obtain a moisture content of approximately 5%. All panels had a homogeneous mixture of 20% Acacia and 80 % rubberwood and were produced based on two target densities of 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup>. Native oil palm trunk starch (OPT-Starch) was chemically modified by using sodium trimetaphosphate (STMP-starch), urea formaldehyde (UF) and a mixture of STMP-starch and UF (STMP-UF) as the binder for those experimental panels produced. Particles mixed with binder were manually laid out in a frame with dimensions of 21 cm  $\times$  21 cm  $\times$  0.5 cm. Mats were initially pre-pressed using hot press machine for 2 min at 5 MPa before they were hot pressed at a temperature of 160 °C and a pressure of 5 MPa for 20 min. A total of 40 panels were manufactured for this experiment.

#### 2.4 Particleboard evaluation and characterization

The physical and mechanical properties of the panels, such as modulus of elasticity (MOE), modulus of rupture (MOR), internal bond strength (IB), density, thickness swelling (TS), and water absorption (WA), were evaluated using the Japanese Industrial Standard (JIS-A-5908, 2003) [13]. Three 5 cm  $\times$  5 cm samples from each panel were used for densitv measurement and dimensional stability characterization of the panels. They were soaked in distilled water for 2 h and 24 h to determine their thickness swelling and water absorption. Each sample was weighed, and their dimensions were measured at an accuracy level of 0.001 g and 0.001 mm before and after soaking. Mechanical tests were carried out by Instron testing machine model 5582. Five samples with 5cm by 12cm were used for bending while internal bond strength was tested on fifteen 5 cm by 5cm square samples. Crosshead speeds of 10mm/min and 2 mm/min for bending and internal bond strength test, respectively, were applied [14].

#### 2.5 Starch characterization

# 2.5.1 Fourier transform-infrared spectroscopy (FT-IR) analysis

Fourier transform infrared spectroscopy (FT-IR) was used to identify the presence of major functional groups in unmodified and modified starch. The FT-IR spectra of the starch samples in a powder form were taken in Nicolet Avatar 360 ESP FT-IR with potassium bromide (KBr) pellets. Approximately 95 mg of finely ground KBr were mixed together with 5 mg of powdered for each sample. The mixture was pressed into pellets with thickness approximately 1 mm. Each sample was scanned 64 times over a region of 4000-400 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>.

#### 2.5.2X-ray diffractometer (XRD) analysis

This analysis was to determine the percentage of a crystalline structure and lattice constant in the oil palm trunk starch and modified starch. Powdered starch samples were taken to be analyzed with a KristalloflexD-5000 X-ray diffraction system (Siemens, Germany). The powdered samples were filled and packed as densely as possible in a sample holder. An air blow was used to smooth the finished surfaces of the samples. Step scan measurements were done using X-rays (Cu-K $\alpha$ ) generated at an opening voltage and current of 40 kV and 30 mA, respectively. The scan was made at a diffraction angle 2 $\theta$  ranging from 10° to 40°, corresponding to a scanning speed of 0.02° and 2°/min. The crystallinity index (CrI) was calculated using Eq. (1) [15].

$$C_r I(\%) = (I_{200} - I_{am}) / I_{200} \times 100$$
 Eq (1)

where  $I_{200}$  is the peak intensity of crystalline fraction and  $I_{am}$  is the peak intensity of amorphous fraction.

#### 2.5.3 Scanning electron microscopy (SEM) analysis

A small portion of starch powder was glued on a stub using tape. Then, the samples were coated using the Polaron SC515 SEM coating system (Fisons Intrusment) with a thin layer of gold to make them conductive. A Scanning Electron Microscope (Model Supra 50 VP) was used to observe at the samples with an acceleration of 15 kV.

#### 2.5.4 Thermogravimetric analysis (TGA)

Thermogravimetric analysis was used to determine the decomposition rate or the total weight loss and thermal stability of the starch samples. Thermal analysis of each starch sample was carried out using Mettler Toledo TGA/SDTA851<sup>e</sup> thermogravimetric (Mettler Toledo Corp., Switzerland) with STAR<sup>e</sup> software (version 9.20). Approximately 10 mg of the powder samples were placed in an aluminum pan before being heated with a heating rate set at 20°C/min over a temperature range between 30 °C and 800 °C under a nitrogen atmosphere.

## 2.5.5 Differential scanning calorimetry (DSC) analysis

The melting temperature  $(T_m)$  of each starch sample was determined using Perkin Elmer thermal analysis (Model DSC 8000). This analysis was carried out to determine the thermal stability of the oil palm trunk starch and modified oil palm trunk starch. The powder sample was weighed out at approximately 5 mg into an aluminum pan and was transferred to the heating pan with the usage of empty pan as a reference. The sample was heated at a rate of 10 °C/min from -15 °C to 280 °C. Subsequently, the sample was cooled back from 280 °C to -15 °C at 100°C/min after being hold for 1 min at 280 °C. Finally, the sample was heated up again from -15 °C to 28 °C at a heating rate of 10 °C/min after being hold for 5 min at -15°C. The measurement was carried out under nitrogen atmosphere.

#### **3. Results and Discussion**

#### **3.1** Starch characterizations

#### 3.1.1 Morphological analysis

The effect of chemical modification on the native starch of oil palm trunk was observed by SEM analysis. Figures 1a and 1b show the SEM micrographs of oil palm trunk starch (OPT-starch) and sodium trimethaphosphate starch (STMP-starch) based on oil palm trunk. There was a slight difference in the physical structure of starch granules after the modification process. The granules were spread and isolated from each other, and the smooth elliptical form of the starch granule was slightly changed. Based on the observation, the crosslinking process with sodium trimetaphosphate give a minor change on the physical appearance of starch derived from oil palm trunk.



Figure 1. Microscopic image of (a) oil palm trunk starch (OPT-starch) and (b) OPT-starch modified with sodium trimethaphosphate (STMP) based on oil palm trunk.

#### 3.1.1 FTIR analysis

The Fourier transform infrared (FT-IR) spectra of oil palm trunk starch (OPT-starch) and sodium trimetaphosphate starch depicted in Figure 2 show the typical peak of the starch backbone. The peaks at 3406 cm<sup>-1</sup> for OPT-starch and 3384 cm<sup>-1</sup> for STMP-starch are the result of O-H stretching vibration. The usual O-H stretching vibration is in the range of 3700-3500 cm<sup>-1</sup>, but for starch, the O-H stretching peak shifts to a lower wavelength, normally in the range of 3400–3200 cm<sup>-1</sup> and this may happen due to the intermolecular hydrogen bonds in the glycosidic ring weakening the O-H bond [16]. On the other hand, there is a decrease in the intensity of vibration for O-H stretching of STMP-starch and this may be due to the reaction involving two starch hydroxyl groups per phosphate [17]. The C-H stretching is observed at 2930 cm<sup>-1</sup> for OPT-starch and for STMP-starch. Peaks at 1638 cm<sup>-1</sup> for OPT-starch and 1640

cm<sup>-1</sup> for STMP-starch are most probably from tightly bound water molecules present in starch molecules. Broad peaks at a range of 1200 cm<sup>-1</sup> to 1000 cm<sup>-1</sup> for both samples are characteristic of C-O stretching in the glycosidic ring of oil palm trunk starch [16].



Figure 2. FT-IR spectra of oil palm trunk starch (OPT-starch) and OPT-starch modified with sodium trimetaphosphate (STMP).

#### 3.1.2X-ray diffractometer (XRD) analysis

The crystallinity index was calculated using Eq. (1). Both OPT-starch and STMP-starch are C-type starches [6]. The crystallinity index for native starch was 58.19%, while the crystallinity index for STMP starch was 49.45%. The major intensity lines for both samples were close to  $13^{\circ}$  of  $2\theta$  angle. Both samples show a high crystallinity index. This may contribute to the high amylose content in both starch samples. The alteration of starch structure by the crosslinking process, which with sodium trimetaphosphate decreases the crystallinity index of the starch. It may result from crosslinking treatment conditions (e.g., alkaline or acid conditions) that might degrade the branch structure [18].

## 3.1.3DSC analysis

Figure 3 shows the melting point  $(T_m)$  of native starch was 86 °C and STMP-starch melted at 96 °C. It shows that the thermal properties of starch were improved after the crosslinking with sodium trimetaphosphate. Although the XRD results suggest that native starch had a higher crystallinity index, due to the additional covalent bond of phosphate groups after the crosslinking process, starch granules are firmly held together, restricting the molecular mobility of the starch molecules, resulting in a higher melting point. The crosslinking also inhibits the swelling of starch granules, thus increasing the melting point [9].



Figure 3. DSC thermograms of oil palm trunk starch (OPT-Starch) and OPT-starch modified with sodium trimetaphosphate (STMP).

#### 3.1.4 Thermogravimetric analysis (TGA)

Based on Figure 4, both samples experienced weight loss in three steps. The initial weight loss was caused by the evaporation of water and any other volatile materials in both samples [19].The second and third weight losses were caused by the dehydration and complete degradation of the samples [4, 20]. The native OPT starch showed a slightly lower weight loss for the second and third stages. For the third stage of weight loss, approximately 31.01% of the weight remained at 353.5 °C and 29.25% of the weight remained at 354.1 °C for both samples. Based on the DTG curve, STMP-starch exhibits a slightly faster rate of decomposition. This may result from the lower crystallinity of STMP-starch. A higher temperature is needed to break the crystalline structure, and the crystalline structure tends to have a higher and sharper melting point.



Figure 4. Thermogravimetric (TG) and Derivative Thermogravimetric (DTG) curves of oil palm trunk starch (OPT-starch) and OPT-starch modified with sodium trimetaphosphate (STMP).

#### 3.2 Physical properties of particleboard

Table 1 shows the physical properties of the manufactured panels. The moisture of the particles and the binders used have been controlled to ensure the allowable range of standard JIS-A-5908 [13] which is between 5 % and 13 %. The types of binder used in board manufacturing did not affect the moisture content (MC) of the specimens.

Table	1.	Physical	properties	of	particleboard	samples
prepar	ed f	rom four d	ifferent adh	esiv	e.	

Target density, g/cm <sup>3</sup>	Type of binder	Density, g/cm <sup>3</sup>	Moisture content, %		
	OPT-starch	$0.64^{a} (0.03)^{b}$	5.62 (-0.17)		
0.6	UF	0.63 (-0.02)	5.62 (-0.17)		
0.6	STMP-starch	0.64 (-0.03)	5.57 (-0.13)		
	STMP-starch- UF	0.62 (-0.02)	5.84 (-0.14)		
	OPT-starch	0.78 (-0.04)	6.04 (-0.14)		
	UF	0.82 (-0.03)	6.17 (-0.18)		
0.8	STMP-starch	0.82 (-0.06)	6.22 (-0.15)		
	STMP- starch-UF	0.83 (-0.04)	6.25 (-0.14)		
<sup>a</sup> Average value <sup>b</sup> standard deviation					

Average value

Figure 5 the thickness swelling and it was observed that higher density panels exhibit higher thickness swelling. The OPT-starch bonded panel with 0.80 g/cm<sup>3</sup> target density had a maximum thickness swelling of 99 % and 104 %, and for 0.60 g/cm<sup>3</sup> the thickness swellings were 85 % and 104 % for 2 h and 24 h, respectively. The thickness swelling was observed to be lower for STMP-starch bonded panels. It was observed that 63 % and 95 % for 0.6 g/cm<sup>3</sup> whereas for 0.80 g/cm<sup>3</sup> STMP-starch panels were 78 % and 107 % for 2 h and 24 h, respectively. Although UF bonded panels had the least thickness swelling, the value was observed at 38 % and 46 % for 0.60 g/cm3 density and 48.00 % and 57.62 % for 0.80 g/cm<sup>3</sup> density when soaked for 2 h and 24 h, respectively. The thickness swelling for 0.60 g/cm<sup>3</sup> STMP-UF bonded panels was 38 % and 46 % for 2 h and 24 h, respectively, while for 0.80 g/cm<sup>3</sup> the thickness swelling was 48 % and 58 % for 2 h and 24 h, respectively.



Figure 5. Thickness swelling graph of all the eight samples prepared from four different adhesives in two different densities.

A similar trend was observed for the water absorption graph (Figure 6). UF-bonded panels showed the least water absorption, whereas the highest was observed for OPT-starch. After 24 h of soaking, water absorption for STMP-starch bonded panels was 132 % and 146 % for 0.60  $g/cm^3$  and 0.80  $g/cm^3$  density, respectively, whereas for OPT-starch bonded panels was 203.57% and 229 % for 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> density, respectively.The water absorption value for STMP-UF bonded panels was slightly better compared to STMP-starch bonded panels. For 0.60 g/cm<sup>3</sup> density, the values were 97 and 129 % for 2 h and 24 h respectively, and for 0.80 g/cm<sup>3</sup> density, the values were 89 % and 106 % for 2 h and 24 h respectively. The modification of OPT starch with sodium trimetaphosphate was proven to enhance the dimensional stability of those experimental panels.



Figure 6. Water absorption graph of all the eight samples prepared from four different adhesives.

However, the starch bonded panels showed very high thickness swelling. A considerable reduction was observed when starch was treated with STMP. Panels bonded with STMP-starch had a lower swelling value, and the value is further reduced when mixed with 2% of UF. The TGA and DSC results supported the theory that STMP treatment improves the crosslinking and gelatinization of starch molecules. Such crosslinking starch molecules not only enhance the structure of the starch but also reduce the hydroxyl group, which leads to an improved water stability of the experimental panels.

The results of mechanical properties, including the modulus of rupture (MOR), modulus of elasticity (MOE), and internal bond strength (IB) of experimental panels, are displayed in Figures 7 and 8. The results showed that 0.80 g/cm<sup>3</sup> panels had better mechanical properties compared to 0.60 g/cm<sup>3</sup> panels. Samples bonded with UF had the highest MOR values for both density levels. For 0.60 g/cm<sup>3</sup> and  $0.80 \text{ g/cm}^3$  particleboard, the panels made using STMP-starch exhibited higher MOR readings than the panels made using OPT-starch. For STMP-starch the values are 4.23 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> and 7.934 N/mm<sup>2</sup> for 0.80 g/cm<sup>3</sup>. The MOR readings for the panels that used OPT-starch were 3.9 N/mm<sup>2</sup> and 4.3 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> densities, respectively. While for STMP-UF bonded panels, the MOR readings for 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> were 4.71 N/mm<sup>2</sup> and 8.4 N/mm<sup>2</sup> respectively. UF bonded panels showed the highest MOR result, which was 5.25 N/mm<sup>2</sup> and 10.1 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> and 0.80  $g/cm^3$ .





Figure 7. Modulus of rupture (MOR) and modulus of elasticity (MOE) of particle boards prepared for different adhesives (a)  $0.6 \text{ (g/cm}^3)$  (b)  $0.8 \text{ (g/cm}^3)$  panel densities.



Figure 8. Internal bond (IB) strength of particle boards, prepared from different adhesives.

Based on the MOE result that was obtained, UF bonded panels have the highest strength with 1751 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> density and 2873 N/mm<sup>2</sup> for 0.80 g/cm<sup>3</sup> density. The lowest value was shown by OPT-starch bonded panels with a value of 1186 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> density and 1637 N/mm<sup>2</sup> for 0.80 g/cm<sup>3</sup> density. After modification, there was an increase in MOE value. STMP-starch bonded panels had shown increasing value. MOE for STMP-starch bonded panels is 1413 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> density and 2258 N/mm<sup>2</sup> for 0.80 g/cm<sup>3</sup> density, respectively. Adding the STMP-starch with UF thus increases the MOE of those experimental panels. The MOE readings for those panels were 1569 N/mm<sup>2</sup> and 2640 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> and 0.80 g/cm3 densities. Both 0.8 g/cm3 STMP-starch bonded with and without UF have met the minimum standard for Type 8 in the JIS A 5908 standard for MOE (2000 N/mm<sup>2</sup>).

Figure 8 shows the internal bond strength (IB). The highest IB strength was found on the UF bonded panels, which was 1.65 N/mm<sup>2</sup> and 2.07 N/mm<sup>2</sup> for panel densities of 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> respectively. The weakest IB strengths were shown by the native starch, at 1.0 N/mm<sup>2</sup> for 0.60 g/cm<sup>3</sup> and 1.1 N/mm<sup>2</sup> for 0.80 g/cm<sup>3</sup> density. The IB strength for STMP-starch bonded panels is 1.19 N/mm<sup>2</sup> and 1.43 N/mm<sup>2</sup> for densities of 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup>, respectively, and for STMP-UF bonded panels is 1.32 N/mm2 and 1.82 N/mm<sup>2</sup> for densities of 0.60 g/cm<sup>3</sup> and 0.80 g/cm<sup>3</sup> respectively. Based on Table 2, UF bonded panels exhibit the highest strength for MOR, MOE, and IB strength. The 0.80 g/cm3 showed higher mechanical properties compared to the 0.60 g/cm<sup>3</sup>. All panels had met the minimum standard of the JIS Standard for Type 8 (0.15 N/mm<sup>2</sup>). From these results, the values of MOR, MOE, and IB strength of those experimental panels made from STMP-starch as binder increased compared to OPT-starch bonded panels. The modification of starch with STMP enhanced the mechanical properties of those panels. This was caused by the chemical structure of the STMP-starch, which was highly branched and easily formed a strong bond with the particles, hence improving the mechanical strength of the panels. In addition, other factors, including starch purity, composition of starch granules, as well as particle size, can contribute to the properties of the samples [7,21].

#### 4. Conclusion

Modified starch exhibits improved mechanical and physical properties over non-modified oil palm trunk starch. However, the performance was further improved when it was applied in combination with 2% UF. In the SEM analysis as a physical appearance, it does not depict any obvious change in the starch granule. The TGA analysis of OPT-starch shows a lower rate of decomposition. This may ensue from the higher crystallinity index of OPT-starch based on XRD analysis. Furthermore, the DSC analysis results exhibit a higher melting point of STMP-starch due to additional covalent bonds from the phosphate group and tightly held molecule via crosslinked structure. With an abundant source of oil palm trunk in Malaysia, it shows great potential to be further developed as a green binder and produce environmentally friendly products.

#### 5. Acknowledgements

We acknowledge Universiti Sains Malaysia for the financial support under research University Grant (1001/PTEKIND/815066). We are thankful to Heveaboard Sdn. Bhd. for the raw material.

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