Flame retardant properties of oil palm trunk particleboard with addition of epoxy resin as a binder and aluminium hydroxide and magnesium hydroxide as additives

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Abstract. This research aimed at investigating the effect of epoxy resin and flame retardant additives on oil palm trunk particleboard. The oil palm trunk was used as a raw material with epoxy resin as a binder, which was synthesized by a reaction of epichlorohydrin and phenolphthalein, while aluminium hydroxide and magnesium hydroxide were used as flame retardant additives to manufacture particleboards. Four different types of particleboards were produced, control board, board with 10% epoxy resin, board with 10% epoxy resin and 6% aluminium hydroxide and board with 10% epoxy resin and 6% magnesium hydroxide, based on a target density of 0.80 g cm^{-3} , at a temperature of 180°C , a pressure of 5 MPa and a pressing time of 20 min. The mechanical, physical, functional group, spectroscopic, morphological and elemental analysis properties were determined in comparison with control particleboard. Thermal degradation and flame retardant analyses were also carried out to analyse the thermal properties of particleboards. The results indicated that particleboard bonded with epoxy resin and flame retardant additives had improved mechanical, physical, thermal and flame retardant properties compared to control oil palm trunk particleboard. Overall results showed that epoxy resin as a binder and aluminium hydroxide and magnesium hydroxide as additives have the potential to be incorporated into the particleboard to improve flame retardant properties.

Keywords. Oil palm trunk; epoxy resin; mechanical; physical; flame retardant analysis; thermal degradation.

1. Introduction

The oil palm (*Elaeis guineensis*) is becoming one of the most valuable commercial agricultural crops in Malaysia as it is easily cultivated [1]. Cultivation of oil palm plantation in Malaysia has increased dramatically from years to years due to its oil producing fruits. In Malaysia, an oil palm planted area reached 5.74 million hectares in 2016, which was an increase by about 1.77% compared to 5.64 million hectares in 2015 [2]. At the same time, the industry generates at least 30 million tons of biomass waste per year in the form of palm kernel, trunks, empty fruit branches and leaves [3]. About 75% of the solid biomass in the form of fronds and trunks are found in the oil palm plantation and it is estimated that about 85 million tonnes of biomass based on dry weight will be generated by the year 2020 [4].

Oil palm trunks will be only available when the economic life span of palm tree achieves replanting period at an average age of about 25 years. Currently, most of the trunks are retained in the field as fertilizer. They are either chipped or felled to decompose naturally in the plantation. The utilization of oil palm trunks into value added products could be an essential principle of waste management by minimizing and

recycling waste materials and also reducing high dependency on wood resources.

Particleboard was successfully developed from oil palm trunk waste by Ahmad *et al* [5] using urea formaldehyde resin. In general, conventional particleboard was incorporated with formaldehyde based resin to obtain a high end product but inversely it emits toxic gases which are detrimental to human health and also to the environment [6]. This prompted the development of particleboard without addition of any binder or resin which is also known as binderless from oil palm biomass by Hashim *et al* [7,8].

Apart from that, particleboards manufactured from wood or non-wood have low flammability as a wood or non-wood material is a naturally growing resource which mainly consists of combustible organic carbon compounds [9]. Fire retardant properties are crucial in the particleboard industry to increase its life span. Therefore, the particleboard industry tends to develop safe and effective flame retardant formulations that could also solve formaldehyde emission.

Epoxy resins have widely been used as adhesives, surface coatings, laminates and matrix materials in wood plastic composites, and in electronic, transport and also aerospace

industries due to their exceptional characteristics such as superior dimensional stability and excellent mechanical properties with high resistance to corrosion, heat and chemicals [10]. However, their flame resistance needs to be improved with addition of additives such as inorganic hydroxides to improve basic properties of non-wood based particleboard for indoor applications. The quantity used must be minimal to ensure the optimum use of the adhesives and their flame resistant function. Previous work has indicated that a range of the low retention level of 5 to 10% should be enough to treat particleboards for a fire retardant purpose [11].

Therefore, in this research, oil palm trunk particleboard is produced by using epoxy resin which is prepared from the reaction between epichlorohydrin and phenolphthalein, and the flame retardant effect is evaluated by adding aluminium hydroxide and magnesium hydroxide as additives. The performance of the particleboard is analysed in terms of physical properties, mechanical properties, thermal properties, functional groups, morphological properties and also elemental composition.

2. Materials and methods

2.1 Preparation of epoxy resin

Epoxy resin was prepared based on a study by Mun *et al* [12]. Phenolphthalein (73.14 g, 0.23 moles), epichlorohydrin (124.88 g, 1.35 moles) and 2 ml of distilled water were stirred and heated until reflux. The solution was cooled until the temperature decreased to 80°C. Five grams of sodium hydroxide pellets were added slowly to the solution. The reflux process was then continued for further 45 min. After excess epichlorohydrin was removed by vacuum distillation, the solution was cooled and 30 ml of benzene was added. Yellow crystals were precipitated and filtered. The filtrate was further heated at a temperature less than 120°C. Finally, the epoxy resin was added to an oven at 140°C for 3 h to obtain phenolphthalein based epoxy resin. The reactions between phenolphthalein and epichlorohydrin are illustrated in figure 1.

2.2 Determination of epoxide equivalent

The epoxide equivalent was determined to analyse the molecular weight of resin [13]. 1.18 g of potassium hydroxide and 4.10 g of potassium hydrogen phthalate were dissolved separately with 100 ml of distilled water. A few drops of the phenolphthalein indicator was added into 10 ml of potassium hydroxide and the mixture was titrated with potassium hydrogen phthalate. Twenty-five millilitres of 37% hydrochloric acid was dissolved in 100 ml of dimethyl formamide. Ten millilitres of this mixture was titrated with standardized potassium hydroxide. One gram of epoxy resin was dissolved in 50 ml of dimethyl formamide hydrochloride solution. The mixture was refluxed for about 20 min. After this, the mixture was cooled and 10 ml of this mixture was titrated with potassium hydroxide solution.

2.3 Manufacturing parameters

Oil palm trunk particles used in this study were supplied by Encore Agricultural Industries Sdn Bhd, Kuala Lumpur. The oil palm trunk particles were ground to obtain oil palm trunk particles of size less than 1 mm. Particleboards measuring $205 \times 205 \times 5 \text{ mm}^3$ with a target density of 800 kg m^{-3} were manufactured using a hot press machine. Control particleboards were manufactured without addition of epoxy resin and inorganic hydroxides. A 10% epoxy resin based on oven dry weight of oil palm particles was used for the epoxy resin added particleboard designated as 10% epoxy resin. A 10% epoxy resin and 6% of inorganic hydroxides, namely aluminium hydroxide and magnesium hydroxide, based on oven dry weight of oil palm particles were added as a function of fire retardant additives for flame retardant epoxy resin added particleboard which are categorized as 10% epoxy resin + 6% $\text{Al}(\text{OH})_3$ and 10% epoxy resin + 6% $\text{Mg}(\text{OH})_2$.

After blending, mats were manually formed in a mould prior to hot pressing. Particleboards were pressed at a platen temperature of 180°C. The pressing time was 20 min and pressing pressure was 5 MPa [14]. A total of 24 particleboards were manufactured with 6 replicates for each type. Particleboards were conditioned at 20°C and 65% relative humidity

Figure 1. Reaction of phenolphthalein and epichlorohydrin [12].

until equilibrium was obtained with moisture content prior to testing.

2.4 Mechanical and physical properties

Samples for mechanical and physical properties testing were prepared according to Japanese Industrial Standards A 5908 for particleboard [15]. The modulus of rupture was obtained from an average of two $210 \times 50 \text{ mm}^2$ samples for each type of particleboard. The internal bond strength was obtained from an average of three $50 \times 50 \text{ mm}^2$ samples for each of particleboard. Thickness swelling after soaking the samples in water for 24 h was obtained from an average of three $50 \times 50 \text{ mm}^2$ samples for each particleboard.

2.5 Flame retardant and thermal properties

Flame retardant properties were measured using a limited oxygen index according to British Standard 2782:141 for the determination of flammability by oxygen index [16]. The limited oxygen index was obtained from an average of three $80 \times 10 \text{ mm}^2$ samples for each particleboard. Thermal properties were analysed using a Perkin Elmer TGA 7 thermogravimetric analyser. Scans were recorded from 30 to 800°C for samples in a powder form of 5–10 mg placed in an aluminium pan with a heating rate of $20^\circ\text{C min}^{-1}$ under a nitrogen atmosphere.

2.6 Functional group, crystallinity, morphology and elemental composition properties

The functional group was determined using a Nicolet Avatar 360 ESP Fourier transform infrared spectrophotometer. The samples in the powder form were analysed between wave numbers of 4000 and 470 cm^{-1} with a resolution of 4 cm^{-1} to detect the functional groups of the compounds of each material. The crystalline index was measured using a high resolution (Hr) X-ray diffractometer (PANalytical X'Pert Pro MRD). A 2θ angle ranging from 10° to 40° in a reflection mode was scanned at 2° min^{-1} . The morphology and elemental composition were analysed using a scanning electron microscope with energy dispersive X-ray analysis (LEO Supra 50 Vp), Field Emission SEM, (Carl-Zeiss SMT, Oberkochen, Germany). Micrographs were

obtained from cross sections of $5 \times 5 \text{ mm}^2$ for each type of particleboard.

3. Results and discussion

3.1 Epoxide equivalent of epoxy resin

Epoxide equivalent weight is the characteristic that determines resin solubility, which is defined as the weight of resin in grams which contains 1 g of equivalent [17]. Thus, the determination of epoxy equivalent is to determine the molecular weight of resin produced. The average molecular weight of epoxy resin produced is $2856.72 \text{ g mol}^{-1}$.

3.2 Evaluation of mechanical and physical properties

The results of mechanical properties, modulus of rupture and internal bond strength, physical properties and thickness swelling of particleboards manufactured from oil palm trunk particles were evaluated by Tukey's test as presented in table 1. It demonstrated the influence of addition of epoxy resin as a function of resin and aluminium hydroxide and magnesium hydroxide as a function of flame retardant additives on the mechanical and physical properties of oil palm trunk particleboards to reveal significant differences between responses analysed and the difference was considered significant at $p < 0.05$. Four types of oil palm particleboards were manufactured; control particleboard, particleboard with 10% of epoxy resin (10% epoxy resin), particleboard with 10% of epoxy resin and 6% of aluminium hydroxide (10% epoxy resin + 6% $\text{Al}(\text{OH})_3$) and particleboard with 10% of epoxy resin and 6% of magnesium hydroxide (10% epoxy resin + 6% $\text{Mg}(\text{OH})_2$).

All the oil palm trunk particleboards satisfied the minimum requirement of modulus of rupture which is 8 MPa, except for control particleboard, and internal bond strength which is 0.15 MPa based on Japanese Industrial Standard for Type 8 particleboard [15].

Tukey's test results show that there are significant interactions between additions of epoxy resin and also flame retardant additives on mechanical properties as observed in table 1. Epoxy resin added particleboard exhibited the highest values of mechanical properties namely modulus of rupture

Table 1. Mechanical and physical properties of oil palm trunk particleboard with Tukey's test result.

Types of boards	Density (g cm^{-3})	Modulus of rupture (MPa)	Internal bond strength (MPa)	Thickness swelling (%)
Control	0.80 (0.15)	5.58 (1.61) ^a	2.37 (1.22) ^a	64.42 (2.76) ^c
10% epoxy resin	0.80 (0.14)	12.48 (1.73) ^c	8.14 (1.82) ^c	47.81 (2.34) ^a
10% epoxy resin + 6% $\text{Al}(\text{OH})_3$	0.80 (0.13)	8.03 (1.57) ^b	6.41 (1.31) ^{b,c}	54.31 (2.35) ^b
10% epoxy resin + 6% $\text{Mg}(\text{OH})_2$	0.80 (0.14)	9.18 (1.51) ^b	5.63 (1.35) ^b	56.12 (2.49) ^b

Values in parentheses are standard deviation.

Note: Different letters in superscript represent statistical significance while same letters indicate a similarity of differences ($p < 0.05$).

and internal bond strength. In comparison with control board, modulus of rupture and internal bond strength values of the epoxy resin added particleboard increased significantly with 55.29 and 70.88%, respectively, because epoxy resin has superior mechanical properties and can provide high strength properties to the board [10]. However, mechanical properties decreased significantly with addition of inorganic hydroxides. Modulus of rupture values decreased 35.66% for addition of 6% $\text{Al}(\text{OH})_3$ and 26.44% for addition of $\text{Mg}(\text{OH})_3$ while internal bond strength values decreased 21.25% for addition of 6% $\text{Al}(\text{OH})_3$ and 30.84% for addition of $\text{Mg}(\text{OH})_3$. This might be due to the degradation of cellulose by the flame retardant additives [18] that cause poor fibre–matrix interface bonding.

Thickness swelling of oil palm trunk particleboards after 24 h immersion results in a significant interaction between additions of epoxy resin and flame retardant additives and values are presented in table 1. The particleboard with 10% of epoxy resin had the lowest thickness swelling values. The thickness swelling values decreased significantly 25.78% with addition of 10% epoxy resin compared to control board because the high resin content in a board can reduce efficiency to absorb moisture or water [19]. However, thickness swelling values increased on addition of flame retardant additives with 11.97 and 14.81% of $\text{Al}(\text{OH})_3$ and $\text{Mg}(\text{OH})_3$, respectively, as illustrated in table 1. The thickness swelling properties of a board is highly dependent on bonding quality [20] and adhesive content. This indicates that the presence of flame retardant additives interrupts the bonding between epoxy resin and fibres. Thus, water easily penetrates into weakly bonded particleboard [21] in comparison with well bonded epoxy resin added particleboard.

3.3 Evaluation of flame retardant properties

A limited oxygen index value is a semi-qualitative indicator to measure the effectiveness of flame retardant properties, and thus it should be more than 26 in order to satisfy the need of being self-extinguishing [22]. Based on table 2, epoxy resin added particleboards with flame retardant additives showed high limited oxygen index values which are greater than 26 compared to control and particleboard with epoxy resin. When particleboard with epoxy resin and inorganic hydroxides was exposed to flame combustion, inorganic hydroxides decomposed into water to form a barrier and make

the combustion process harder and eventually, more oxygen was needed to propagate combustion [23]. This exhibits that both inorganic hydroxides are highly potential flame retardant additives.

3.4 Evaluation of thermal properties

The influences of epoxy resin and flame retardant additives on thermal properties of oil palm trunk particleboards were analysed by thermogravimetric analysis. The thermogravimetric diagram of samples is shown in figure 2, where the thermogravimetric curves exhibit three main weight loss stages. The initial stage from 100 to 250°C, which is followed by a slight drop in weight, attributed to the evaporation of water and easily volatile materials, accompanied by thermal decomposition of hemicellulose, carbon dioxide and water [24] of samples at the second stage with the temperature ranging from 250 to 400°C. Particleboard with epoxy resin and flame retardant additives resulted in a higher onset temperature of weight loss in the second stage than epoxy resin added particleboard and control particleboard. The increase in the onset degradation temperature is due to the inclusion of polar hydroxyl and carbonyl groups which leads to an increase in the bonding strength between resin and fibre chains with inorganic hydroxides which act as barriers; consequently more energy is needed for degradation. The final degradation stage

Figure 2. (a) Thermogravimetric and (b) derivative thermogravimetric curves of oil palm trunk particleboards.

Table 2. Limited oxygen index of oil palm trunk particleboards.

Types of boards	Limited oxygen index (%)
Control	24.45 (0.15)
10% epoxy resin	25.25 (0.14)
10% epoxy resin + 6% $\text{Al}(\text{OH})_3$	28.55 (0.16)
10% epoxy resin + 6% $\text{Mg}(\text{OH})_2$	27.95 (0.15)

Values in parentheses are standard deviation.

is attributed to ash formation where the samples decompose almost completely. Control particleboard had the lowest onset temperature at each stage which indicates that it is unable to withstand high temperature or possess weak thermal stability. From figure 2, it can be concluded that addition of flame retardant additives, namely aluminium hydroxide and magnesium hydroxide, to epoxy resin added particleboard significantly improved the thermal stability.

3.5 Evaluation of functional group

The functional group was analysed by Fourier transform infrared (FTIR) spectroscopy to observe differences between control particleboards and the manufactured particleboards with addition of epoxy resin and 6% of inorganic hydroxides. The FTIR spectra in figure 3 show the absorbance of the samples at various wavenumbers. The functional groups of

Figure 3. FTIR spectra of oil palm trunk particleboards. (A) Control board, (B) board with 10% epoxy resin, (C) board with 10% epoxy resin + 6% $\text{Al}(\text{OH})_3$ and (D) board with 10% epoxy resin + 6% $\text{Mg}(\text{OH})_2$.

the board samples were almost the same, however there was minimal difference in the peaks of all the board samples. The possible reaction between oil palm fibre and epoxy resin is shown in figure 4.

Based on figure 3, the intensities of the absorption band at a peak from 3400 to 3500 cm^{-1} in the board samples were corresponding to the OH stretching. The intensity of this peak becomes higher with addition of epoxy resin and flame retardant additives which indicates the presence of more OH stretching. A peak at 2900 cm^{-1} showed the presence of $\text{C}=\text{O}$ vibrating bands of the carboxylic group in hemicellulose [5]. There is a significantly different peak at around 1755 cm^{-1} which indicated the presence of the $\text{C}=\text{O}$ stretching absorption band contributed by the carbonyl group of epoxy resin while the peak at around 1650 cm^{-1} indicates the stretching of $\text{C}=\text{O}$ in the amide group [25]. Particleboards with epoxy resin and flame retardant additives exhibit a sharp peak at 1250 cm^{-1} which indicates the presence of aromatic ether from epoxy resin in the samples [26], while a peak around 1400 cm^{-1} denotes the presence of hemicellulose and arabinoxylan [25].

3.6 Evaluation of crystalline properties

X-ray diffraction (XRD) analysis was conducted to evaluate the crystallinity of the oil palm trunk particleboards with and without additions of epoxy resin and flame retardant additives. Figure 5 shows the diffraction patterns obtained for control, epoxy resin added, epoxy resin and aluminium hydroxide added and epoxy resin and magnesium hydroxide added particleboard. It exhibited a major peak intensity around $2\theta = 16^\circ$ and a secondary peak around $2\theta = 22^\circ$.

The crystallinity index was determined according to Segal *et al* [27] for all the oil palm trunk particleboards and the results are summarized in table 3. Control particleboard has the highest crystallinity index and it decreased slightly with additions of epoxy resin and flame retardant additives. This observation might be due to the existence of flame retardant additives which deposited only on the surface of fibres and

Figure 4. Possible reaction between oil palm fibre and epoxy resin.

Figure 5. XRD patterns of the oil palm trunk particleboards.

did not affect the crystalline region of the oil palm fibres. This statement is supported by the SEM observation in section 3.7.

3.7 Evaluation of morphological properties

Figure 6 illustrates the scanning electron microscopy (SEM) micrograph of control particleboard and the particleboard

Table 3. Crystallinity indices of the oil palm trunk particleboards.

Types of boards	Crystallinity (%)
Control	34.11
10% epoxy resin	32.68
10% epoxy resin + 6% Al(OH) ₃	30.47
10% epoxy resin + 6% Mg(OH) ₂	31.49

Figure 6. SEM micrograph showing the cross section view of (a) control particleboard and (b) particleboard with epoxy resin and flame retardant additives made from oil palm trunk.

Figure 7. EDXA spectra of cross-section of oil palm trunk particleboard with their elemental compositions.

sample with 10% epoxy resin and 6% flame retardant additives. Figure 6a clearly illustrates the compressed cell walls, fibres and parenchyma ground tissue due to the pressing method during manufacturing of particleboard and even a small amount of silica was detected. These cells provide good bonding between the fibres and improve the interfacial adhesion as no adhesive was used when producing the control board. A granule like substance found on the cell wall of fibre represents the epoxy resin which acted as a binder as shown

in figure 6b. It also showed that aluminium hydroxide and magnesium hydroxide act as flame retardant additives well dispersed and deposited on the surface of fibres and parenchymatic cells.

3.8 Evaluation of elemental composition properties

Figure 7 shows the elemental composition present in the oil palm trunk particleboards analysed by a SEM with

energy dispersive X-ray analysis (EDXA). Carbon, oxygen and silica contents were detected in all oil palm trunk particleboards. A small increase in the carbon percentage of epoxy resin added particleboard compared to control particleboard was due to the loss of some volatile nitrogen, chlorine, potassium and oxygen compounds during hot pressing [28], while the oxygen content by weight percentage of all samples was almost the same. Control board had the highest silica content because it does not contain any resin and flame retardant chemicals that will alter its cell wall structure. The presence of aluminium as shown in figure 7c and magnesium as shown in figure 7d is in agreement with the presence of small granules shown in figure 6b.

4. Conclusions

The properties of control particleboard, particleboard with addition of 10% epoxy resin and particleboard with addition of 10% epoxy resin and 6% flame retardant additives, namely aluminium hydroxide and magnesium hydroxide, were investigated. Based on the result, epoxy resin was found to be a potential binder as it improved physical and mechanical properties of oil palm trunk particleboard. Besides, the mechanical properties of all types of boards have met the minimum requirement of Japanese Industrial Standard for Type 8. Particleboard with 10% epoxy resin and aluminium hydroxide or magnesium hydroxide showed a significant improvement in flame retardant and thermal stability properties. The results indicated that particleboard with epoxy resin and flame retardant additives had improved mechanical, physical, thermal and flame retardant properties compared to control oil palm trunk particleboard. Overall results showed that epoxy resin as a function of a binder and aluminium hydroxide and magnesium hydroxide as a function of additives have the potential to be incorporated into particleboard to improve flame retardant properties.

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