Flammability and Mechanical Properties of Arenga Pinnata Fibre/PET Reinforced Epoxy for Fire Retardant Composites

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Abstract. The aim of this paper is to study the effect of magnesium hydroxide burning rate, mechanical properties and surface morphology analysis of Arenga pinnata fibre hybrid with polyester yarn (APF/PET) reinforced epoxy composite with the addition of magnesium hydroxide as flame retardant. The composites were prepared with different amount of fibres (i.e. 0%, 20%, 35% and 50% by weight percent) and two constant material of 5% which are the magnesium hydroxide and the polyester yarn with epoxy resin as the matrix. With the incorporation of the flame retardant the horizontal burning rate of APF/PET reinforced epoxy decreased with the amount of 35% APF. The fibre loading increased to 50% as the mechanical properties of the hybrid composite decreased which due to the lack of interfacial bonding between the fibres and matrix. Surface morphology studies through scanning electron microscopy showed the similar distribution of Arenga pinnata fibres and matrix with less adhesion which reduce the mechanical properties of the hybrid composites.

1. Introduction
As years passes by, people continue to design and produce material science product that are in green technology though the development of natural fibre as the reinforcement for composite material. The use of sisal, bamboo, kenaf, cotton, jute, coir, ramie and sugar palm fibre as natural resources. This eco-friendly, lightweight and low costs resources help engineers to develop a high performance of product in wide range of application [1-2]. The natural fibres serve as an alternative way to change the use of synthetic fibres that are non-biodegradable and expensive. Hybrid composite are known to be applied on fibres especially natural fibre. Hybrid composite are known as the composites that made from one or more reinforcement fibres. The combination of one or more reinforcement fibre are because to increase the material mechanical performance such as strength and impact resistance with a reasonable price. The hybridization of one natural fibres or synthetic fibre with a natural one can improve the physical, mechanical and thermal properties of composites. The reinforcing materials provide a synergetic effect called a “hybrid effect” that determines new or enhanced properties [3].

In the past, extensive studies had been done on the preparation and characterization of thermosetting and thermoplastic composites material reinforced with the most commonly used fibres with or without treatment for example alkaline solution. The solution can change the surface wettability of the fibre making the natural fibre mechanical and physical properties to change. As the previous research, as the fibre content increases in accordance with the mixture rule, the strength and
modulus of longitudinal composite in tensile and flexural loading also increase [3]. One of the most valuable resourceful engineering plastics that use to make the soft drink bottles is the Polyethylene Terephthalate (PET). Since it is highly resistance to atmospheric and biological agents, PET also raise the environmental issues of increasing in wastes. Therefore, the fundamental of finding a simple a simple and economic way to recycle the PET waste is an important practice for sustainable recycling and contributes to the conservation of raw petrochemical. Beside that polyester are also made from raw PET, which to make polyester yarn and then clothing.

2. Experimental

2.1. Material
Arenga Pinnata fibres or sugar palm were obtained from Negeri Sembilan, Malaysia. The fibre was comb, free from untangled and cut into 200 mm. Four fibre contents of 0, 20, 35, and 50% by weight were studied in this study. The polyester yarn and magnesium hydroxide was prepared into 5 wt% as the constant. Magnesium hydroxide was supplied from R&M Chemicals and the epoxy resin w supplied by Miracon (M) Sdn. Bhd.

| Composition | APF (wt%) | PET yarn (wt%) | Mg(OH)₂ (wt%) | Epoxy (wt%) |
|-------------|-----------|----------------|---------------|-------------|
| C 1         | -         | 5              | 5             | 90          |
| CA          | 20        | 5              | 5             | 70          |
| CB          | 35        | 5              | 5             | 55          |
| CC          | 50        | 5              | 5             | 40          |

2.2. Fabrication of Composites
The epoxy and hardener were prepared using ratio of 2:1 and then the Mg (OH)₂ was added into the resin. The Arenga Pinnata fibre and polyester yarn are lay in form of intermingled continuous fibres. Before laying up the fibre into the mould, the transparent plastic is prepared at the bottom of the mould. The mixtures were then poured over the fibres and distributed completely.

Next, another transparent plastic was placed on the unfinished composited plate and was pressed and spread using jointing knife to removed bubbles. Then another same shape mould steel was place on the composite as weighing. The curing process was carried out at room temperature for about 24 hours. After the hybrid composites samples was prepared twice based on horizontal burning test and tensile test and it was cut to the required size using an electric saw. Finally, the test specimen was polished using a sand paper. The grit size of the sand paper was characterized by supplier’s code ‘0’, which indicates very fin. Similar approaches were used for woven and short/chopped random fibre composites.

Figure 1. Arrangement of APF and PET yarn with epoxy in the metal mould.
2.3. **Horizontal Burning Test**
All composite specimens were cut into the size of rectangular shape. The tests were carried out by following the ASTM D635 standard. The flame retardants hybrid composites produced were cut to test samples with dimensional of 125 mm x 13 mm x 3 mm. The test samples were supported horizontally at one end as shown in figure 1. On the other free end, it is exposed to a specified gas flame for 30 s time and extent of burning are measured and reported if the specimens do not burn. An average burning rate is reported for a material if it burns to the 100 mm mark from the ignited end.

2.4. **Tensile Test**
Tensile test on fire retardant hybrid composites was determined according to ASTM D3039 standard. The measurements were done using Instron universal tensile machine (Model 5567) at a speed of 5mm/min at room temperature. The test specimens were positioned vertically in the grips of the testing machine. The elongation of the specimen was continued until the rupture point of the specimen was determined. The tensile strength and modulus were then determined by the computerized system.

2.5. **Surface Morphology Analysis**
A study on the surface morphology of the specimen after undergoing tensile test was carried out using the Scanning Electron Microscope (SEM). The specimens were cut into 2 cm by 2 cm of size before placing it on a stainless-steel plate for coating with a gold adhesive. The gold coating was used in ordered to have a clear view of the surface during scanning process. An imaged of specimen fracture surface were analysed.

3. **Results and Discussions**

3.1. **Horizontal Burning Rate**
Each specimen composition was tested on the horizontal burning test according to the ASTM D635 standard. The fire was put to stop when it reached the 75mm mark for each specimen. From figure 2, CB has the lowest burning rate which is 13.25 mm/min. The flame takes longer time to propagate along the CB specimen and at the same time produce char. The production of char is the reaction of g(OH)$_2$ during burning in front of the flame. Charring is known as a chemical process of incomplete combustion of certain solid when subjected to high heat. Thus, by the action of heat, charring removes hydrogen and oxygen, so that only carbon remained in the char. Besides that, as an acid and halogen-free flame retardant, Mg(OH)$_2$, released water of hydration when it decomposed endothermically that helps to in the flame retarding action. Moreover, content of moisture in APF contribute to the flame retarding [4]. In conclusion, CB (35%) have better composition of *Arenga Pinnata* fibre loading include 5 wt% of PET yarn and Mg(OH)$_2$ reinforced epoxy resin.

3.2. **Tensile Properties**
Tensile test was conducted on all the samples composition and data were collected from the test for a three times test of each specimens.

Table 2 shows the tensile properties of APF for different volume content and control samples in average value. From the table, for the control samples it shows that samples C1 (0%APF/5%PET/5% Mg(OH)$_2$ /90%Epoxy) has the highest tensile strength which are 10.82 N/mm$^2$. Meanwhile for the samples with fibres CB (35%APF/5%PET/5% Mg(OH)$_2$ 55%Epoxy) has the highest tensile strength which are 9.69 N/mm$^2$. It shows that on figure 3 the tensile decreases when the fibre content increases to 50%. The amount of fibre content caused the epoxy resin not fully dispersed between the fibre and not fully bind with the fibre when it is already cured where fibre pull-out holes left on the matrix surface. This can be supported by Mukhtar et al. (2016) where having poor adhesion at the interface will weaken the fibre and consequently yield poor mechanical properties that will render the material incapacitated in many applications [6].
Figure 2. Graph of horizontal burning test of each specimen.

Table 2. Tensile properties of each specimen.

| Sample Fibre Composition | Tensile Strength (N/mm²) | Tensile Modulus (MPa) | Max. Load (N) | Energy (J) |
|--------------------------|--------------------------|-----------------------|---------------|------------|
| C1                       | 10.82                    | 324.704               | 487           | 1.747      |
| CA                       | 7.347                    | 63.310                | 203           | 0.045      |
| CB                       | 9.694                    | 145.416               | 1047          | 2.076      |
| CC                       | 6.917                    | 103.794               | 747           | 7.025      |

Figure 3. Graph of tensile strength.

Graph on figure 4 presents the tensile modulus of APF/PET reinforced epoxy composite for different fibre content. For this test, the elastic modulus of specimens is decreasing linearly, which control specimen C1 (0%) has the highest tensile modulus, 324.704 MPa. Followed by specimen CA (20%), CB (35%) and CC (50%) with the value of 165.200, 145.416 and 103.794 MPa. The elasticity
of the specimen increases when there is no addition APF but and only reinforced with PET yarn and reduced when APF is added. From the previous study again by Sapuan and Bachtiar (2010), it was more difficult to obtain consolidation of the composite during the fabrication process thus reducing the extent of fibre wetting when the fibre is more than the matrix in terms of high fibre content [7]. Beside this is also may be due to that the fibre have not treated by alkaline solution (sodium hydroxide) to increase the interfacial bonding between the matrix.

3.3. Morphological Analysis

Surface morphology were obtained after all specimens has undergoes tensile test by using scanning electron microscope (SEM) and captured the images at the fracture cross-section area. The side surface where fracture occurs after ultimate stress can be described as the result of tensile damaged. According to Thomas Jolivet et al. (2013) there are different steps of micro-damage in composite material as it occurs earlier or later which depends on the type and direction of the reinforcement as well as the stress applied on it [8]. Figure 5 show the examples of tensile damaged on the composite surface where fracture occurred. However, during lay-up process or fabrication of the composites also might cause defects to occur. The possibility of composite failure will increase as there is various of defects in the composites during fabrication [9].

![Tensile Modulus Graph](image)

**Figure 4.** Graph of tensile modulus.

![Morphological Analysis Diagram](image)

**Figure 5.** Example of tensile damage on surface fracture.
Figure 6. SEM micrograph for (a) 20\%, (b) 35\%, and (c) 50\% of fire retardant hybrid composite.

From figure 6 (b) and (c) it shows the lack of fibre matrix interfacial bonding on the surface which lead to the fibre breakage. When the composite continuously put under load pressure, the fibre breakage will occur during rupture as there is less interfacial bonding of fibre and matrix. On this experiment it clearly shown that the adhesion of the fibre matrix are more common defects occur if the fibre is not treated using alkali or seawater thus more fibre breakage can be seen later after tensile test or other load test upon it. Consequently, the poor adhesion followed by fibre breakage will reduced the tensile properties of the specimens. Arenga Pinnata fibre have few drawback as natural fibre the most worrisome drawback is the lack of interfacial adhesion between the filler and matrix [6, 10, 11].
However, the hydrogen bonding and electrostatic also might cause the interaction to be weak which is the Van de Waals [12].

As for (a) on figure 6 the matrix cleavage breakage can be found on the surface since less of APF loading in the matrix. As mention by Thomas Jolivet et al. (2013), interface failure is the first damage that requires low energy consumption while the last stage is fibre breakage which requires more significant energy level on the same damaged process [8]. Matrix cleavage breakage started to form at the area that has low strength where there is also fibre-matrix interfacial bonding which is 20% specimen that has more bonding of matrix and PET yarn fibre that are well trapped.

Next from figure 7, the control specimen (0%) have rich resin zone has interface breakage however there is PET yarn fibre that well trapped but also has holes left on the surface due to fibre breakage during rupture for other specimens that has rich resin zone. Deformation on this zone cause the swelling of the resin and will subjected to larger stress which then produce more matrix cleavage breakage or interface breakage after tensile damaged. However, with the reinforcement of PET yarn fibre, it reduces the weak interfaces in surrounding since the fibre also provides mechanical strength in small amount. PET is known to have strong, resistant to stretching and shrinking properties.

Lastly, defect of fibre pull-out in the composite which leaving holes on the matrix as shown in figure 8. This happen due to the poor of fibre matrix interfacial adhesion. During tensile test, some of the APF does not break after the specimen start to form necking until rupture. Starting of failure mode, the tensile damage on the composite are increasing as the surface became more vulnerable when the composites need to overcome the load applied on it and thus creating stress and strain on the fibre and matrix which lead to the surface defects. For instance, the tensile failure has affect the APF adhesion in the resin matrix during pulling as the fibre become less adhere to the matrix surface walls.
4. Conclusions
As we know, the natural resource has increase in demand because of its mechanical properties, lightweight, durability, eco-friendly and low cost of production. However, natural fibre reinforced polymer composite is prone to fire, water absorption, reduced in mechanical strength. So, this project has showed a few test and experiment using magnesium hydroxide and hybrid of APF and PET. The addition of magnesium hydroxide showed a better flame retarding for 0% and 35% of the hybrid composites and with PET yarn reinforced the mechanical properties decrease when reaches 50% of APF loading. This can be supported from the poor interfacial adhesion of APF and matrix.

5. Recommendation
The flammability and mechanical properties of hybridization of APF/PET can be improved by increase the amount of magnesium hydroxide and give alkaline treatment for the natural fibre for a better mixture of fibre and epoxy matrix. Besides that, the LOI and TGA of the composite should also be investigate for future references.

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