Physical characterization of carbon fiber reinforced composite with blended epoxidized Jatropha oil (EJO) resin.

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Abstract. Jatropha oil is an inedible oil that is mainly used for bio-fuel in cars, lorries, machineries and jets as it provides clean combustion, reduced carbon dioxide emission and additional power with unlimited renewable resource; jatropha plant. Several studies have been carried out by researchers to diversify jatropha oil’s uses such as lubrication, bio-fuel, traditional medicine etc. This time, jatropha oil was being used as matrix in composite structure by epoxidizing jatropha oil (EJO) itself. This paperwork will present about the physical characterization (density, visual assessment, fiber volume fraction) of EJO blended with synthetic epoxy as resin in carbon fiber reinforced composite panel by vacuum assisted resin transfer moulding VARTM or vacuum infusion technique. It is found that by blending EJO with synthetic epoxy, their physical characteristics are almost the same and comparable except the color of blended EJO was a bit yellowish and matte. Density of composite panel with blended EJO was 4% lower than synthetic epoxy which contributed to lighter materials that could be built and the surface morphology of both are identical.

1. Introduction

Thermoset or epoxy derived from petroleum is very synonym in composite sector, is used as resin to bond fibers as one body in order to resist a certain amount of applied loading. It is thermosetting polymer which are formed starting from liquid state into solid state by an irreversible curing process. Recently bio-based composite is very popular in composite research world. They used natural fiber as reinforcement and natural thermoplastic or natural thermoset as matrix. This bio-composite attracting many attention from manufacturers and researchers as the finding are interesting and could be highly optimized by various process. More than 60% of global production of epoxy were used in coating industry, total balance of 40%were used in adhesive/assembly parts, assembly concretes, sandwich panels for aircraft and automobiles [1]. Price of petroleum is one of the main factor besides its
footprints towards environment. As the price of petroleum is not stable, it is a good leap to find alternative, environmental-friendly and renewable resources for an alternative replacement of epoxy.

One of an alternating replacement of epoxy is bio-based epoxy derived from crude jatropha oil, local unwanted plant in Malaysia as its oil is inedible. For the moment, jatropha oil is used as bio-fuel in cars, lorries machineries and jets as it burns clean combustion, emitted less carbon dioxide, and provide additional power with unlimited renewable resources [2]. There were several other natural epoxidized oils such as soya oil, cottonseed oil, algae oil, karanja oil, pine oil, corn oil, palm oil and sugar can oil. Epoxidation process differed from each other depend on nature of natural oil, including types of applications from aircraft panel to food packaging products [3-4].

In this paper, an investigation was carried out on composite panel made of carbon fiber with blended Bio-based epoxy derived from crude jatropha oil. The purpose of this research work is to improve the amount of renewable materials in aircraft interior parts as more than 80% of aircraft body made from composite materials. The comparison study of physical appearance, physical properties, density, thickness and fiber volume fraction were carried out between composite panel with 100% synthetic epoxy and composite panel with 18% blended Bio-epoxy.

2. Experimental

2.1 Fabrication and Materials of Composite Panel.
Two composite panels were fabricated in this experiment. The first one was using 100 % synthetic epoxy which would be named 0 % bio-epoxy panel. The second one was using epoxy resin blended with 18% bio-epoxy by weight percentage. Synthetic epoxy brand was Epoximate 100 with slow hardener 103 with instruction ratio of 28:1. 10 layers of 2 × 2 twill weave carbon fiber 3 K 200 gsm were used for each panel. Carbon fibers and synthetic epoxy were supplied by Mechasolve Engineering, Malaysia, and Bio-epoxy was supplied by in-laboratory collaboration of Faculty of Engineering University Putra Malaysia (UPM) with Aerospave Malaysian Innovative Centre (AMIC) Malaysia. Bio-epoxy was derived from crude Jatropha Oil supplied by Bionas Malaysia Sdn. Bhd.

Dimension of composite panel fabricated by VARTM was 300 mm × 300 mm × 2 mm (width × length × thickness). The composite panel 0% Bio-epoxy was the control specimen and composite panel with 18% Bio-epoxy was the evaluated specimen.

A flat mold was used as base surface for fabricating the composite panel. Release agent was applied onto the surface of flat mold for easy release. Then, 10 layers of carbon fiber were stacked and arranged. Then peel ply, media distributor, EnkaFusion mat, and bagging plastic were organized in order as in figure 1 with one inlet for resin, and one outlet for vacuum. All these materials were secured by sealant tape surrounding them to trap air and resin from escape. Once all were settled, air was sucked out by vacuum pump until it reached around 12 mbar. Afterwards, inlet pipe was open to let the resin infused through the carbon fiber, media distributor and so on. Inlet pipe was closed once almost all resin have been wetted. The set up was let to stay around and cure at room temperature 3 to 4 hours before the vacuum pump was shut off as in figure 2. Finally after 24 hours in room temperature, the composite panel was cured and carbon fiber composite panel was retrieved from the set up.
2.2 Burnt off test.
For burnt off test, specimens were prepared by cutting them into 25 mm × 25 mm according to ASTM D3171-09. Aluminium crucibles were used to support the specimens during the burning process in an electrical muffled furnace at 550 °C for two hours. Weight of the specimens and crucibles were measured before and after burning process. Specimens’ density were measured by a water displacement density calculator.

Rule of Mixture was use to compare the fiber volume fraction of specimen theoretically by using Equation (1) and experimentally measuring the weight of specimen obtained by this burnt off test.

\[
V_f = \left( \frac{M_f}{M_i} \times 100 \times \frac{\rho_c}{\rho_f} \right)
\]

Where;
- \(M_i\) : initial mass of the specimen
- \(M_f\) : final mass of specimen after combustion
- \(\rho_c\) : the density of the specimen
\[ \rho_f : \text{density of the fiber reinforcement} \]

Theoretical constituent content of composite panel could be calculated by using following Rule of Mixture, Equation (2):

\[ V_{cp} = V_f + V_r = 1 \text{cm}^3 \]  

Where;
- \( V_{cp} \): volume of composite panel
- \( V_f \): volume of fiber
- \( V_r \): volume of total resin

Density of composite panel could be calculated by using Equation (3):

\[ \rho_{cp} = \frac{M_{cp}}{V_{cp}} \]  

Where;
- \( \rho_{cp} \): density of composite panel
- \( M_{cp} \): mass of composite panel
- \( V_{cp} \): volume of composite panel

2.3 \textit{Bio-epoxy derived from crude Jatropha Oil.}

Through the process of addition, removal, stirring, and washing the mixture of crude jatropha oil with acids and amberlite, bio-epoxy derived from crude Jatropha oil [5]. In the Table 1 below, are presented some physical properties of crude Jatropha oil and its Bio-epoxy (Epoxidized Jatropha oil) that have been extracted.

| Physical Property          | Crude Jatropha Oil (CJO) | Bio-epoxy from CJO | Synthetic Epoxy |
|----------------------------|--------------------------|--------------------|-----------------|
| Appearance                 | Liquid (clear yellow color) | Liquid (yellow color) | Liquid (clear color) |
| Density (g/cm\(^3\))       | 0.89304                  | 1.298              | 1.1             |
| Specific Gravity           | 0.8948                   | 1.302              | 1.1             |
| Dynamic Viscosity at 25 \(^\circ\)C (cP) / (Pa.s) | 46.8 cP                  | 546 cP             | 650 cP          |
| Kinematic Viscosity (mm\(^2\)/s) | 52.41                   | 588                | -               |
| Epoxy Equivalent Weight (EEW) (gr/eq.) | 263.16                  | 328.95             | -               |
| Epoxy Value (eq/100 gm)    | 0.38                     | 0.30               | -               |

3. \textit{Results and discussion}

3.1 \textit{Fiber Volume Fraction.}

In figure 3, an example of specimen after burning process is shown. For the specimen with 18 \% Bio-epoxy, its fiber volume fraction was 42 \% compare to 47 \% for specimen with 0 \% Bio-epoxy. That was reduction of 5\% of fiber volume fraction. Theoretical values of fiber volume fraction for 18 \% Bio-epoxy and 0 \% Bio-epoxy were 60 \% and 75 \% respectively. Both show similar reduction trend in fiber volume fraction property. Despite the big gap between theoretical value and experimental value of fiber volume fraction, by achieving more than 40 \% of fiber volume fraction are considered good fiber volume fraction because of different type of woven and different type of resin and filler, would differ the volume fraction of composite structure [6-11].
3.2 Density and Thickness.
For the specimen with 18% Bio-epoxy, its density was 1.415 g/cm\(^3\), while density of specimen with 0% Bio-epoxy was 1.474 g/cm\(^3\). This decrement of density shows improvement in weight of part that blended with Bio-epoxy. This weight reduction of 4% in overall composite structure could bring a significant improvement impact in aerospace application as it could improve fuel efficiency since more than 50% of airplane materials are composite structure [12-13]. With the same amount of 10 layers carbon fibers, the thickness of the composite panel with 18% Bio-epoxy is slightly thicker by 0.22 mm compare to composite panel with 0% Bio-epoxy. This increment show blended Bio-epoxy used up more space in bonding with fiber carbon compare to synthetic epoxy. This is due to bio-epoxy is vegetable oil based, which contains many fatty acids which has small molecular chains compare to petrochemical based epoxy. The length of molecular chain contribute to weight of the cured epoxy [14]. Furthermore, fatty acids in bio-epoxy is known to be bulky and flexible thus slightly increasing the volume or thickness of the cured bio-epoxy [15]. Refer to figure 4 for result comparison of theoretical & experimental result for density and fiber volume fraction.

![Figure 3. Sample in crucible after burnt off test](image)

![Figure 4 Thickness, Density and Fiber volume fraction comparison between composite panel with 18% Bio-epoxy and 0% Bio-epoxy.](image)
3.3 Physical appearance.
For the specimen with 18% Bio-epoxy, the blended resin show translucent color compare to specimen with synthetic epoxy which shows transparent color. This is due to size of molecules and crystalline/amorphous structure inside the cured epoxy. Small size molecules will allow light to pass through it compare to bigger molecules which scatters the light away [16]. As the bio-epoxy becoming thickened, it is getting opaque as in figure 6. This is due to crystalline structure is higher in bio-epoxy compare to synthetic epoxy. As shown in figure 5, we could categorize the composite to be shining and reflective finishing and matte with less reflective finishing. Both finishing has advantage and disadvantage according to application such as controlling the thermal effects by sun radiation as used in NASA devices and equipment like garments, spacesuits etc. [17].

![Figure 5](image1.jpg)

**Figure 5** Specimens with 0% Bio-epoxy or synthetic epoxy (a), while (b) specimens with 18% blended Bio-epoxy.

![Figure 6](image2.jpg)

**Figure 6** 0% Bio-epoxy or synthetic epoxy (a) and 18% blended Bio-epoxy (b).

3.4 Microstructure from Scanning Electron Microscopy (SEM).
The surface characteristics of both composite panels were compared through scanning electron microscopy. Their cross sectional view of both panels presented shown in figure 7. The purpose of SEMs’ images were to compare 0% bio-epoxy with 18% bio-epoxy in term of the interfacial bonding
between fibers and resins, internal cracks or defects came from fabrication process. As images between both specimens were compared, it could be seen that both specimens were pretty much similar as no significant different could be seen. The interfacial bonding between carbon fibers and resin blended with 18% bio-epoxy indicated good interfacial properties. For the defects, almost no void occurred between fibers and resin for 18% bio-epoxy. Those fractured fibers and broken resins were resulted from cutting process in obtaining the cross sectional area. Apart from that, very little cracks due to fabrication process could be seen. The slight expansion of thickness for 18% bio-epoxy did not degrade the composite structure even though there are bio-epoxy content in the resin. Normally by adding impurities, fillers or other elements in the resin, there would be increment or decrement of surface morphology (such as porosity, opening planar network), defects (cracks and debonded fibre to resin) and performance itself [18-20].

The SEM images are important as both specimen show high similarity to ensure good performance in mechanical properties. These images also proved that the vacuum infusion method were qualified to be used with this new bio-epoxy.
4. Conclusion
From the investigation of physical characterization of carbon fiber reinforced with blended bio-epoxy derived from crude Jatropha oil, they were found that by adding 18% of bio-epoxy the density of composite panel was reduced by 4% that could lead to better fuel efficiency if it used as proper parts in aerospace application. The thickness was comparable as it differed only by 0.22mm and its fiber volume fraction also is comparable to synthetic epoxy. Both are fabricated by VARTM process and by adding bio-epoxy no sign of defects to change to other type of the fabrication process.

The appearance of composite panel with Bio-epoxy show translucent color which reflect less light compare to synthetic epoxy which is almost 100% clear. The interfacial bonding between fibers

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**Table**

| Specimen with blended 18 % Bio-epoxy | Specimen with 0 % Bio-epoxy |
|--------------------------------------|-----------------------------|
| ![Specimen with blended 18 % Bio-epoxy](image1) | ![Specimen with 0 % Bio-epoxy](image2) |
| ![Specimen with blended 18 % Bio-epoxy](image3a) | ![Specimen with 0 % Bio-epoxy](image3b) |
| ![Specimen with blended 18 % Bio-epoxy](image3c) | ![Specimen with 0 % Bio-epoxy](image3d) |
| ![Specimen with blended 18 % Bio-epoxy](image3e) | ![Specimen with 0 % Bio-epoxy](image3f) |

**Figure 7** (a,c,e) are cross sectional view of specimen with 0 % bio-epoxy while (b,d,f) are specimen with blended 18 % bio-epoxy. From top to bottom, image with different magnification.
and bio-resin were similar to fibers with synthetic epoxy as seen from cross sectional view in SEM.

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