EFFECTS OF DIFFERENT PRE-TREATMENTS ON THE PERFORMANCE OF KENAF FIBER REINFORCED ALUMINUM LAMINATES SANDWICH COMPOSITE

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ABSTRACT: Fiber metal laminate (FML) offers some superior mechanical properties as compared to either conventional polymer composites or high strength monolithic aluminum. Treatment of individual elements of composites is one of the effective methods to improve the performance of composites. This study focuses on the effects of different pre-treatments of fiber and metal sheet to the performance of metal laminate composite containing kenaf reinforced epoxy as the core composite. The kenaf fibers and aluminum sheet underwent different types of treatment to improve the interfacial adhesion. The results showed that the composites with surface roughened aluminum sheets gave higher values of flexural strength than those with alodine treated aluminum sheets. However, composites with alodine treated aluminum showed the highest impact strength, contributed by interfacial delamination as a result of less firm adhesion between Al sheet and composite core. The fiber metal laminate sandwich composites also showed improvement in water resistance as compared to the kenaf fiber reinforced composite, particularly those with alkaline treated fibers. In conclusion, surface treatments on the Al sheets and kenaf fibers were effective to improve the mechanical and water absorption properties of kenaf fiber reinforced aluminum laminates (KeRALL).

KEYWORDS: *Fiber Metal Laminate; KeRALL; Pre-Treatment; Kenaf Fiber; Aluminium Sheet*

1.0 INTRODUCTION

Fiber metal laminate (FML) refers to composite materials that consist of layers of metal sandwiching a fiber reinforced polymer core. FML offers some superior mechanical properties as compared with either conventional polymer composites or high strength monolithic aluminum alloys [1-4]. FML such as glass fiber reinforced aluminum laminate (GLARE), aramid fiber reinforced aluminum laminate (ARALL) and carbon fiber reinforced aluminum laminate (CARALL) are well-known examples of the materials. The two most commercially available FMLs are aramid fibers based ARALL and high strength glass fiber based GLARE1 [3-6]. Lately, study on natural fiber metal laminates has been fast moving forward. For examples, carbon-jute reinforced aluminum laminate (CAJRALL) and carbon-jute reinforced magnesium laminate (CAJRMALL) have been studied by Vasumathi and Murali [6] focusing on the potential of replacing carbon fiber with jute fiber. The study observed that the tensile and flexural stresses of CAJRALL and CAJRMAL were directly proportional, while the flexural modulus was inversely proportional to the number of layers. Meanwhile, Vieira et al. [7] studied the mechanical properties of sisal fiber reinforced aluminum laminate (SiRAL). It was observed that the tension and flexural strength increased by 132% and 430%, respectively in the SiRAL laminate as compared to sisal fiber reinforced composite (SFRC).

Kenaf (Hibiscus cannabinus L.) has been used as plant-derived reinforcement in composites in combination with resins such as polyester and epoxy [8]. Kenaf is a dicotyledon, which means its stalk has three main layers: an outer cortical (or ‘‘bast’’) tissue layer (phloem), an inner woody (called the ‘‘core’’) tissue layer (xylem), and thirdly, a thin central pith layer [9]. It is a lignocellulosic fiber that falls into the bast fiber category like jute, hemp, sisal, flax and ramie. On average, kenaf fibers contain cellulose (56–64 wt%), hemicellulose (21–35 wt%), lignin (8–14 wt%) and small amounts of extracts and ash [10]. A single fiber of kenaf can have a tensile strength and modulus as high as 11.9 GPa and 60 GPa, respectively [11]. Researchers revealed that treated kenaf fibers reinforced in epoxy managed to increase the flexural strength of the composite by about 36 %, while only 20% of increase was observed for the untreated fibers [12]. It was also reported that 6% NaOH yielded the optimum concentration of NaOH for the chemical treatment of kenaf fiber, as too high concentration of NaOH could damage the fibers, thus reduces fiber strength [13].

Meanwhile, for the metal sheets, the following features such as contamination free, high roughness, good wettability as well as mechanically and hydrolytically stable surface can be obtained from the treatment of the substrate surface [14]. Park et al. [15] claimed that mechanical abrasion as an essential preliminary preparation step in the multi-stage schedules and effective to produce macro-roughened surface, different roughness levels of the surface textures and to remove an unwanted oxide layer on the metal substrate. Abrasive scrubbing by using sand paper is typically applied as mechanical treatment against the substrate surface. It was reported that mechanical treatment introduced physico-chemical changes, which yielded a wettable surface and modified the surface topography, such as produced a macro-roughened surface [16]. Chemical treatment of aluminum surfaces will decrease in alkaline solutions or organic solvents and subsequently etch in aqueous chromic–sulphuric acid solutions can produce a good adhesion between aluminum sheet and resin [5].

Although some researches on the effects of pre-treatment to FML have been performed [5, 17-18], the effects of fiber and metal sheet pre-treatment in natural fiber metal laminate composites still have not been studied. In the present work, non-woven mat of kenaf is used as the reinforcement in the composite core of newly developed kenaf fiber reinforced aluminum laminates (KeRALL) via compression method. The influences of pre-treatments of metal sheet and kenaf fiber towards the performance of KeRALL are studied in order to obtain high strength to weight ratio of KeRALL. Compression method has been applied because of the main advantages of this process are low fiber slow destruction and high fabrication speed [19]. Moreover, damage behavior and fractography analysis are performed for further evaluation on the effectiveness of the fiber and metal surface pre-treatment. Finally, performance of water resistivity of KeRALL is also revealed.

2.0 EXPERIMENTAL

2.1 Materials

In this study, kenaf bast fiber (KF) was purchased from Innovative Pultrusion Sdn. Bhd. Malaysia and received in a form of non-woven mat with a surface density of 800 g/m^2. Kenaf fiber will be acted as reinforcement for composite part of FML sandwiches. As for matrices, epoxy resin (EPO DM 15 (F3) – A) and hardener (EPO DM 15 (F3) – B) were used. The ratio of epoxy and hardener used was 5:1.

2.2 Fiber Treatment

For fiber treatment process, the raw KF mat was alkalized using a 5% sodium hydroxide (NaOH) solution to remove impurities on fiber surface. The alkalization process was carried out for 48 hours by immersing the raw KF mat in the NaOH solution. The mat was then rinsed with distilled water 5 times followed by drying at 70°C for 24 hours in the oven.

2.3 Metal Sheet Treatment

The aluminum sheets (2024-T3) with thickness of 0.5 mm were used for the protective layers in FML. Two types of treatments were carried out on the Al sheets such as mechanical and chemical (alodine) treatments. For the former, the Al sheets were sanded in one direction using 60-grit sandpaper. For the latter, the Al sheets were immersed in alodine 1201 solution for 5 minutes to perform alodining etching process. In advance, the Al sheets were sanded with sand paper of 100-grit and cleaned with acetone to ensure the effectiveness of alodining process. Next, the sheets were rinsed with water and dried at 50°C for 1 hour. Finally, the treated Al sheets underwent water break test through water spillage to ensure the metal surface were totally clean, uniform and free from oily surface. Table 1 shows the designation used for the kenaf fiber reinforced aluminum laminates (KeRALL) and kenaf fiber reinforced composites (KFRC) in this study.

Table 1: Designation for KeRALL and KFRC samples

Sample		A	B
		Kenaf fibers (KF)	Al sheets
KeRALL	A1B1	Untreated	Mechanical abrasion
	A1B2	Untreated	Alodine treatment
	A2B1	Treated	Mechanical abrasion
	A2B2	Treated	Alodine treatment
KFRC		Treated	None

2.4 KeRALL and KFRC Fabrication

All samples were fabricated via warm compression molding method using a hydraulic hot press (GOTECH) at 80 °C. This temperature was selected by considering the glass transition temperature of the epoxy resin as determined by differential scanning calorimetry (DSC). The pressure of 1000 psi (65 kg/cm^2) was applied with a holding time of 15 minutes. A square steel mold with a dimension of 150 x 150 x 4 mm (length x width x thickness) was used. The mould was applied with release agent to ease the re-molding process. For the kenaf fiber reinforced aluminum laminates (KeRALL), a kenaf/epoxy core was sandwiched by two aluminum alloy sheets. As for pristine KFRC, it contained a fiber volume fraction of 20 wt%.

2.5 Specimen Preparation, Testing and Analysis

For specimen preparation, mechanical cutting equipment was used to cut the specimen according to the American Society for Testing and Materials (ASTM) standards. The samples were tested for different behaviors such as flexural, impact and water absorption. Flexural and impact tests were performed to evaluate the mechanical properties of the KeRALL as compared to the KFRC. The flexural test was carried out by using GOTECH A1-7000-LA 50 kN at 23 ± 2°C and 50 ± 5% relative humidity in accordance to ASTM D790. Meanwhile, the impact test was performed using Instron - CEAST 9050 Impact Pendulum with pendulum energy of 2.75 J (KeRALL) and 0.5 J (KFRC) according to ASTM D256 for edgewise notched Izod impact test. Moreover, water absorption test (ASTM D 570) was also performed at 30°C for 20 days in water bath, to evaluate the improvement in water resistivity of KeRALL. Microstructural analysis was performed using optical stereomicroscope (Leica EZ4D) and scanning electron microscope (SEM) in order to study breakage behavior as well as for fractography analysis.

3.0 RESULTS AND DISCUSSION

3.1 Structural Determination of KeRALL and KFRC

A cross-sectional image of KeRALL is shown in Figure 1. The laminated fiber metals are characterized based on their metal volume fraction (MVF) which is defined in the following equation [20]:

$$MVF = \frac{\sum_{1}^{n} t_{\mathrm{metal}}}{t_{\mathrm{laminate}}} \tag{1}$$

where t_{metal} = thickness of each metal layer, n = number of the metal layers and t_{laminate} is the thickness of the total laminate.

From Figure 1, the value of MVF for KeRALL is 0.25 indicating a predominantly composite fraction available in KeRALL. If MVF value is equal to zero therefore, it is referring to a full composite while if MVF value roughly 1 then, it is almost monolithic metal. Moreover, Table 2 shows the average volume fraction of composite (kenaf and epoxy) and Al sheets in KeRALL, which are 77% and 23%, respectively.

Table 2: Volume fraction for KeRALL and KFRC samples

Sample	Al sheet (%)	Epoxy resin (%)	Kenaf fiber (%)
KeRALL	23	54	23
KFRC	-	80	20

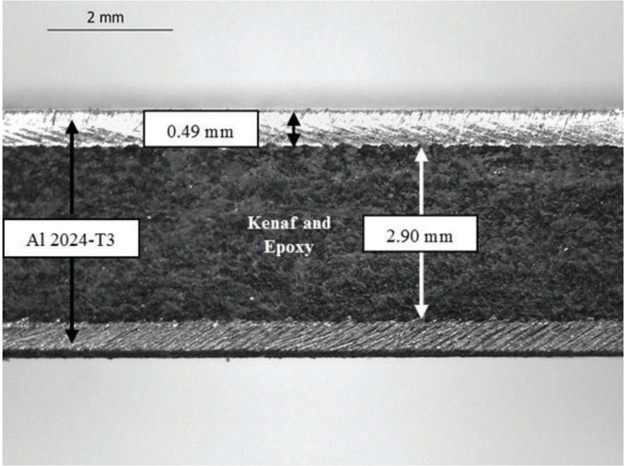


Figure 1: Cross sectional area of KeRALL

Densities of KeRALL, KFRC, kenaf fiber and Al sheet (2024-T3) are shown in Table 3. The result shows that the KeRALL density is in the range of 1.3 - 1.5g/cm³ which is about 50% lower as compared to the Al sheet.

Table 3: Densities of KeRALL, KFRC, kenaf fiber and Al sheet

Sample	KeRALL	KFRC	Kenaf fiber	Al sheet
Density (g/cm³)	1.3 – 1.5	1.0 – 1.2	1.4	2.7

3.2 Mechanical Properties

Figure 2 shows the flexural strength for the KeRALL and KFRC composites. KeRALL shows the great improvement in flexural strength as compared to KFRC with as much as 283% of increments. Addition of Al layers significantly increases the flexural strength by about 3-fold increase. The increase is due to the high mechanical properties of the Al alloys that affect the energy absorption of FML as reported by Vlot and Gunnink [20]. The Al layers in FML also contribute significantly to yielding of the composite at high load, a stable deformation before the break, high residual strength, fatigue performance, excellent blunt notch resistance and resistance to short cracks as reported by other researchers [21].

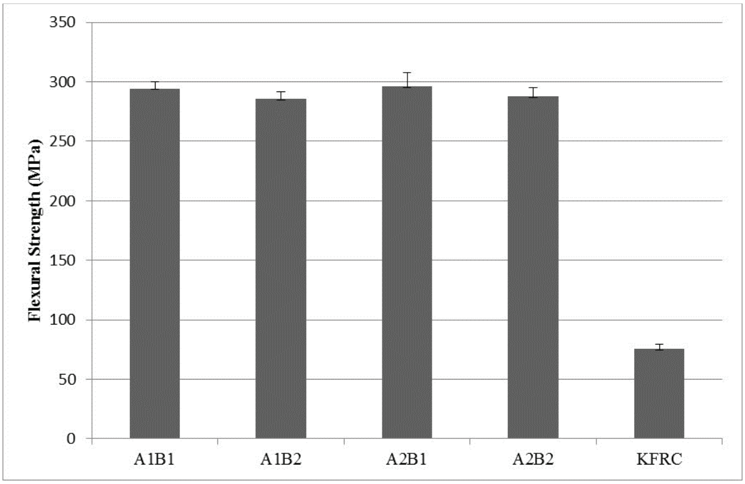
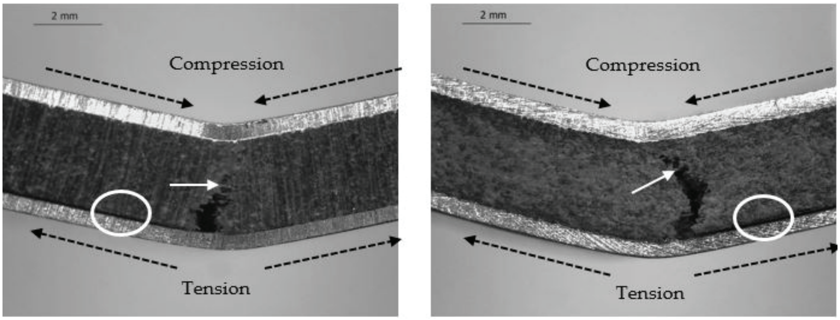


Figure 2: Flexural strength of KeRALL as compared to KFRC

In general, the composites with mechanical treated Al sheets show better flexural strength compared to those with alodine treated Al sheets. The highest flexural strength is shown by A2B1 (treated fiber, mechanical abrasion) at 296 MPa, followed by A1B1 (untreated fiber mechanical abrasion) at 294 MPa. Meanwhile, composites with alodine treated Al sheets show relatively low flexural strength at 288 MPa (alodine treatment, treated fiber) and 285 MPa (alodine treatment, untreated fiber). There are about 2-3% differences in flexural strength between composites with mechanical and chemical treated Al sheets. The results indicate that metal surface treatment by mechanical abrasion is more effective than alodine treatment to increase the flexural strength of KeRALL laminates. The increase is possibly due to the increase in adhesion level of the Al sheets. Grit blasting or other mechanical abrasion methods are recognized for providing a useful increase in initial adhesion levels [16].

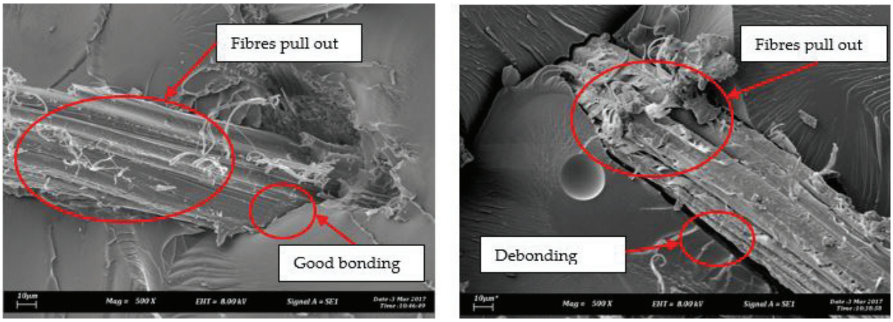


(a) (b)

Figure 3: Load distribution and failure mode upon flexural test on various types of KeRALL specimens: (a) A2B1 and (b) A2B2 at 8x magnification

Figures 3(a) and (b) show images of load distribution and failure mode during flexural load application on A2B1 (treated fiber, mechanical abrasion) and A2B2 (treated fiber, alodine treatment), respectively. The stress working on the sample are indicated accordingly either as compression or tension mode. The optical stereomicroscope analysis was performed at 8x magnification. From the images, crack propagation (arrow) and delamination (circle) were observed during the testing which led to the failure of the FML composite. Initially, delamination between aluminum sheet and composite part of KeRALL happened, before followed by crack propagated along the composite part of KeRALL. As shown in Figure 3, the crack seemed to be initiated from the side of the laminate subjected to tensile stress. Moreover, no failure was observed at the top aluminum sheet of the sample, which is in agreement with the result reported by Vieira et al. [7].

The effect of fiber treatment on the KeRALL is found to be relatively less significant. KeRALL with alkaline treated kenaf shows an increment of about 0.6% in flexural strength. Alkalized kenaf was observed to give additional strength to the KeRALL as compared to the non-treated kenaf, probably attributed to the increased strength in the composite part of the KeRALL. Figures 4(a) and (b) show the micrographs of the composite part of KeRALL with treated and untreated kenaf fiber, respectively. From Figure 4(a), treated kenaf fiber in the KeRALL composite shows a good bonding with the epoxy matrix even after the flexural test. Meanwhile, untreated kenaf fiber (Figure 4(b)) shows obvious debonding and fibers pulled out from epoxy matrix after the application of flexural load. This observation suggests that the alkaline treatment helps the kenaf fiber to have better interfacial bonding with the epoxy matrix. Alkaline treatment on natural fibers has been reported to modify their surface and increases their adhesion to polymer matrices [22]. In addition, improvement of fiber strength has also been obtained using alkaline treatment [23-24].



(a) (b)

Figure 4: KeRALL with (a) treated and (b) untreated kenaf fibers

Figure 5 shows the results of Izod impact resistance for KeRALL and KFRC. The impact strength of the KeRALL was almost more than 14-fold higher as compared to KFRC. The result indicates the significant contribution of Al layers to the impact strength of FML. Furthermore, the result reveals that KeRALL with alodine treated Al sheets show higher impact strength than those with surface roughened Al sheets. The highest impact strength was recorded by A1B2 (untreated fiber, alodine treatment) at 38 kJ/m^2, followed with A2B2 (treated fiber, alodine treatment) at 36 kJ/m^2. The structure, geometry and layup of the laminates and the property of the constituents are some of the parameters that influence the impact behavior in FML [25]. Interestingly, the results of impact strength show an opposite pattern to that of flexural strength. It suggests that KeRALL with less firm adhesion between Al sheet and composite core provide better impact toughness. The result is in agreement with the explanation by Cortes and Cantwell [26] which claimed that interlaminar and interfacial delamination were important mechanisms for absorbing impact energy.

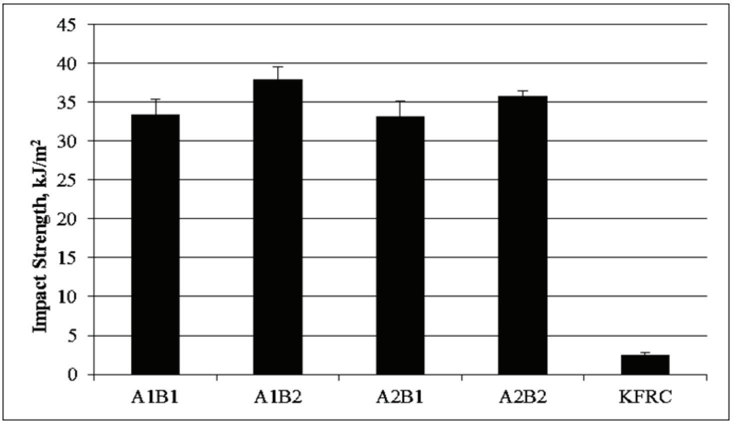


Figure 5: Impact strength of KeRALL as compared to KFRC

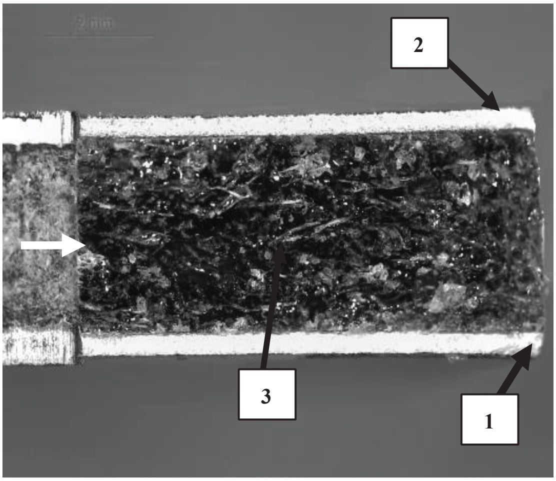


Figure 6: Failure mode upon Izod impact test on KeRALL (A2B1) specimen at 10x magnification

As shown in Figure 6, KeRALL displays four major stages of energy dissipation during impact resistance; i. Plastic deformation of the aluminum layers (tearing of Al sheet as indicated by arrow 1); ii. Delamination may include the matrix cracking (ended area of impact as indicated by arrow 2); iii. Matrix and fiber damage (brittleness of composite part as indicated by arrow 3). The area pointed by the white arrow indicates notched area and hammer striking point. The same conditions have been reported by Laliberte et al. [27] for low velocity impact of GLARE fiber metal laminates.

3.3 Water Absorption Behavior of KeRALL and KFRC

Water absorption test was performed for 20 days (480 hours) using water bath. The water temperature was set up to 30°C throughout the testing period. The result shows that KeRALL in general have low water absorptivity (4 ~ 9%) as compared to KFRC (19%). Moreover, KFRC demonstrated a catastrophic damage in its structure between day 6 to day 9, in the forms of matric cracking and crack propagation along the cross sectional areas. These explain the accelerated water absorption within that particular period of time as shown by circles in Figure 7. On the other hand, KeRALL shows a slower water absorption activity, even under the relatively harsh conditions, owing to the barrier provided by the aluminum outer layers [28]. The Al outer layers contribute to a reduction of more than 50% for water absorbability in KeRALL. Furthermore, KeRALL with alkalized kenaf (A2B1 and A2B2) show tremendous improvement in water resistivity. As noted by Edeerozey et al. [13], chemical treatment of kenaf fiber using NaOH can remove the fiber surface, chemically modify the surface, stop the moisture absorption process and increase the surface roughness when 6% optimum concentration of alkaline is used. Aspect of the degradation became significantly reduced as compared to the ordinary composites through this water resistivity [5].

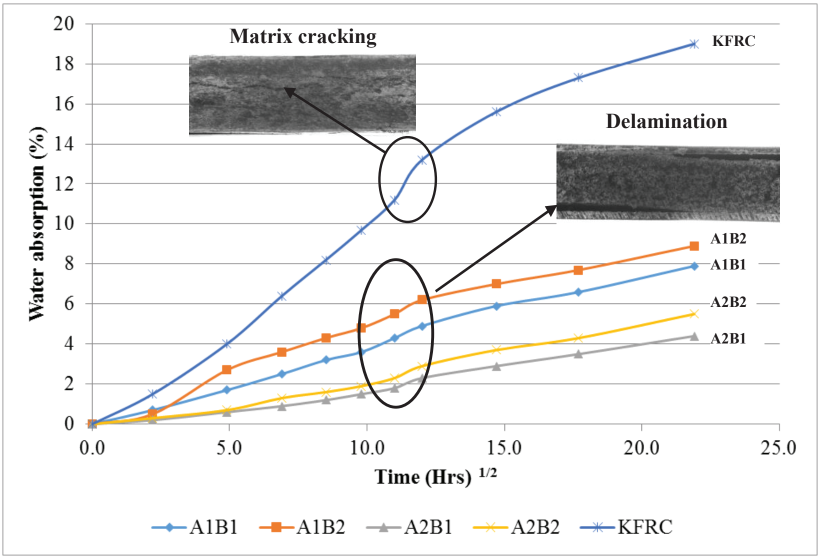


Figure 7: Water absorption behaviour of KeRALL and KFRC

However, it was observed that KeRALL started to show delamination between the metal layer and composite part after 6 days, whereby the water absorbed slightly increased as demonstrated in Figure 7. This phenomenon happens because when polymer composite is exposed to humid environment, it absorbs some water by diffusion and induces swelling. The polymer material as matrix resin becomes soften and increases in size due to diffused water in the composite structure especially through the reinforcement fibers. In many cases of polymer composite, swelling cannot develop freely when reinforcements do not absorb water [29-30]. Once it enters the polymers, it can exist in several ways: as bound water, characterized by strong interaction of the molecule with matrix and free water, present in capillaries and micro cavities within the polymer [31].

4.0 CONCLUSION

Various types of pre-treatments have been applied to kenaf fiber and Al sheets in the kenaf fiber reinforced aluminum laminates (KeRALL). Surface treatments applied to the Al sheet and kenaf fiber are found to contribute to the improvement of physical and mechanical properties in KeRALL. For the Al sheet treatment, surface treatment by mechanical abrasion is found to be more effective than alodine treatment in increasing the flexural strength of KeRALL laminates. It is revealed that KeRALL with surface roughened Al sheets gives the highest value of flexural strength as compared to KFRC, with as much as 283% of increase. Meanwhile, KeRALL with alkalized kenaf shows huge improvement in water resistivity with 4.4% water absorption, compared to that with untreated kenaf (7.9% water absorption). SEM analysis also confirms that treated kenaf fibers possess better bonding with the epoxy matrix. As for conclusion, pre-treatments of metal sheet and fiber such as mechanical abrasion, alodining and fiber alkalization are effective to improve the properties of KeRALL with various degree of effect. The finding also suggests that KeRALL definitely has high potential as a new sustainable FML composite and can be considered as a promising candidate for future industrial applications.

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