

## Influence of hygrothermal conditioning on the properties of compressed kenaf fiber / epoxy reinforced aluminium laminates

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**ABSTRACT** – Increasing environmental concerns have encouraged researchers to utilize natural fibers in the development process for various applications of polymer composites. Hydrophilic nature and low strength of natural fiber become a significant issue and need improvement for wide structural application. Therefore, fiber metal laminates (FML) are selected for overcoming the disadvantages of natural fiber composites with their outstanding degradation resistance. This study has been carried out to evaluate water and temperature effect also known as hygrothermal effect onto kenaf fiber reinforced aluminium laminates (KeRALL) and kenaf fiber reinforced composite (KFRC) as compared to pristine sample. Samples were fabricated by warm compression method and immersed at 30°C, 60°C and 80°C in water bath for 5 days. KeRALL at temperature 30°C, showed the lowest water absorption rate compared to temperature 60°C and 80°C. Both KeRALL and KFRC, at temperature 80 °C showed the fastest water absorption and the earliest to reach saturation state, followed by temperature of 60°C and 30°C. Flexural and impact properties shows the decremented trends at temperature of 30°C, 60°C and 80°C. Interlaminar shear stress (ILSS) show a decrement by 7 % at 30°C, followed by 66 % at 60°C and 54 % at 80°C. Dynamic mechanical analysis (DMA) shows storage and loss modulus of KeRALL were decreased as temperature increased. The decrement is associated with fiber pull out, crack propagation, matrix fracture and delamination as the result of the hygrothermal influence as manifested by the fractographic images. It can be concluded that hygrothermal gives the significant effect on the properties of KeRALL. The finding suggests that KeRALL has high potential as a new sustainable FML composite and can be considered as a promising candidate for future structural applications.

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## INTRODUCTION

Today, natural fiber composites/laminates are now attracting more attention from researchers because they are lightweight, environmentally friendly, have a high availability, and are cost-effective [1]. Polymer composites reinforced with natural fibers such as kenaf, flax, hemp and ramie show high potential to be further developed and applied [2]. According to Aziz and Ansell [3], kenaf fibers have been widely used over the past few years which is a particularly attractive option due to its rapid growth over a wide range of climatic conditions and available in abundant quantity and its consequent low cost as well as possess good mechanical properties [4, 5].

Natural fiber composites demonstrate low mechanical performance, high degradation of natural fibers especially when exposed to humid environment and low heat processability. Also, Ibrahim et al. [6] stated that the mechanical properties of individual fibers from plants were much lower when compared with those of synthetic fibers. The improvements on mechanical properties of the natural fiber based composites are often reported in the literatures in recent years [7–11]. Poor adhesion between fiber surface and polymer matrix, high moisture absorption due to the nature of the hydrophilic natural fibers, variations of natural fiber's parameters such as unstable properties due to different origin plant, water uptake during growing process and harvesting time are among the arising issues of using natural fiber as the reinforcement constituent in polymer composites [2].

With all the arising issues, FML is an effective way to solve the disadvantages of natural fiber composite. Fiber metal laminates (FML) are lightweight structural materials consisting of alternating layers of fiber reinforced polymer composites and metal alloys. The properties of FML is beyond dispute and outstanding such as superior corrosion and

degradation resistance, higher damage tolerance and lower density of composites. At present, most of the previous works on FML have been dedicated to glass fiber reinforced aluminum laminates (GLARE), aramid fiber reinforced aluminum laminates (ARALL) and carbon fiber reinforced aluminum laminates (CARALL), mainly on their physical, mechanical, thermal and chemical properties [12–14]. The combination of natural fiber and metal is possible to produce a new composite that is lightweight with good mechanical properties and can be used in structural application. Although many researchers have been exploring FML, however, there are fewer studies of FML based on natural fiber reinforced composite. Since natural fiber has a sustainable life cycle, low density, lightweight, nontoxicity, renewable, biodegradable and low cost [15], exploration on natural FML has become more attention in recent years.

The properties and performance of the composite structure is a function of its moisture distribution, environmental history, and temperature exposure as it stated by Daniel and Ishai [7]. Thus, among the main concerns for the use of natural fiber reinforced composite materials are their susceptibility to moisture absorption and its effects on the physical and mechanical properties, by Thwe and Liao [8]. While Yang et al. [9] mentioned that moisture absorption can lead to the degradation of fiber-matrix interface region, which in turn creates poor stress transfer efficiencies and result in reduction of mechanical properties. Also, exposure to elevated temperature can result in degradation of mechanical properties, cracking, chalking and flaking of polymers as referred to Lin et al. [10]. However, published data on the properties of fiber metal laminates involving natural fiber are lacking.

So, the objective of this study is to emphasize on the hygrothermal effect on KeRALL performance. Mechanical test such as flexural, impact and thermal analysis were performed by Universal Testing Machine (UTM), IZOD Impact Tester and Dynamic Mechanical Analyzer (DMA). The effects of hygrothermal towards KeRALL further explain, considering that moisture and heat are the determinant drawbacks for the natural fiber and thermoset resin, respectively. Further analysis on the specimen fracture surface was carried out by using a scanning electron microscope and an optical microscope.

## METHODOLOGY

Kenaf fiber (non-woven mat with surface density of 800 g/m<sup>2</sup>, Innovative Pultrusion S/B), epoxy resin (EPO DM A and B, Chemrex Corp. S/B) and aluminium sheet (Al 2024 T3 with 0.5 mm thickness, Kird Enterprise) were used as reinforcement, matrix and face sheet respectively. Prior to compression, surface modification was carried out for both aluminium sheet and kenaf fiber. For this purpose, aluminium sheets underwent mechanical abrasion by using 60-grit sandpaper and kenaf fiber was alkalinized by 5% sodium hydroxide. KeRALL and KFRC were fabricated through warm compression using a hydraulic press (GOTECH) at 80°C. The pressure of 65 kg/cm<sup>2</sup> was applied with a holding time of 15 minutes. The volume fraction for KeRALL and KFRC samples was shown as in Table 1.

**Table 1.** Volume fraction for KeRALL and KFRC samples

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

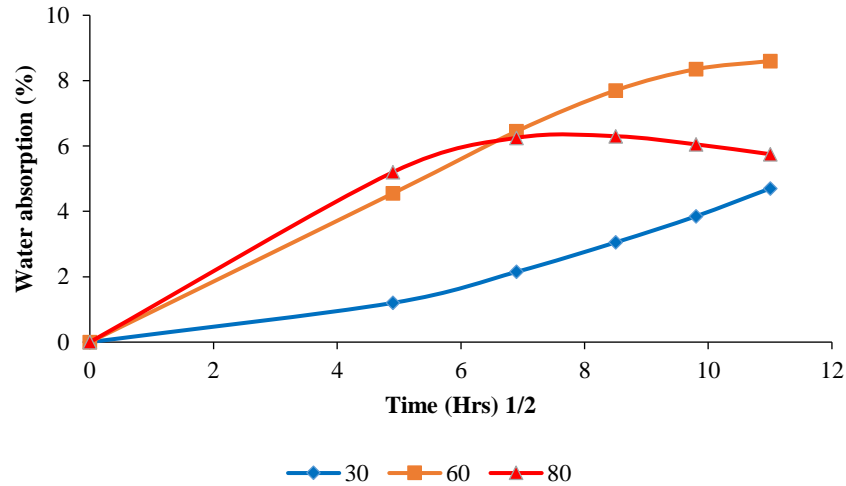
Mechanical cutting equipment was used for sample preparation. The sample was cut according to ASTM D790 and ASTM D256. Prior to testing, all samples underwent immersion process in water bath (YAMATO BK 610) at 30, 60, and 80 °C. The readings of weight changes of sample were consistently taken every day. The duration of immersion process was carried out in 5 days. The change due to water and heat affect was closely monitored in terms of their delamination, matrix and fiber swelling, cracking and others. The conditioned samples were tested by 3-point bending test to investigate the flexure properties of the samples. GOTECH A1-7000-LA 50 kN was used to perform the test at 23 °C ± 2 °C and 50% ± 5% relative humidity in accordance to ASTM D790. While the IZOD impact test was performed by Instron-CEAST 9050 Impact Pendulum with pendulum energy of 2.75 J (KeRALL) and 0.5 J (KFRC) in accordance to ASTM D256 for edgewise notched Izod impact tests. Besides of impact and flexural tests, Dynamic Mechanical Analyzer (DMA) has been conducted by DMA-Q800 with sample dimension of 60mm × 12.7mm × 4mm (length × width × thickness). In this test, the sample was clamped at both edges while the push rod located at its midpoint. Lastly, scanning electron microscope (SEM) and optical microscope was used to examine morphological analysis and observe fractographic image.

## RESULT AND DISCUSSION

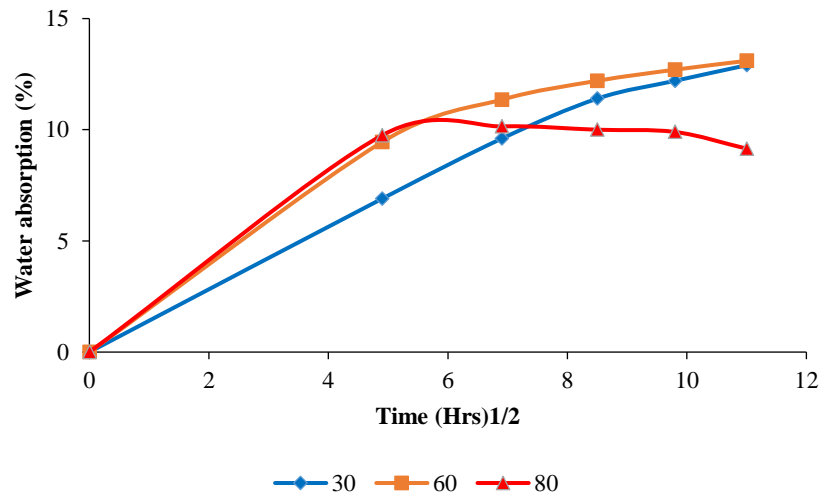
The effect of hygrothermal on KeRALL and KFRC was studied at temperature 30, 60 and 80°C for 5 days. The mechanical, physical and thermal performances were performed to elucidate the hygrothermal effect on KeRALL. Fractographic and microscopic observation was conducted to reveal the effect of hygrothermal on structure of KeRALL. This observation was conducted before and after performing the mechanical test.

## Water Absorption and its Behaviour

Figure 1 and Figure 2 show the percentage of water absorption as a function of exposed time at temperature of 30°C, 60°C, and 80°C and immersed in water for KeRALL and KFRC samples. It was observed that water diffusion behaviour of KeRALL and KFRC can be classified into the Fickian behaviour, where water absorption increases linearly with the square root of time, and gradually slow until an equilibrium state was reached as mentioned by Chandrasekar et al. [16]. This behaviour can be seen clearly at temperature 80°C for KeRALL and KFRC samples. Meanwhile, at the temperature of 30 °C and 60°C, both samples show non-Fickian and Anomolous behaviour, respectively. The dissimilar behaviour of KeRALL and KFRC at elevated temperature indicated that application of heat and humidity will give different findings especially on the matrices of the composite.



**Figure 1.** Water absorption behaviour of KeRALL at temperature 30, 60 and 80°C



**Figure 2.** Water absorption behaviour of KFRC at temperature 30, 60 and 80°C

KeRALL at temperature 30°C show the lowest water absorption rate compared to temperature 60°C and 80°C. Besides, percentage of water absorb still increases after 5 days of immersion. This phenomenon occurred because of the saturation state at temperature 30°C did not reached up to maximum value. It can be concluded that heat and humid environment faced by the samples did affect epoxy resin on the composite part. Meanwhile, both KeRALL and KFRC, at temperature 80°C show the fastest water absorption and reached the earliest saturation state. These were followed by temperature 60°C and 30°C. Consequently, heat and humid environment faced by the samples did affect the matrices of the composite. The sample can reach the fastest saturation at high temperature environment. This is because the hot and humidity environment can cause the acceleration of plasticization and degradation rate of the matrix epoxy. This phenomenon is reducing the percentage of water absorption caused by voids deduction. The expansion of epoxy matrix in regards of heat may result in decrease number of voids. Thus, the number of voids will decrease and influence the earliest saturation state of epoxy matrix. According to Ferguson and Qu [17], there are three mechanisms that contribute

to water penetration at the interface in epoxy adhesive structures: bulk diffusion, wicking along the interface, and capillary action associated with micro-cracking. Indeed, kenaf fiber existed on KeRALL was aggressively water absorbed caused by hydrophilic nature of the fibers. So, the volume of kenaf fiber has significantly affect the percentage of water absorbs. The mechanism for water penetration of KeRALL is wicking along the interface of matrix to kenaf fiber as well as Al sheet metal. Also, capillary action is associated with micro cracking of epoxy matrix and micro pathway of kenaf fiber.

The absorption percentage of KeRALL samples were slowly increasing compared to KFRCs. It is because of Al metal in KeRALL acts as protective layer from the temperature effect. In summary, KFRC samples absorb water more than 10% compared to KeRALL within the range of less 10%. The same achievement was also reported in Botelho et al. [18], who claimed that the outer skin of aluminium layers over the Glare composites reduces the susceptible area prone to moisture attack, since only free edges of the laminate were exposed to the environment. As a result, a lower rate of moisture absorption was observed for Glare laminates compared to the fiber reinforced polymer composites, reaching only 0.15% from mass gain. In general, the percentage of water absorption in FML regardless of the metal was very low when compared with the GFRP (Glass fiber reinforce polymer) and CFRP (Carbon fiber reinforced polymer) composite, by Hariharan and Santhanakrishnan [19].

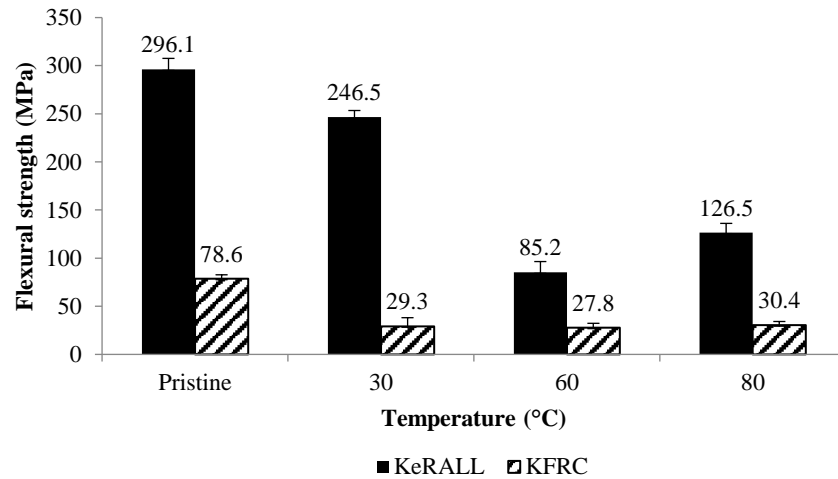
**Table 2.** Summary of hygrothermal effect on KeRALL and KFRC samples in 5 days

Sample	Temperature (°C)	Type of behaviour	Saturation (Hrs)	Water absorption	Degradation rate
KeRALL	30	Obey Fick's Law	> 120	Low	Slow
	60	Non-Fick's Law	120	High	Medium
	80	Anomalous (S shape)	24 to 48	Medium	Fast
KFRC	30	Obey Fick's Law	> 120	Medium	Slow
	60	Non-Fick's Law	120	High	Medium
	80	Anomalous (S shape)	24 to 48	Low	Fast

Even though KeRALL samples show the lowest water absorption rate compared to KFRCs, the unmoving water can penetrate the sample. This phenomenon will also affect adhesion bonding and could damage the samples for the long run. According to Fan and Suhir [20], the mechanisms for adhesion bonding failure is the swelling of polymeric materials upon exposure to moist environments. Consequently, causing an additional mismatch between volumetric expansions of substrate and adhesives. This is even more pronounced when the joint between a polymer and metal is investigated. Since the metallic substrate is impermeable to moisture, only the polymeric adhesive absorbs moisture and causes a mismatch in hygroscopic strains. Table 2 present the findings to summarize the hygrothermal effect on KeRALL and KFRC.

### Flexural Properties and its Behaviour

Figure 3 shows both KeRALLs and KFRCs flexural strength decreases as water temperature increased. Both samples indicated a decrease in flexural strength at temperature 30°C to 80°C as shown in Table 3. The highest rate of decrease is at temperature 60°C, followed by temperature 80 °C and 30°C, which occurred close to the  $T_g$  point. At 60 °C, the highest percentage of water absorb was observed as referred to Figure 1 and 2. This induced plasticization of the polymer matrix with concurrent swelling and directly affects the flexure strength reduction for KeRALL and KFRC. Sideridis et al. [21] reported that thermal expansion and swelling of a composite due to temperature and moisture variations significantly influence the mechanical properties of its constituents, through residual stresses between the fiber and matrix. Due to the failure of adhesion bonding between composite part and Al metal, KeRALL shows highest percentage of flexural strength decrement. Concerning the latter, it is well known that immersion of the composite materials into aqueous environments results in a degradation of their thermo-mechanical properties. This phenomenon is related to the moisture-induced plasticization and micromechanical damaging such as interfacial damage and matrix cracking.

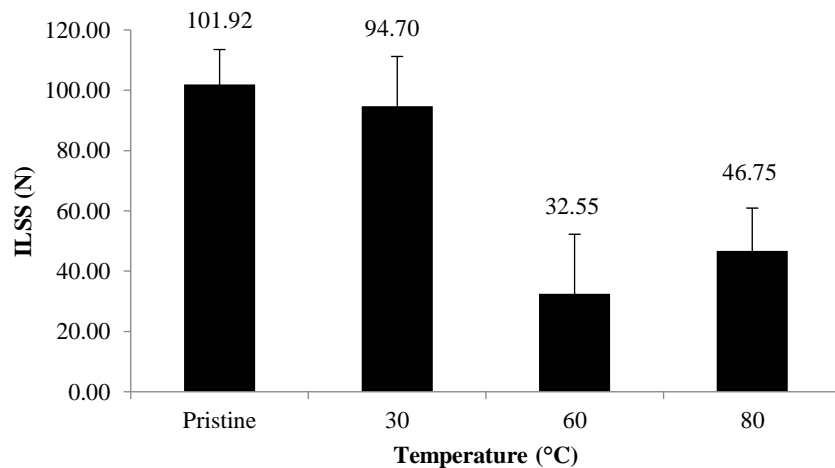


**Figure 3.** Flexural strength of KeRALL and KFRC at temperature 30, 60 and 80 °C hygrothermal conditions

**Table 3.** Percentage decrement in flexural strength of KeRALL and KFRC after hygrothermal conditions

Sample	30 °C	60 °C	80 °C
KeRALL	17 %	71 %	57 %
KFRC	63 %	65 %	61 %

The tested KeRALL was characterized by a decrease in interlaminar shear strength defined in the 3-point bending test after hygrothermal condition. ILSS value shows a decremented proportional to the temperature increase as referred to Figure 4. The result shows decremented of ILSS at 30°C, 60°C and 80°C by 7%, 66% and 54%. The highest value of decrement achieved at 60 °C probably because of saturation state before the  $T_g$  point. The hydrophilic nature of kenaf fiber also encourages the occurrence of earlier saturation stage of epoxy. Previous studies by Ypma and Borgonje [22] claimed that the decrease in ILSS due to environmental exposure is about 15% and it was strongly influenced by the temperature.

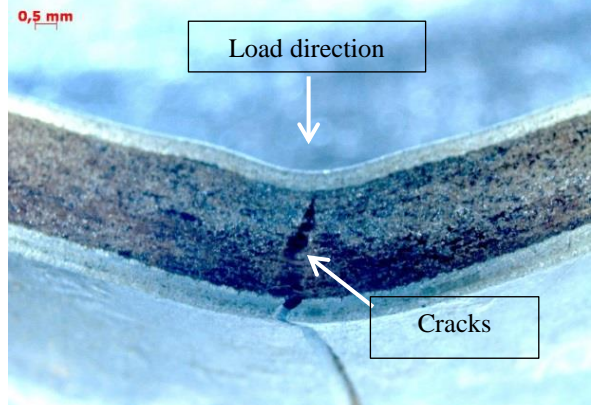
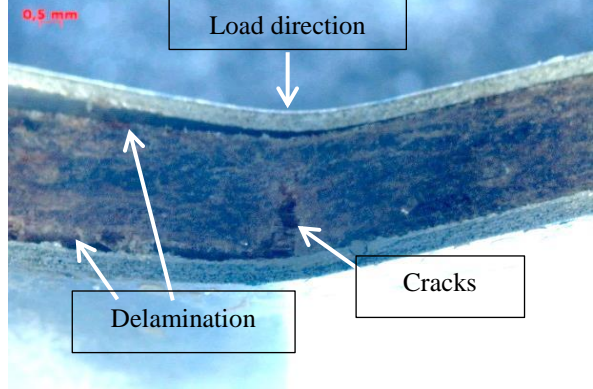
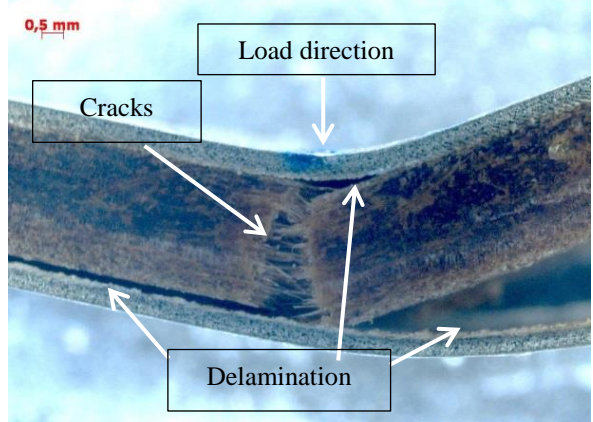


**Figure 4.** ILSS of KeRALL at temperature 30, 60 and 80 °C hygrothermal condition and without hygrothermal (pristine KeRALL)

Fractographic image after flexural test has been observed for study behavioural fracture of the sample. There are three assumptions that can be made based on the fracture behaviour in KeRALL structures. Firstly, the temperature increases from 30 °C to 80 °C causing catastrophic damage for KeRALLs sample when flexure load was applied. Secondly, the catastrophic damage such as delamination on both upper and lower part of metal, cracking on composite part of KeRALL and tear on the back side of KeRALL especially at temperature 80 °C predictably caused by tension and shear mode of flexural load. Thirdly, the difference of thermal expansion between metal and composite part as explained earlier is also

part of the major factor for KeRALL experiencing catastrophic damage caused by matrix swelling. Table 4 shows the fractography stage of KeRALL at temperature 30 °C, 60 °C and 80 °C. The labelling on the images is indicating load direction, cracks and delamination is regards with the behaviours of KeRALL after test.

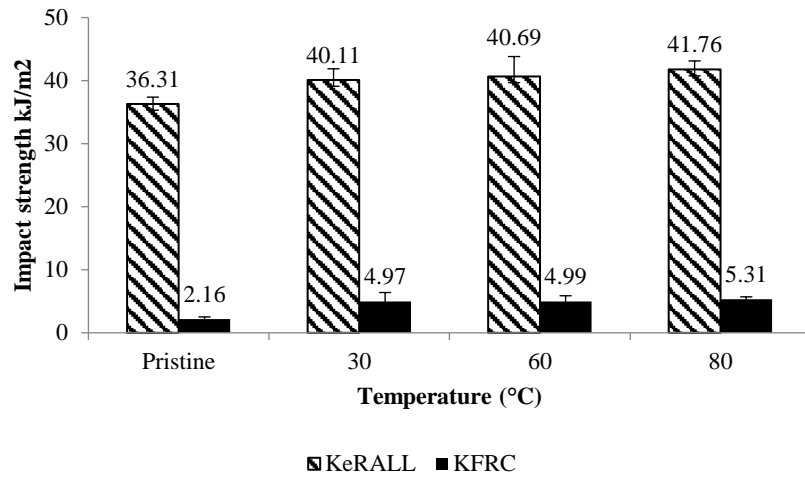
**Table 4.** Summary of fractographic images of hygrothermal conditioned KeRALL upon flexural load test and its description

Fractographic Images	Description
	<p><b>At temperature 30 °C :</b></p> <p>Composite part of KeRALL and Al metal sheet was broken together. No delamination has happened at all.</p>
	<p><b>At temperature 60 °C:</b></p> <p>Delamination occurred at upper side due to compression mode of flexure load without breakage on the composite part (only minor cracks appeared).</p>
	<p><b>At temperature 80 °C:</b></p> <p>Catastrophic damage (delamination for both upper and lower side of metal part and broken of composite part due to tension, compression and shear mode of flexure load).</p>

### Impact Properties and its Behaviour

Figure 5 shows the impact strength of KeRALLs and KFRCs at 3 different temperatures, 30°C, 60°C and 80°C. It shows that both samples have similar criteria of impact strength that were influenced by temperature. The result shows that the impact strength for both KeRALL and KFRCs were increased from the temperature of 30 °C to 80°C. At 30°C, 11% increment of KeRALL were recorded, followed by 12% at 60 °C, and 15% at 80°C. The outstanding incremental is shown by KFRC with the increment in ranging from 130% – 150%. This means that the structure of KeRALL was enhanced by the composite part of the samples. The enhancement is due to the plasticizing effect of water exposed towards the matrices. In this case, the polymer matrix absorbed water which end up increasing the polymer free volume existed in the structures. This phenomenon caused a decrease on the stiffness of the polymer together with an increase on the toughness of polymer matrix. Marom [23] reported that the short-term effect of water is to increase the mode I fracture toughness, while it deteriorates in the long run. They found deterioration of fracture toughness with the increase in

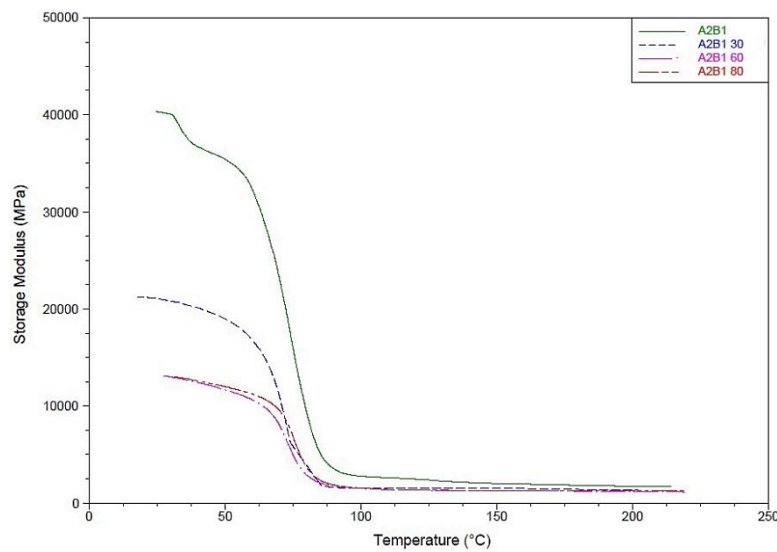
temperature. Similar finding was obtained by Abdel-Magid et al. [24] where the modulus, strength, and strain of the E-glass/epoxy composite material are affected by the presence of moisture and mechanical loading when compared to control specimens. They also found that combined effects of load, moisture and temperature on the properties of E-glass/epoxy composites exhibited an increase in strength, decrease in strength modulus, and increase in strain to failure but decrease to a longer time exposure.



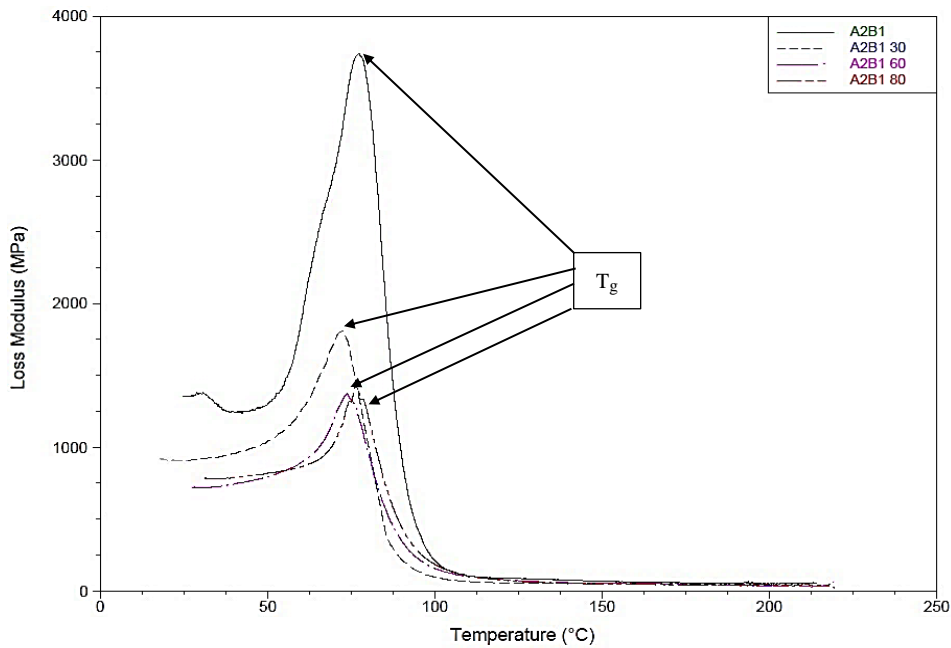
**Figure 5.** Impact strength of KeRALLs and KFRCs at temperature 30°C, 60 °C and 80°C hydrothermal condition and without hydrothermal (pristine)

### Dynamic Mechanical Analysis after Hydrothermal

Figure 6 to 7 show DMA results of KeRALLs at 30°C, 60°C and 80°C of hydrothermal condition. The result shows storage and loss modulus of KeRALL were decreased as temperature increased. The result shows that storage modulus was decreased to 50% at 30°C and more than 50% at 60°C and 80°C of hydrothermal condition. In this case, 30°C shows a more elastic character or solidlike nature of KeRALL compared to 60°C and 80°C. The effect of vibration with applied temperature gave a significant outcomes for mechanical properties of KeRALL. All samples suddenly experienced mechanical failure after  $T_g$  point of polymer matrix. This means that the strength of KeRALL is totally dependent on the properties of epoxy matrix. Once the epoxy was affected by moisture and temperature like plasticization and swelling, mechanical and physical changes will be imposed to the KeRALL as shown in Figure 6 and 7.

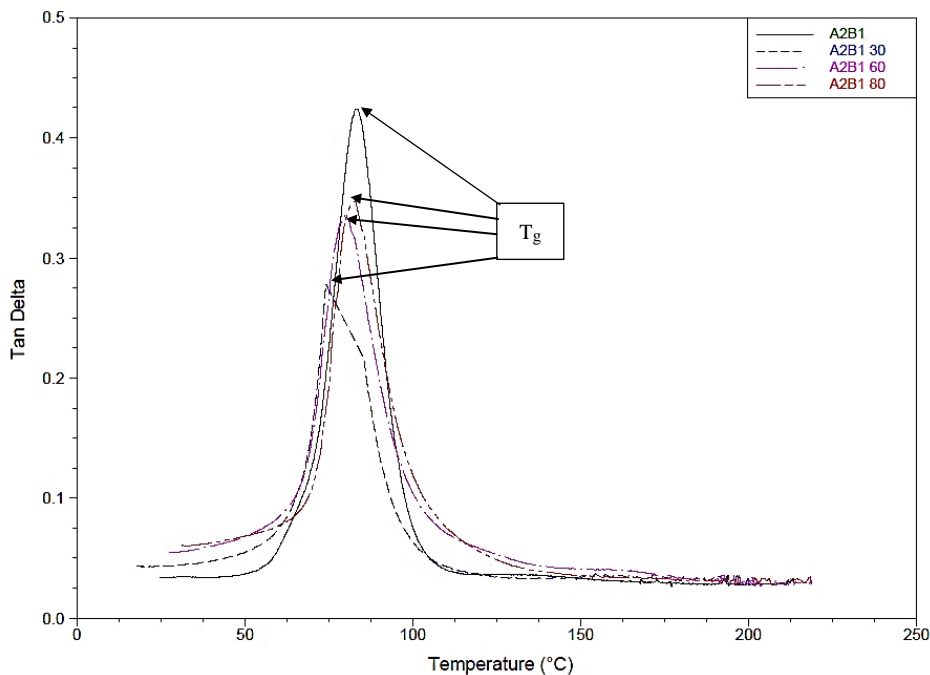


**Figure 6.** Storage modulus of KeRALL at temperature 30°C, 60°C and 80°C hydrothermal condition and without hydrothermal (pristine)



**Figure 7.** Loss modulus of KeRALL at temperature 30°C, 60°C and 80°C hydrothermal condition and without hydrothermal (pristine)

Damping behaviour which is refer to loss modulus was reduced by temperature as referred to Figure 7. It is similar for temperature 30 °C, to a more viscous character or liquid-like nature of KeRALL. As a result, the energy required to deform the sample is elastically recoverable for KeRALL at temperature 30 °C. In a physical sense, the storage modulus is related to the stiffness of the material and the loss modulus is reflected in the damping capacity of the material as claimed by Menczel and Prime [25].



**Figure 8.** Tan delta of KeRALL at temperature 30, 60 and 80°C hydrothermal condition and without hydrothermal (pristine)

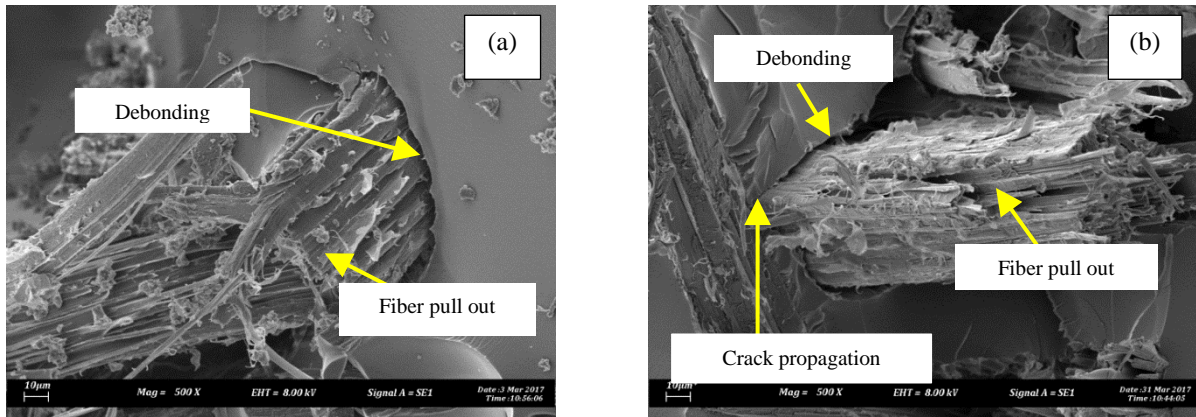
Figure 8 shows the shifting of  $T_g$  point for KeRALL at temperature 30°C, 60°C and 80°C. The results indicate the shifting in the range of 17% for minimum value at 80°C to 35% for maximum value at 30°C. The increasing of temperature from 30 to 80°C was probably the temperature imposed for heat treatment towards the epoxy resin of composite part. The absorbed moisture and application of heat during hydrothermal conditioning changes the mechanical properties of polymeric materials existed in KeRALL. Moisture can change the elastic modulus and shift the glass



transition temperature of polymers to lower values [17, 26]. As a result, it affects the overall mechanical properties of KeRALL.

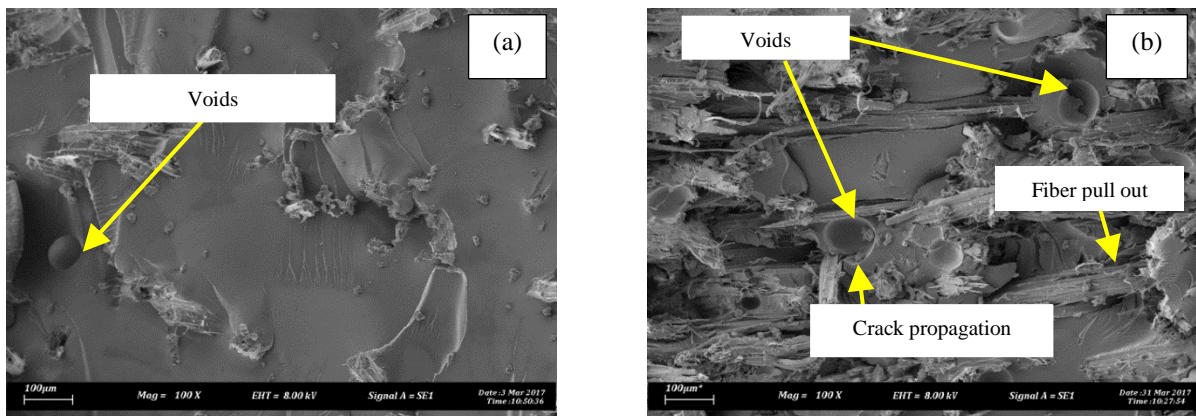
### Morphological Analysis

Hygrothermal effect on the microstructure was observed to clarify the fracture criterion of KeRALLs especially after flexural tested. Hence, the fracture observed will be divided based on the type of defect on the component of KeRALL i.e. fiber pull out, debonding, crack propagation, voids, and delamination between fiber-matrix as well as Al sheet adhesion bonding as shown in Figures 9 to 11.



**Figure 9.** SEM image of fiber pull out and fracture surface of KeRALL: (a) without hygrothermal and (b) with hygrothermal at temperature 80°C

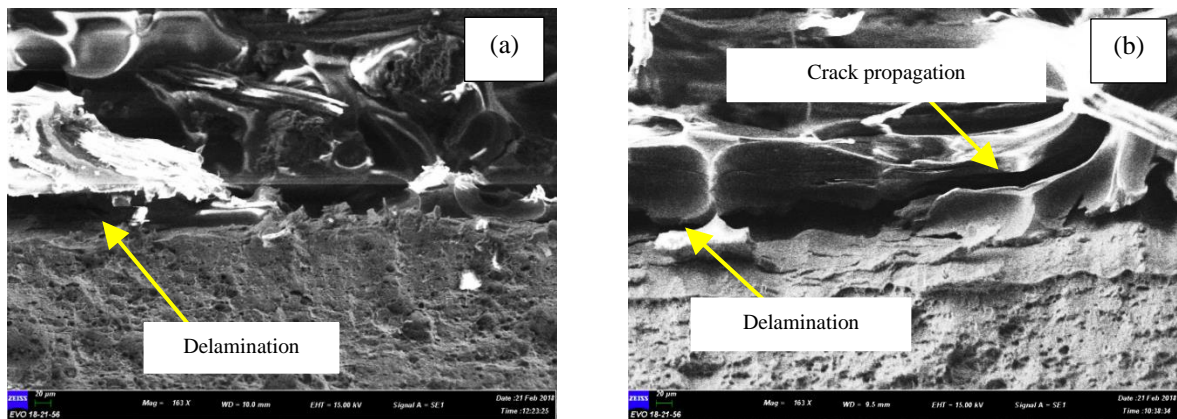
Figure 9(a) and (b) show that kenaf fiber and epoxy matrix condition with and without hygrothermal conditions. As illustrated, temperature and water absorbed during hygrothermal condition contribute catastrophic damage of epoxy matrices. Most of the matrices are crack and induce micro pores surrounding the fiber. Fiber surface is rougher and some split into thinner fibrils, caused by water absorption. The plasticization of KeRALL caused by water absorption was found to decrease the modulus of KeRALL after hygrothermal conditions.



**Figure 10.** SEM images of fiber-matrix adhesion of KeRALL: (a) without hygrothermal and (b) with hygrothermal at temperature 80°C

Figures 10(a) and (b) show the matrix region for KeRALL sample with and without hygrothermal conditions. The SEM image shows a weaker bonding between the fibers and the matrix in hygrothermal conditions compared to without hygrothermal conditions, which results in a less efficient transfer of stress along the fiber-matrix interface before composite failure. Also, it is observed that most of the fibers were pulled out from the matrices. Therefore in this case, it results in a decrease of KeRALL strength after hygrothermal conditions.

Figure 11 (a) and (b) show fiber/matrix-Al sheets adhesion on KeRALL with and without hygrothermal conditions after flexural test. The SEM images of KeRALL with hygrothermal indicate matrix bonding fracture and induce crack propagation along the matrix region (Figure 11(b)). Furthermore the delamination's gap at 80°C temperature is bigger than KeRALL without hygrothermal conditions. With this failure, the overall strength of KeRALL has been reduced since its strength is dependent on the adhesion between composite part and Al sheet. However, the Al sheet region shows the same behavior before and after the hygrothermal conditions, and besides, they are not affected by water absorption in 5 days of immersion and temperature up to 80°C.



**Figure 11.** SEM images of fiber/matrix-Al sheets adhesion of KeRALL: (a) without hygrothermal and (b) with hygrothermal

## CONCLUSION

The behaviour of KeRALL at elevated temperature revealed and noted that application of heat and humidity will give different findings especially on the matrices of composite. In this paper, the effect of hygrothermal on KeRALL at temperature 30°C, 60°C and 80°C for 5 days were successfully studied. Flexural, impact and water absorption test as well as dynamic mechanical analysis were investigated to establish the performance of KeRALL. It can be concluded that KeRALL at temperature 30°C, showed the lowest water absorption rate compared to temperature 60°C and 80°C. KeRALL at temperature 80°C showed the fastest water absorption and the earliest to reach saturation state, followed by temperature of 60°C and 30°C. A mechanical property which is ILSS properties shows the decremented trends at temperature of 30 °C, 60 °C and 80 °C. For dynamic mechanical analysis (DMA), the results show that storage and loss modulus of KeRALL was decreased as temperature increased. The mechanical test results were supported by the microstructural analysis through the defect such as fiber pull out and fracture surface, fiber-matrix as well as Al sheet adhesion bonding on fractographic images. Therefore, the newly developed KeRALL sandwich composite opens a greater commercial potential for kenaf fiber in structural engineering applications.

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