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***Research paper***

# Effect of Kenaf Alkalization Treatment on Morphological and Mechanical Properties of Epoxy/Silica/Kenaf Composite

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## Abstract

The mechanical performance of silica modified epoxy at various concentration of sodium hydroxide for surface treatment of multi-axial kenaf has been analyzed. Epoxy resin with amine hardener was modified with silica powder at 20 phr and toughened by treated kenaf fiber that immerses in various concentrations of sodium hydroxide (NaOH) ranging from 0% to 9% of weight. The composite was analyzed through differential scanning calorimetry (DSC) to ensure complete curing process. The mechanical properties of the composites were analyzed through flexural test, Charpy impact test and DSC to ensure the complete curing process. DSC analysis results show epoxy sample was completely cured at above 73°C that verifies the curing temperature for preparation for the composite. Hence, 3% NaOH treated composite exhibits the best mechanical properties, with 10.6 kJ/m$^2$ of impact strength, 54.1 MPa of flexural strength and 3.5 GPa of flexural modulus. It is due to the improvement of fiber-matrix compatibility. Analysis by SEM also revealed that a cleaner surface of kenaf fiber treated at 3% NaOH shown cleaner surface, thus, in turn, improve surface interaction between fiber and matrix of the composite. The composites produced in this work has high potential to be used in automotive and domestics appliances.

***Keywords***: *Charpy Impact Test; Flexural Test; Kenaf; Natural fiber; Scanning Electron Microscopy*

## 1. Introduction

Based on the World Commission on Environment and Development (WCED), sustainability was defined as the needs of the present without compromising the capability of the next generation to survive with their own need [1]. Sustainability is a concept to maintaining the global catastrophic that overwhelming world society. One of the major crises facing mankind is the increase in world population and suppress of environmental sources for daily usage. The incremental of the demand for built infrastructure has led to significant waste energy and material used mainly in the construction and manufacturing industries. In this case, the improvement can be made as all industries work together in embracing the reuse of industrial based-product and renewable materials in their own respective industry.

Currently, the use of conventional fiber has been decreasing in demand as the natural fiber exhibit the same characteristics with the same strength and quality. The recent studies have proven that the additional additives such as fillers and catalyst with also the application of chemical treatment have led the material of natural fiber-based product can substitute the conventional fiber. Natural fiber hybrid composite is the best option to have a structure with the lightweight application [2]. The replacement of conventional fiber with natural fiber in the manufacturing and automotive industries can gain economic, environmental and social aspects. Better electrical resistance, good in mechanical, thermal and insulating properties and high resistance to fracture possesses the demand for natural fiber [3].

In recent studies, a major flaw of the effectiveness of natural fiber composites due contradictory between hydrophilic fibers and hydrophobic matrix. The counter effect of this case leads to poor adhesion of fiber/matrix as the presence of two conflicting elements which is pendant hydroxyl and polar groups. In this case, it causes the decreasing quality of mechanical properties as high moisture uptake occurs [4]. Thus, the improvement of fiber matrix attraction either may be executed by matrix modification engaging additive or improving fiber surface is the vital issue to be settled down [5].

Current study that conducted by Fiore *et al.*, 2015 showed that alkaline treated of kenaf fiber was fabricated in laminated form through vacuum bagging technique for observing mechanical properties by varies the time of immersion of kenaf in alkaline treatment [6]. While in Bajuri *et al.*, (2017) findings also stated that they are applying the vacuum bagging technique for fabrication laminate in determining the effect of inclusion percentage of silica nanoparticle [7]. To date, no studies have done using hand lay-up method for alkaline treatment. Therefore, in this study focusing on the effect of kenaf alkalization treatment on morphological and mechanical properties of Epoxy/Silica/Kenaf composite via hand lay-up is explored.

The objective of this work is to determine the flexural and impact properties of kenaf reinforced epoxy with the variation of concentrated sodium hydroxide (NaOH) for kenaf surface treatment. Alkalization or mercerization is one of the surface treatments that use to improve the performance of natural fiber in the composite. It does help in bridging the gap compatibility between hydrophilic fiber and hydrophobic matrices features [6]. For instance, Vilay *et al.*, (2008) stated that the application of mercerization by using

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NaOH for fiber loading ranging 0 to 20 vol%. They found that specimen increases the flexural strength in 11 % while flexural modulus increases about 20% when treated fiber with 6 % concentration of NaOH as compared to untreated fiber composite [8].
Silica particles have tremendous large surface area and smooth surface. Those features lead to form a strong physical interfacial contact when it embedded in resin. Currently, silica particles are available in all size raging in micrometer up to nanometre. Normally, it used about 5-10 vol% in polymers with a particulate size of 5-100µm. The best balance of mechanical properties for a composite to form with silica particle is fillers with high surface area/volume ratio (S/V). In this case, the effect of particle size of fillers in epoxy resins also helps in improvising the mechanical properties [9].
Hand lay-up is one of open molding process that applies for manufacturing several of composites product from small to large size. The processes involve in this technique are started with the mold is prepared with the required size and shape and removed the residue and dust on it. Then, a first layer of releasing agent is applied to all along the mold surface to have a good quality surface after cured for a couple of hours. Next, a roll stock of fiber reinforcement is placed on the mold and subsequently put a laminating resin on it by pouring, blushing, spraying or using a paint roller. The process is gaining acknowledgment because it is a cost-effective process as it is low start-up cost as it not need to require a specific machine to help the process. This kind of process is a key part of the composite industry because of its adaptability and quantity [10].

# 2. Material and Methods

## 2.1. Preparation of Material

The matrix used for fabrication was the epoxy resin of Epoxamite 100 with the cycloaliphatic amine. The epoxy's resin to hardener ratio is 100:25 by weight.
The kenaf fiber used was originating from the Kelantan state of Malaysia. The orientation of the fibers was random in the mat form. Silica powder was used with sized of 44 µm diameter. The kenaf used in this project is bast part to extract the fiber. This is due to it is tough fiber outer layer but low in weight about 35% by weight. Besides, it has long, strong fibers make a higher quality product. Thus, it provides better engineering qualities for composites product.



**Fig. 1:** Kenaf Fiber Mat

## 2.2. Alkaline Surface Treatment

NaOH solution was distinguished by its concentration in 3%, 6%, and 9%. The NaOH pallets were drowned into the water to form up NaOH solution. When the solution was prepared, the kenaf mat is immersed in the particular NaOH solution. The treatment of the kenaf was undergoing in 24 hours in the condition of room temperature at which the optimum time for the kenaf to modify its surface to have better mechanical properties.
When the treating of kenaf surface was completed, the kenaf mat was removed from the solution. It was washed and rinsed by using tap water three times to remove the leftover of sodium hydroxide on the surface. When it was done, the treated fiber was dried at room temperature in 48 hours. Later, the post-drying process was carried out in an oven as the temperature of the oven maintained at 100 °C for 6 hours.

## 2.3. Composite Fabrication

The composites were fabricated into 180 mm × 180 mm plates to be cut into the size required for the flexural and impact tests respectively. For the preparation of the silica powder, the silica powder was wet ball milled about 48 hours by using ball milling machine. Before the ball milling started, the composition of the ball milling process is 1:1:2 for silica powder, distilled water and zirconia ball respectively. Then, the solution of silica was dried in the oven for overnight to get finer silica powder.
For the preparation of composite, epoxy resin was mixed with the hardener in the scale of 1:10 of proportion with silica powder with the scale of 10 phr. The mixture was stirred at room temperature until it blended completely. When the mixture was prepared, a mold was cleaned until the dust and residue were removed. Before poured, the mold was covered with honeycomb wax that acted as a releasing agent. The first layer resin mixture was laid up uniformly on mold followed by modified kenaf fiber mat. The process was repeated until four layers were obtained. The composite was cured in the oven at 80 ℃ for 2 hours and post-cured at 110 ℃ for one hour after the composite detached from the mold. Table 1 represents the preparation of epoxy composites with its composition. Six types of composites were produced where pure epoxy (PE), epoxy silica composite (ES), untreated kenaf epoxy with silica composite (UT), epoxy silica composite with treated 3% of NaOH fiber (3T), epoxy silica composite with treated 6% of NaOH fiber (6T), and epoxy silica composite with treated 9% of NaOH fiber (9T), as shown in Table 1.

**Table 1:** Epoxy/Silica/Kenaf Composite Composition

| Composite | Volume Ratio | | Silica (vol%) | Sodium Hydroxide (NaOH) Concentration (%) |
|---|---|---|---|---|
| | Epoxy Resin System (vol%) | Kenaf (vol%) | | |
| PE | 100 | - | 0 | 0 |
| ES | 100 | - | 20 | 0 |
| UT | 75.5 | 24.5 | 20 | 0 |
| 3T | 75.5 | 24.5 | 20 | 3 |
| 6T | 75.5 | 24.5 | 20 | 6 |
| 9T | 75.5 | 24.5 | 20 | 9 |

## 2.4. Differential Scanning Calorimetry (DSC) test

Differential scanning calorimetry (DSC) test was applied under Mettler Toledo DSC 823. First DSC analysis was conducted to analyze the parameter for epoxy resins. From the DSC curve, the optimum curing temperature and heat of curing were determined. The dimension of epoxy resin with hardener is 12 mg. The sample was placed in the DSC cell and heated from 25 ºC to 250 ºC at a heating rate of 10 ºC/min under nitrogen atmosphere. For the second DSC test, it was analyzed to determine the degree of cure of epoxy composite. About 46.9 mg of molded epoxy cured with an amine in a ratio of 100:50 part by weight was used in this test. The sample was heated from room temperature 25 ºC to 250 ºC, with a heating rate of 10 ºC/min.

## 2.5. Charpy Impact Test

Charpy impact test was applied under ASTM D6110 under GUNT WP400 impact tester. The dimension of specimens was 63.5 mm x 12.7 mm. Impact test was conducted to simulate the mechanical properties of the composite in resisting high-speed loading. For

each loading, the results are taken from average 5 specimens tested.

### 2.6. Flexural Test

3-Point bending test was applied under ASTM D790 under INSTRON Series 8500. The dimension of the specimens was 125 mm x 12.7 mm. The support span was set the span-to-depth ratio of 16:1. The specimen straining rate 0.01 mm/mm/min. A flexural test was conducted to identify the ability of sample which is the composite material that can withstand bending forces. For each loading, the results are taken from average 5 specimens tested.

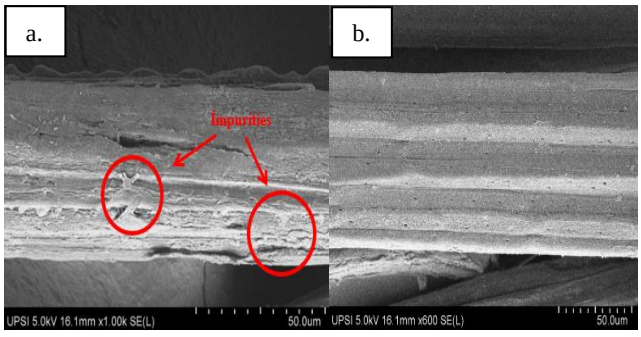
### 2.7. Scanning Electron Microscopy (SEM) Test

Scanning Electron Microscopy (SEM) was applied under Hitachi SI 8020. About 7 mg of samples from untreated kenaf, 3%, 6% and 9% treated kenaf and 10 mg of ES, UT and 3T were used. SEM was conducted to analyze the effect of alkaline treatment of kenaf fiber and morphology of the fracture surface of impact specimens. It was used at 5kV. Every sample was coated with a thin layer of platinum to improve the conductivity and prevent charging.

## 3. Results

### 3.1. Effect of Alkaline Treatment on Kenaf Fiber

Figure 2 shows the SEM micrograph of untreated kenaf fiber, 3%, 6% and 9% of treated kenaf fiber with NaOH solution. The results showed many impurities that covered the surface of kenaf fiber that was indicated by a red circle. Structure of the surface was rough and scaly as the impurities made up a majority on kenaf surface area. The argument from Kalia *et al.* (2009) proven that impurities such as cellulose, hemicellulose, and lignin and highly concentrated with hydroxyl groups densely appeared on the surface of kenaf as it develops rough topography on the surface [11]. The effect of alkalization that was discussed by Kasiviswanathan *et al.* (2015) to wash away the impurities that stayed on kenaf surface is it allows the compatibility of kenaf fiber and epoxy matrix [12]. Based on Figure 2(a), the impurities on the kenaf surface become less (treated with 3% NaOH). However, Figure 2(b) and (c) shows the cracks started to propagate as the higher the concentration of the alkaline solution gave corrosive to the structure of kenaf as seen in a blue circle. The residue and by-product of the chemical reaction of sodium hydroxide to kenaf surface started to deposit on the surface as shown in a green circle [13]. Figure 2(d) explains that kenaf surface is denatured since the 9% of NaOH was corrosive and destroyed the structure of kenaf. This shows that the higher concentration of sodium hydroxide solution exhibited characteristics of corrosive reaction [14]. The residue and by-product of the chemical reaction of NaOH solution to kenaf surface started to deposit on the surface as shown in a green circle [13].



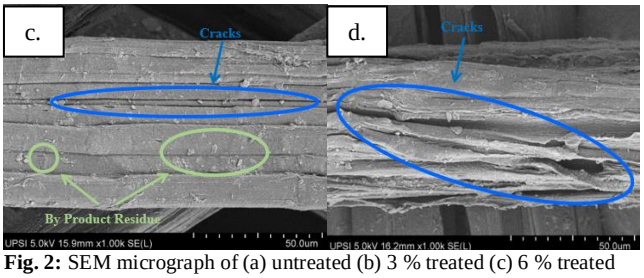




**Fig. 2:** SEM micrograph of (a) untreated (b) 3 % treated (c) 6 % treated (d) 9 % treated with NaOH solution

### 3.2. Differential Scanning Calorimetry (DSC)

Figure 3 represents the DSC curve of epoxy resin. From the results, epoxy started to cure at 42 $^0$ C and curing peak was at 103 $^0$ C. The heat released during curing ($\Delta H$) was 269.16 $Jg^{-1}$. The curing process completed when the temperature achieved at 250 $^0$ C. The data obtained from this analysis was used to determine the curing cycle of the composite which was pre-cured at 80 $^0$ C for 2 hours and post-cured at 110 $^0$ C at 1 hour. The cured epoxy resin system following the proposed curing procedure was analyzed to confirm the degree of curing.

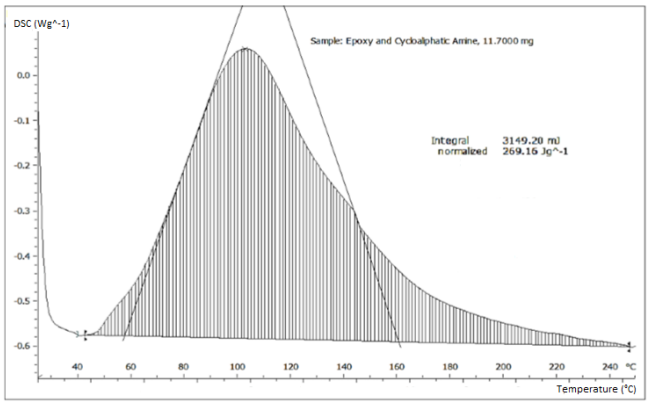



**Fig. 3:** DSC curve for epoxy and amine

In the DSC test, a cured specimen was used to analyze the degree of conversion in order to ensure complete curing of the composite t a different temperature, ranging from 25 ºC to 250 ºC. The mass of the sample used was 46.9 mg. Based on Figure 4, the glass transition temperature, $T_g$ was at 73 ºC. At this point of temperature, the epoxy sample was believed to be changed from a glassy state to a rubbery state. During this stage, the material had undergone transition phase which was glass as it was in a form of hard, solid, amorphous material and transform to a soft, rubbery, liquid phase. The curing reaction of epoxy is an irreversible poly-addition exothermic process [15]. Despite that, there was no exothermic reaction found in the DSC curve (Figure 4). This indicated that the epoxy system was fully cured and the conversion of the epoxy was 100%. Thus, the curing process at 80 ºC and post-cured at 110 ºC were proven to produce a fully cured composite.

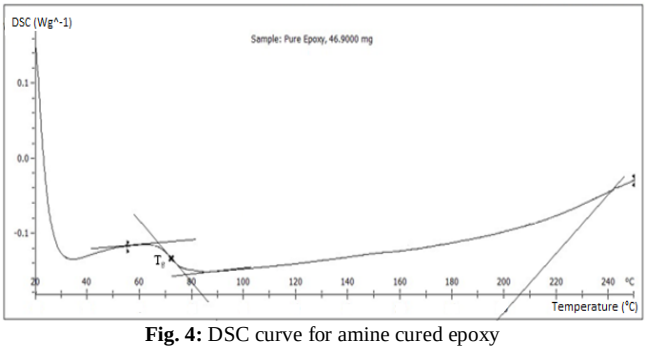



**Fig. 4:** DSC curve for amine cured epoxy

### 3.3. Charpy Impact Test

Figure 5 shows the impact strength of composites analyzed through the Charpy impact test. The results showed the addition of 20 phr silica filler in composite ES does improve the impact strength significantly (177%) compared to pure epoxy (Composite PE). This is due to the silica particle acted as fillers that reinforced the epoxy matrix in particle phase in size less than 44 µm. The fillers (silica particles) with micro-sized particle helped in providing a larger surface area to increase the surface-to-volume ratio. Thus, it increased the stress transfer between filler-matrix. Moreover, the fillers also exhibited a good interfacial adhesion in the polymer matrix that also helped in higher stress transfer between filler and matrix [6]. Composite UT had higher impact strength compared to composite ES due to the presence of kenaf fiber which allowed more energy absorption when impact.

Composites UT, 3T, 6T and 9T were fabricated using alkaline treated kenaf. The results improve the impact strength of epoxy composite form composites UT to 3T. Also, 3% and 6% of NaOH treatment of kenaf silica modified epoxy composites (composite 3T and 6T) provided a minor impact strength improvement compared with the untreated kenaf silica modified epoxy composite (composite UT).

Composite 3T exhibited the highest impact strength of 10.57 kJ/m$^2$ which improved by 187.73% in comparison with pure epoxy. It is because the continuous fiber of kenaf mat gives additional energy absorption during impact [16]. Despite that, composite 9T showed the lowest impact strength performance compared with composite UT, 3T, 6T, and 9T. It was probably due to 9% of sodium hydroxide treatment caused denature of kenaf fiber as the higher concentration of alkali might corrosive the fiber structure. The result is an agreement with the finding Fiore *et al.* (2017) that the formations of crack and by-products from the reaction of alkalization caused a lower ability of kenaf fiber to matrix subsequently reduces the interaction between the fiber with the matrix. Finally, the mechanical properties of the composite material are reduced [6].

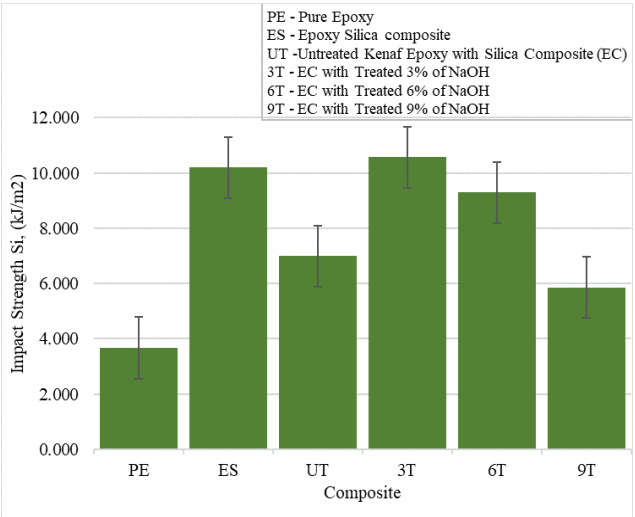



**Fig. 5:** Impact strength of silica modified epoxy 9Tcomposites

### 3.4. Flexural Test

Figures 6 and 7 show the flexural strength and moduli of Composites ES to 9T respectively. Flexural strengths of composites UT to 9T are commonly less than 54.1 MPa (Composite 3T) and similar to flexural strength was found for composites ES to 9T. The results also showed that composite 3T had the highest flexural modulus which was 3.5 GPa compared with composites ES, UT, 6T and 9T. This proved that the alkaline treatment of kenaf fiber mat in epoxy composites improved both flexural strength and flexural modulus. This is probably due to the presence of impurities on the surface of kenaf as it will cause a low ability to transfer stress from matrix to fiber. Therefore, microcracking might occur in the microstructure of composite that caused low the flexural modulus [6].

The result agrees with the findings by Yousif *et al.* (2012), which studied on the pre-treatment by using NaOH solution for kenaf fibers [17]. The composite 3T exhibited this result could be due to the concentration at 3% of NaOH did clean the kenaf surface effectively from lignin, cellulose, hemicellulose and other hydroxyl group components as compared to the higher concentration of sodium hydroxide. This can be proven as the flexural strength and modulus are the highest at composite 3T as the hydroxyl group components tend to acts as hydrophilic properties has been removed from kenaf fiber. This lead to an increase of the surface interaction between kenaf fiber and epoxy resin. Later, it causes higher ability of bond between fiber and matrix. Apart from that, the flexural strength was decreasing from composites 3T to 9T due to the higher concentration had denatured of the kenaf structure, where the high concentration of alkali solution caused corrosion to surface.

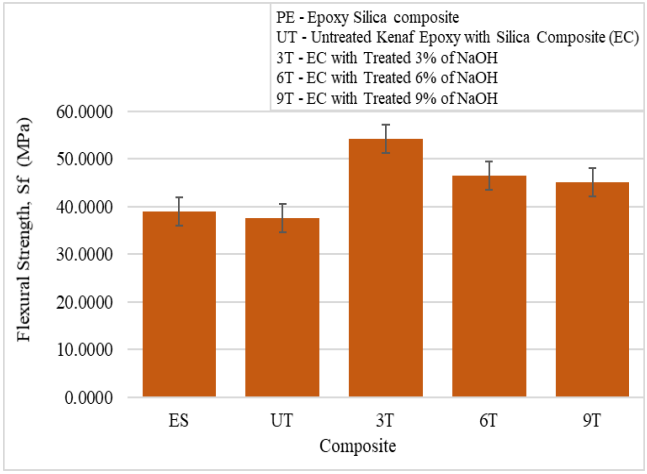



**Fig. 6:** Flexural strength of silica modified epoxy composites

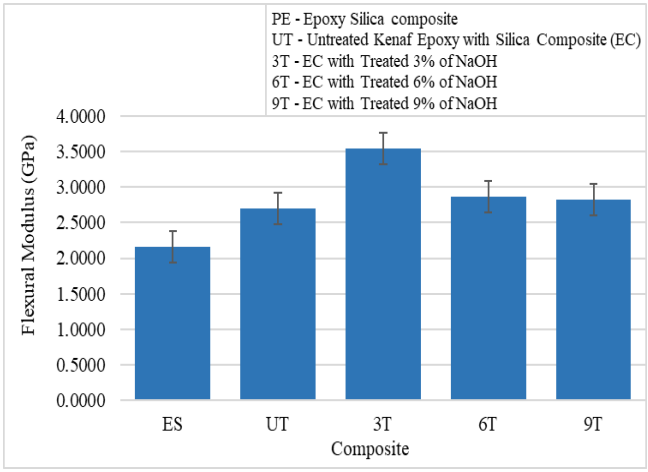



**Fig. 7:** Flexural modulus of silica modified epoxy composites

Overall results showed that kenaf treatment using NaOH solution improved flexural strength, flexural modulus and impact strength of silica modified kenaf epoxy composites. This is due to alkalization treatment did help to wash the fiber from the hydroxyl component which allowed a strong bond between the kenaf fiber surface and epoxy matrix. This also gave the natural fiber a strong polar and hydrophilic material apart from polymer materials, as well as allowed hydrophobicity [11]. Thus, the treatment helped in improving surface interaction between fiber and matrix. Moreover, the application of silica powder did allow plastic void growth around the debonded particles, which increased the diffi-

culty of crack propagation during fracture [18]. This cause a relatively high loading of silica particle which is up to 20 vol% that was required to achieve a reasonable toughening effect in epoxy composite, and it could also increase the viscosity of the resin mixtures [19]. Thus, alkaline treatment did improve in mechanical but the increase of alkali concentration during the alkaline treatment also reduced impact strength. Composite 3T denoted the best impact strength among silica modified epoxy composites. In short, 3% NaOH treatment is having the best impact strength value probably due to adequate concentration to remove almost all impurities and does not cause any denaturing of kenaf surface structure.

### 3.5. Morphology of Fracture Surface Analysis

Based on the results above, 3% of NaOH treated kenaf epoxy composite showed the highest impact and flexural strength with flexural modulus among those composites being tested. The impact strength showed a trend of decreasing from silica modified epoxy composite (composite ES) to untreated kenaf epoxy composite (composite UT) and then it is increasing reaching untreated to 3% treated kenaf epoxy composite (composite 3T). Therefore, only composites ES, UT, and 3T were being analyzed via SEM test.

Figure 8 shows the high magnification fracture surface images of impact specimens of composites ES, UT, and 3T. The silica particle can be seen as cloudy spherical particles in the matrix. Apart from this, silica at 20 phr is well distributed in the epoxy matrix which is compatible with the findings of Xue *et al.*, (2015) [14]. The surface in Figure 8(a) is smooth without dark circle formation. This is probably due to the silica was homogeneously distributed to the entire composite during preparation. The dark circles in the matrix indicate the void created by the fiber pull-out. The red arrow in Figures 8(b) and (c) show the unbroken fiber that pulled out from the opposite sample's surface. The clear pull out of the fibers shows the interfacial adhesion of the kenaf fibers with the low epoxy matrix. Based on the findings by Yousif *et al.*, (2012), the existence of dark circles shows the failure of composite that was contributed by the poor interfacial adhesion between fiber and matrix, instead of the strength of the fiber itself [17]. The density of dark circles in composite 3T is more than composites ES and UT, thus it shows it has a rougher fracture surface. This shown that Composite 3T created more voids in composite, lowered the efficiency of stress transfer. Hence, Composite 3T exhibited higher impact strength.

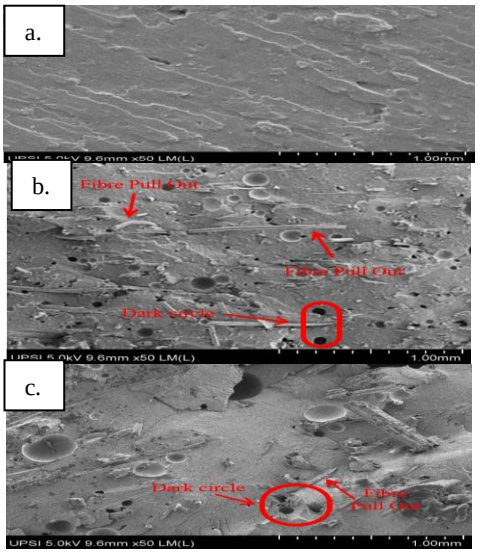



**Fig 8**: Higher magnification images of fracture surfaces of impact specimens (a) Composite ES (silica modified epoxy composite) (b) Composite UT (untreated kenaf silica modified epoxy composite) and (c) Composite 3T (3 % treated kenaf silica modified epoxy composite)

## 4. Conclusion

The fabrication of epoxy/silica/kenaf composites with various alkaline treatment concentration has been done. Flexural and impact strength increased from untreated kenaf fiber to 3% of NaOH solution of treated kenaf in epoxy composites modified silica. Beyond the 3% of NaOH solution of treated kenaf, epoxy composite shows the decreasing of flexural and impact properties. Epoxy/silica/kenaf composite with treated 3% of NaOH results in the highest impact strength, flexural modulus, and strength which 10.6 kJ/m2, 54.1MPa and 3.5 GPa, respectively. SEM analysis proved the alkalization treatment of kenaf with a higher concentration of NaOH has removed impurities from kenaf surface and also led to poor stress transfer subsequently caused to low mechanical properties. Therefore, 3% of NaOH was found the most optimum concentration. At 6% of NaOH, the fiber structure started to crack due to a higher concentration of NaOH and by-product residues formed on the surface as the reason were the reaction between cellulose components and NaOH. This led to reducing in the interaction between fiber and matrix. Consequence, stress transfer between fiber and matrix become poor and lower the mechanical properties. At 9% of NaOH, the crack became bigger and more residues were deposited on the surface of the fiber. Hence, the lowest mechanical properties to other composites were Composite 6T.

## 5. Acknowledgement

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