# FABRICATION OF PALM KERNEL SHELL EPOXY COMPOSITES AND STUDY OF THEIR MECHANICAL PROPERTIES

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***ABSTRACT***
*The solid waste from palm oil mill industry in Nigeria has been on the increase annually. This study thus investigates the utilization of Palm kernel shells (PKS) bio-agricultural wastes as a filler material for epoxy composite fabrication in which the filler was treated in an alkali solution. The composites were fabricated by employing filler particle size of 150μm and loadings of 5%, 10%, 20%, 30% and 40% using hand mixing technique. The composites were tested following appropriate ASTM standards for tensile, impact, hardness, morphological and water absorption properties. Fabricated composites showed good mechanical properties with 5-10% filler loadings attaining maximum values which improved on alkali treatment. The water absorption properties of the composites were not encouraging as saturation point was reached just after three days of immersion but, treatment with alkali reduced water absorption rate. SEM analysis of the fractured tensile specimens further highlighted the reason for the improved mechanical properties recorded by the alkali washed palm kernel shell composites due to the removal of natural impurities hence, providing larger surface area for mechanical interlocking. PKS composites therefore, have adequate properties for applications in the automotive industry as vehicle door and as partition panels and particle board products in building construction.*

***Keywords:*** *Epoxy, Mechanical properties, Palm kernel shell (PKS), Scanning Electron Microscope (SEM)*

## 1. INTRODUCTION

The physical and mechanical properties of cellulosic natural fibre in composite material have become subject of international research interests, most especially when such fibres are residues of agro industrial process with acceptable raw properties for composite fabrication [Li *et al.,* 2007; Reddy and Yang, 2005; Rowell, 1998]. The reason for this interest is not just from the technological and scientific point of view, but also social and economical, in terms of employment, cost and environmental concerns. Agricultural products such as: groundnuts, coconut and palm kernel are widely grown in the tropical countries like Nigeria, but less than 10% of the wastes generated are used domestically as fuel with the larger percentage of the wastes yet to find global sustainable application [Wang *et al.,* 2007]. In addition, studies have shown that the burning of composite components with natural fibres produced lower air emissions due to their reduced energy consumption when compared to equivalent synthetic fibre composites [Cicala *et al.,* 2010].

The solid waste from palm oil mill industry in Nigeria has been increasing annually where it has been reported that about 4.6 million metric tons of fresh fruit bunch is produced every year [Ohimain and Izah, 2014], which means that PKS is always available. Oil palm is an important tree because of the value of the crude palm oil, fronds, stems and leaves. Due to the magnitude of this industry, several residues are co-produced with palm oil. These include the empty fruit bunch, palm fruit fibre and palm kernel shell. The palm kernel shell is a waste byproduct from palm oil processing [Obeng *et al.,* 1997] and approximately 15 to 18 tonnes of fresh fruit bunches are produced per hectare per year with PKS comprising about 64% of the bunch mass [Adewumi, 2009]. In the developing world, waste PKS is either burned to supply energy at palm oil mills or left in piles to compost [Olumuyiwa *et al.,* 2012].

Some previous studies showed that PKS has potential for use as reinforcement in concrete (Alengaram *et al.,* 2008). PKS was recommended to replace aggregates of stone dust and bitumen in 10% blend with asphalt for heavily trafficked roads [Ndoke, 2006]. Also, a study reported that palm kernel shell has been used as friction material in the formulation of non-asbestos brake lining [Ibhadode and Dagwa, 2008].

The use of PKS as fillers in infrastructure materials is employed to fully take advantage of the natural properties that this composition provides and to reduce the cost of composite materials. In spite of this, not much work has been reported on the development of polymer matrix composites containing PKS as reinforcement. This study therefore, reports the use of PKS as a filler in epoxy composites and the analysis of some selected mechanical properties.

## 2. MATERIALS AND METHODS

### 2.1 Preparation of reinforcing Fillers

Palm kernel shells (Figure 1) obtained from the Central market in Sabon Gari Local Government Area, Zaria were washed and dried before grinding using a milling machine. The particulate powders were sieved by vibratory sieve shaker to obtain 150 μm particle size.

Figure 1: Raw as-received Palm Kernel Shell (PKS)

### 2.2 Treatment of palm shell powder

The powdered shell was divided into two portions. A portion was treated with 5% solution of sodium hydroxide for one hour at room temperature (27 ± 2 °C). It was then washed and decanted, after which acetic acid was added in drops in order to neutralize the alkali and a litmus paper used to test for neutrality. The procedure was repeated severally until a neutral pH was obtained, and then oven dried at 60 ºC for two hours. This was later sieved to the required size (150 µm) before composite fabrication. The second portion was left untreated.

### 2.3 Fabrication of composites

This was done using hand mixing method with a glass mould of 100 mm x 100 mm x 4.0 mm dimensions. The epoxy resin and hardener were mixed in the ratio of 2:1 according to supplier's instruction with filler loadings of 5%, 10%, 20%, 30% and 40%. The mixture was stirred at a relatively low speed to avoid bubbles until it became uniform at ambient condition (27 °C and 68% RH). The glass mould was covered with an aluminum foil (serving as mould release) to prevent the composites from sticking to the mould on removal after curing at room temperature.

### 2.4 Analysis of the Mechanical Properties of the Composites

After fabricating the composites, they were cut into various sizes for different ASTM standards using BOCSH motorized jigsaw (model: GST 85 PBE) ready for testing.

#### 2.4.1 Tensile strength determination

The test was conducted on the dog-bone shaped samples using Hounsfield Monsanto tensometer (model 9875) according to ASTM D638-10 with a gauge length of 33 mm on specimen dimension of 100 mm x 20 mm x 4 mm. Average value from five tested samples were used in each case.

#### 2.4.2 Water absorption test

The samples of 10 mm x 10 mm x 4 mm dimensions were cut, weighed and immersed separately in water at room temperature (27 ± 2 ºC). The samples were removed, dried and the weight taken after each day. The water absorption test was conducted according to ASTM D570-2010. The amount of water absorbed by the composites at room temperature was calculated using:

$$\%\ \text{water absorption} = \frac{\text{finalweight} - \text{initialweight}}{\text{initialweight}} \times 100$$

Eq. (1)

#### 2.4.3 Impact test

Impact test was carried out using the Izod impact testing machine according to ASTM D 256. Samples were tested in replicates of three each at ambient condition (27 °C and 68% RH) by a single swing of the pendulum hammer using a Resil Impactor impact tester (Model no. 6957, capacity of 25 joules). The specimen size was 100 mm x 15 mm x 4 mm. Each sample was placed on the vice and clamped firmly. The formula below was used to calculate the Izod impact strength of the samples, expressed in kilo Joules per square metre.

$$\text{Impact strength} = \frac{E_c}{hb} \times 10^3 \quad \text{Eq. (2)}$$

where

$E_c$ is the corrected energy absorbed by breaking the test sample in joules,

$h$ is the thickness of the test sample in millimetres and

$b$ is the width of the test sample in millimeters.

#### 2.4.4 Hardness test

Indentation test for composite samples was carried out according to the standards specified by ASTM-D2240 and EN/ISO 7619 using Shore A Durometre (Model - 5019). Each sample with dimension 100 mm x 15 mm x 4 mm was loaded and the indenter was lowered to make an impression on each sample at different spots. Average of three readings was obtained and recorded on five specimens tested per sample.

#### 2.4.5 Scanning Electron Microscope (SEM)

The morphology of the composites was investigated using a Phenom ProX SEM scanning electron microscope (SEM), at a voltage of 15 KV. Fracture surfaces of the specimen samples obtained from tensile testing were sputter-coated with gold prior to their observation.

## 3.0 RESULTS AND DISCUSSION

### 3.1 Fabricated composites

Figure 2 shows the fabricated composite sheets and the cut samples for some mechanical tests.


Figure 2: Prepared composites sheets (a) and cut samples for some mechanical tests (b)

### 3.2 Tensile Strength of Composites

The effect of filler loading and alkali treatment on tensile strength (T.S) of palm kernel shell epoxy composites is as shown in Figure 3, it was observed that tensile strength of the composites increased marginally before decreasing steadily with respect to the unfilled epoxy composite with a value of 40.3 MPa.

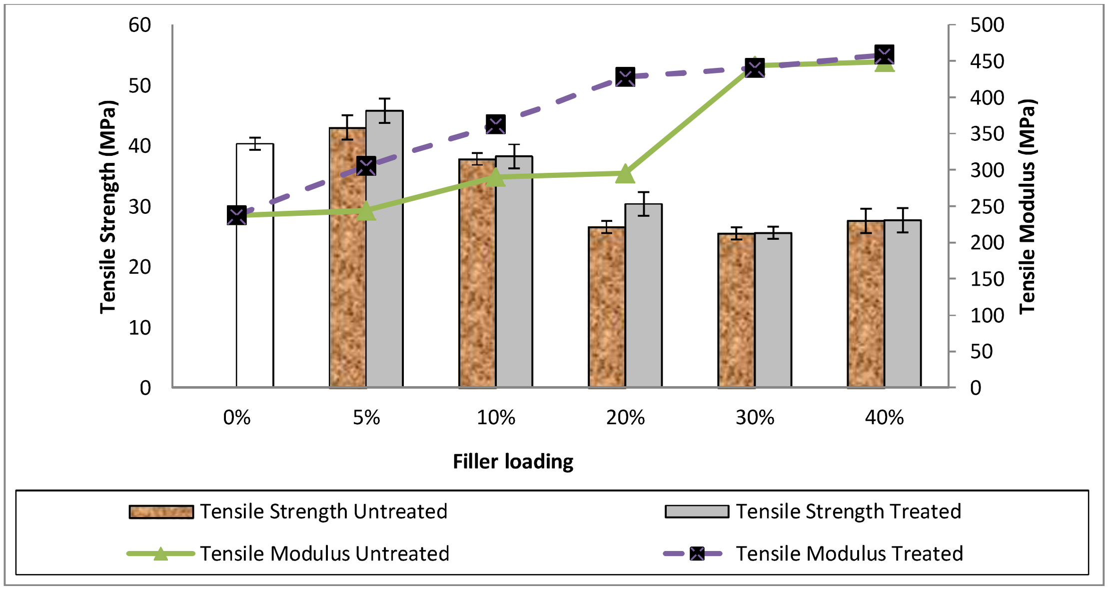


Figure 3: Effect of alkali treatment and filler loading on the tensile strength and modulus of PKS epoxy composites.

There could be good interaction between the filler and the matrix resulting into better stress transfer between the filler particles and the matrix and consequently, the enhancement in the tensile strength of the composite, a trend reported also by Abdul Khalil *et al.,* [2013]. 5% untreated and treated have the highest tensile strength of 43 MPa and 45.8 MPa respectively. Though, the difference in strengths for the 5% filler loading is marginal, but it is still visible. The decrease in strength at higher filler loadings above 5% could probably be due to poor filler–matrix interaction, weak interfacial bond, and the effect of the agglomeration of filler particles which then led to premature failure as shown by the SEM micrographs of Figure 8 for 10% filler loading.

Treatment with alkali has shown slight improvement in the tensile strength of the treated (T.S.T) composites with the 5% treated having an increase of 13% with respect to the unfilled epoxy and also values higher than the untreated (T.S.U). Similar finding was reported when the tensile properties of composites made from alkali-treated cellulosic reinforcements were superior to the untreated ones [Achukwu *et al.,* 2015]. Increase in filler loading probably increased the micro spaces between the filler and matrix which weakened the filler matrix interfacial adhesion. As reported by several authors; tensile strength of particulate composites generally decreases with filler content and it follows a power law in the case of poor filler matrix bonding [Bikiaris *et al.,* 2005; Sun *et al.,* 2006].

### 3.3 Tensile Modulus of Composites

Figure 3 shows that there was an observed increase in the tensile modulus of treated and untreated palm kernel shell epoxy composites, which simply means that tensile modulus increased with increasing filler loading. The incorporation of PKS was found to increase the tensile modulus with respect to the unfilled epoxy composite. A minimum tensile modulus for filled composites was obtained at 5% loading with values of 305.3 MPa and 244.3 MPa for treated and untreated fillers respectively. The unfilled epoxy has the lowest tensile modulus of 237.1 MPa. It has also been reported that the uniform dispersion of filler in the matrix decreases the inter-particle distance or free space between the particles which leads to the reduction in the flexibility of the polymer chain, thus, increases the tensile modulus [Ayatollahi *et al.,* 2011].

### 3.4 Impact Strength of Composites

The impact strength of the fabricated composite specimens as plotted against the filler loading is shown in Figure 4. The unfilled epoxy composite had the highest impact strength of 10.5 kJ/m$^2$.

There was an increase of 29% and 13% in impact strengths at 10% filler loading for the untreated and treated respectively, with respect to the 5% loading. Considering the error bar limits, alkali treatment has no significant effect on the impact strength except at 5% filler loading which is really marginal. Further increase in filler loading led to steady decrease in the impact strengths of the composites. The 10% filler loading recorded the best performance in impact strength. Impact property is thus found to be directly related to its overall toughness which is highly influenced by the nature of the constituent material as also reported by Joseph *et al.,* [2003].

### 3.5 Hardness test

The hardness strength of the fabricated composite specimens as plotted against the filler loading is shown in Figure 5. It can be observed that the inclusion of PKS generally has beneficial effect on the hardness of the unfilled composite for up to 10% with respect to the unfilled composite. There was no observed increase in hardness as filler loading increased beyond 10%. This could be due to increase in the percentage of the hard phase of the filler particles compared to the matrix system thus, reducing stress transfer and consequently the hardness value.

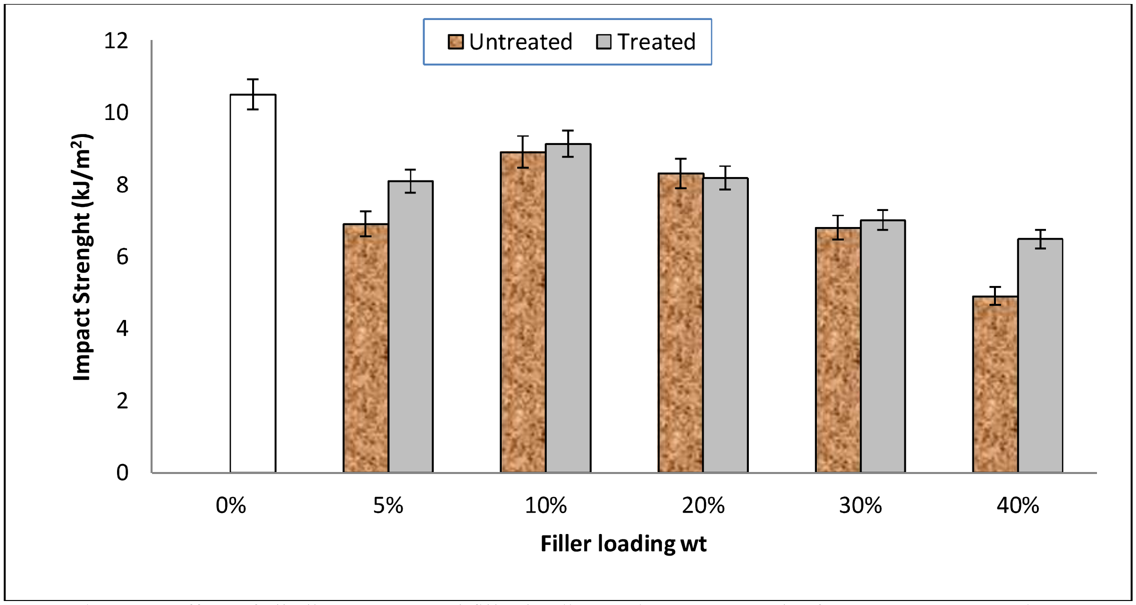


Figure 4: Effect of alkali treatment and filler loading on impact strength of PKS epoxy composites.

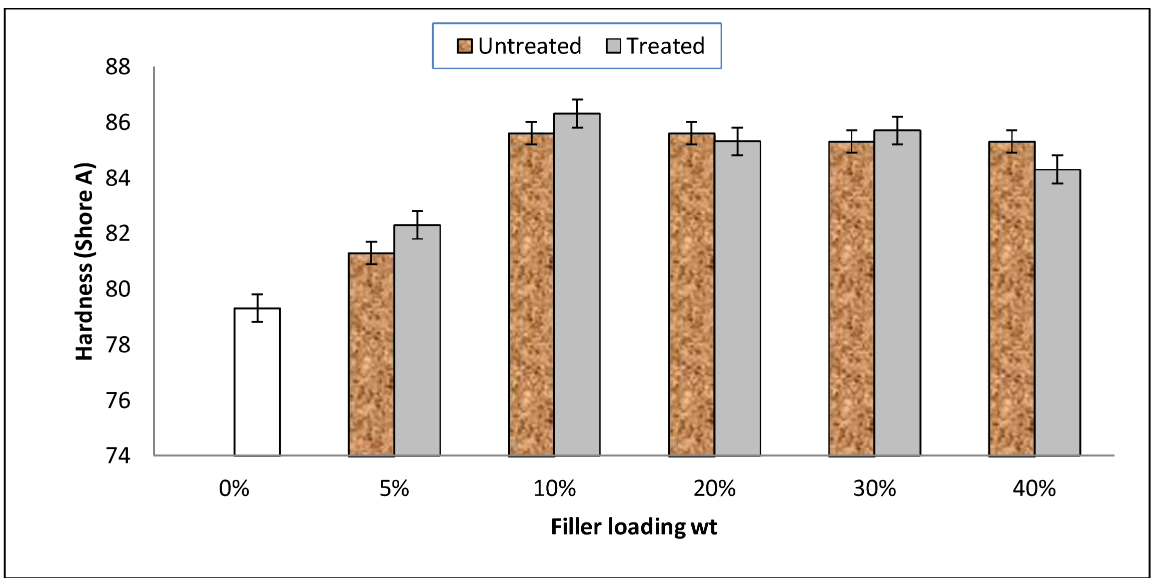


Figure 5: Effect of alkali treatment and filler loading on the hardness properties of PKS epoxy composites.

The alkali treatment had no significant improvement on the hardness of the fabricated composites considering the deviations from the mean values shown by the error bars, except for the 5 and 10% filler loadings. Achukwu et al., (2015) in their previous research with waste soda-lime glass particles reported that increase in filler loading improved the hardness of unsaturated polyester composites to a certain degree before decreasing for the particle sizes of 150µm. In comparison with the unreinforced matrix, it can be said that the introduction of palm kernel shell improved the hardness values of PKS epoxy composites.

**3.6 Water Absorption for untreated and treated palm kernel shell epoxy composites**

The graphs of water absorption properties are shown in Figures 6 and 7. In this study, there was an observed increase in water absorption of the PKS composites as the filler loading increases relative to the unfilled epoxy composite (Figure 6). 40% filler loading recorded the highest water absorption of 1.6% while the epoxy composite without any PKS recorded the lowest water absorption of 0.6%. The behaviour of polymer-filled composites to water absorption at a particular environmental condition has been found to be determined by many factors, such as processing techniques, matrix filler characteristics, composition of the composites, and duration of immersion in water [Ramakrishna *et al.*, 2006].

As can be seen, water absorption increases with immersion time, reaching saturation point in 3 days after which the composites were no longer absorbing water. Increase in filler loading of cellulosic content has been found to lead to high concentration of the hydroxyl group and voids in the composites which results in an increase in water absorption [Abdul khalil *et al.*, 2001]. Furthermore, the hydrophilic nature of the fillers causes the increased water uptake due to the formation of hydrogen bonds between the palm kernel fillers and water molecules. Thus, increased filler loading will increase the formation of hydrogen bond between the filler and water molecules.

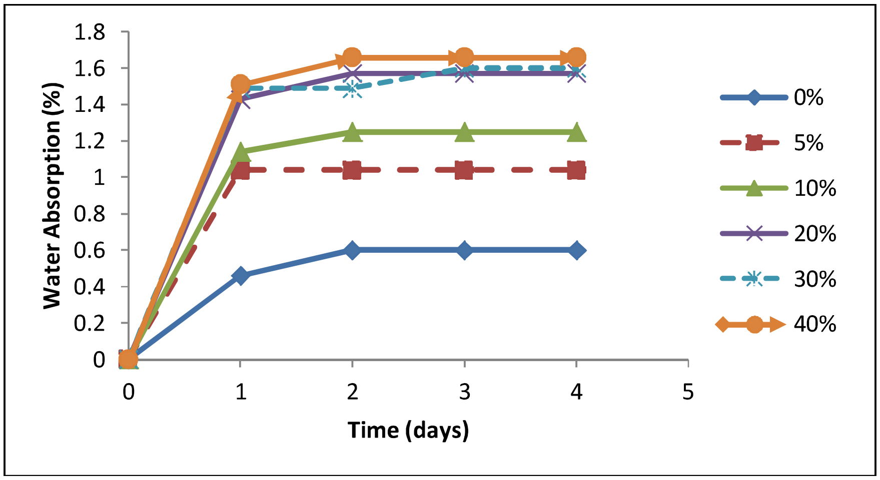


Figure 6: Effect of alkali treatment and filler loading on water absorption of treated PKS epoxy composite.

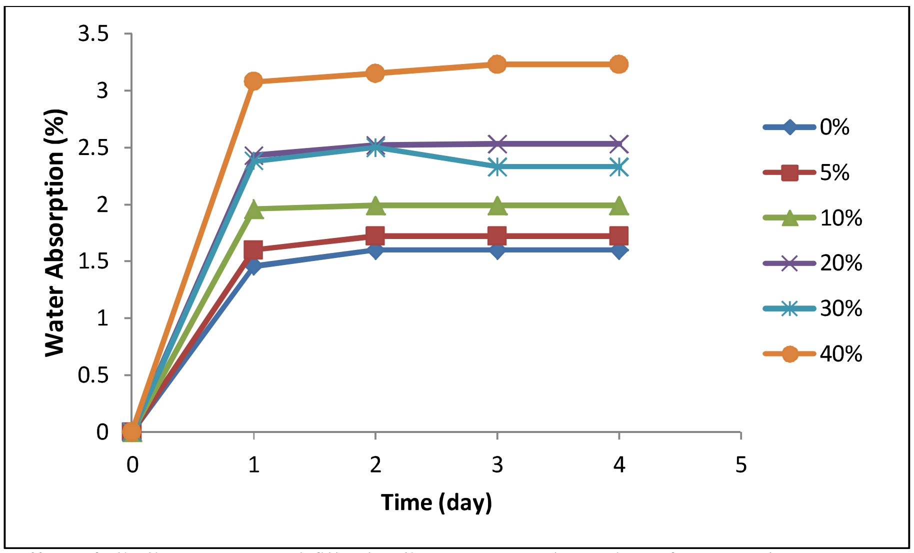


Figure 7: Effect of alkali treatment and filler loading on water absorption of untreated PKS epoxy composite

For the untreated fillers (Figure 7), 40% has the highest water absorption of 3.2% while the unfilled epoxy has the least water absorption of 1.6%. The untreated filler tends to absorb more water making the composite to have higher moisture content compared with the treated, similar behaviour reported by Abdul Khalil *et al.,* [2011].

In this study, alkali treatment shows a significant positive impact on the water absorption behaviour of the composites, because it washed the impurities and replaced most of the available hydroxyl ions that are water loving leading to reduction in the amount of water absorbed. Figures 6 and 7 followed Fick's law which states that the local rate of transfer of solute per unit area per unit time is proportional to the concentration gradient of the solute, and defines the proportionality constant as the diffusivity of the solute.

**3.7 Scanning Electron Microscopy**

Figure 8 (a-d) shows the micrographs of fractured tensile specimens taken at different magnifications using the 10% filler loading as a representative sample (having relatively highest property values). It can be clearly seen that the treatment has actually washed clean the palm kernel shell (b), contrary to the dark patches seen in the untreated palm kernel shell composites (a). Natural occurring matters (lignins and pectins) and dirts have also been removed from the palm kernel shell prior to composite fabrication. This treatment effect is evidenced by the improved mechanical properties recorded by the alkali washed palm kernel shell composites. Similarly, the fractured surface morphology of the 10% untreated at higher magnification of 1000x (c and d), further revealed the presence of impurities which could be wax or pectin and other impurities in the surface of untreated fillers. These observations could be the reason for relatively poor interfacial adhesion in the untreated composites when compared with treated composites and support the increased mechanical performances and observed bonding nature in the treated composites.

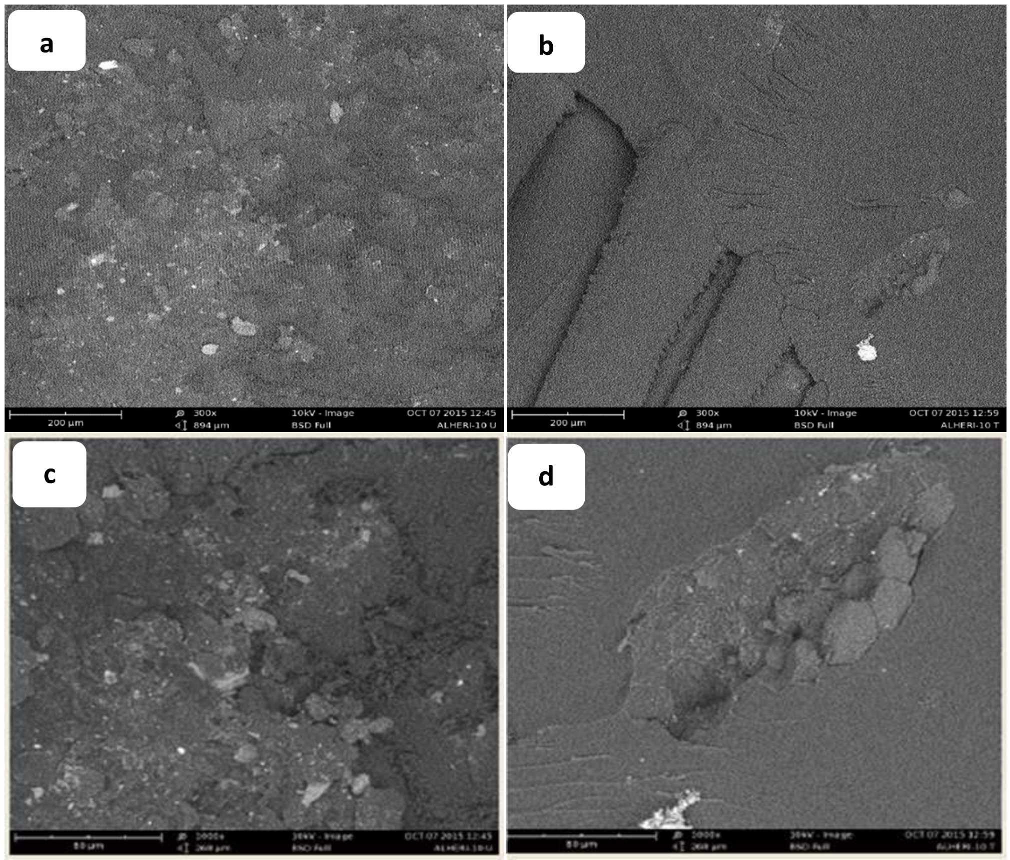


Figure 8: SEM micrographs of tensile fractured specimens showing effect of alkali treatment for 10% filler loading: (a) untreated at 300x magnification (b) treated at 300x magnification (c) untreated at 1000x magnification (d) treated at 1000x magnification

## 4.0 CONCLUSIONS

At the end of this study, it can be concluded that:

Mechanical properties of palm kernel shell (PKS) epoxy composites encouraged the use of agro waste materials as reinforcement to create new composite materials with reasonably good mechanical properties thereby helping to reduce environmental pollution.

The effect of alkali treatment and filler loading on the mechanical properties of PKS epoxy composites revealed that the alkali treatment only had weak or marginal improvement on the physical and mechanical properties. This is further explained by the result of the scanning electron microscopy (SEM). Filler loading increase was found to have marginal beneficial effect on the selected mechanical properties of palm kernel shell epoxy composites up to 10%.

Finally, based on the properties of fabricated composites, PKS as fillers have adequate properties for applications in the automotive industry vehicle inner panels and in in-door building construction as partition panels and particle board products.

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