Modified palm kernel shell fiber/particulate cassava peel hybrid reinforced epoxy composites

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ABSTRACT

This work was carried out to investigate the influence of chemically treatment palm kernel shell fiber (PKSF) and particulate cassava peel (PCP) as hybrid reinforcements on some selected mechanical properties and wear behavior of PKSF/PCP hybrid reinforced epoxy composites. Open mould technique was used for the composites developed by incorporating fixed amount of PKSF in both treated and untreated conditions into varied proportions of epoxy and PCP, respectively. The cured samples after 28 days were subjected to tensile, flexural and wear tests. Porosity of the composites were estimated by considering the experimental and the theoretical densities of the composite samples. The fractured surface morphology was investigated with Scanning Electron Microscopy (SEM) while X-Ray Fluorescence (XRF) was used to determine the elemental constituent of the cassava peel. From the results, it was discovered that, chemically treated PKSF/PCP hybrid reinforced samples performed better than the untreated PKSF/PCP hybrid reinforced counterparts in most of the properties considered. The addition of chemically treated PKSF/PCP hybrid reinforcement into the epoxy matrix brought about some enhancement in the stiffness of the developed composites which made them have better resistance to deformation under different loading conditions for the estimation of young's modulus of elasticity, flexural strength at peak and wear. Optimum values were obtained from the addition of 6 wt.% PCP reinforcement composition.

1. Introduction

The emergence of polymers in the beginning of the 19th century ushered in a new era of research. At the same time, interest in the synthetic fibers due to its superior dimensions and other properties gained popularity and hindered its replacement by the natural fibers in areas of applications. However, diversification in the use of raw materials, large quantum of energy and the environmental pollution generated during the production of synthetic materials now divert the attention of researchers towards natural fibers due to their distinct advantages. Natural fibers are renewable raw materials, biodegradable with relatively high strength and stiffness. Their low-density values encourage the production of composites that combine good mechanical properties with low specific mass. A number of natural fibers and their derivatives, such as jute [1], kenaf [2], sisal, palm kernel shell [3], coconut shell [4], and bagasse [5–8], have been considered as reinforcements in polymer matrix composites with outstanding performances.

Despite the benefits offered by natural fiber/particulate reinforced composites, incompatibility between the polymer matrix and reinforcements surface has been identified as major challenge in the processing of polymer composites containing natural fiber/particulate reinforcements [9]. This results in wetting problem, weak interfacial adhesion, and limited stress transfer between the two interfaces, with great consequences on the properties of the developed composites. However, some treatment protocols have been developed to alleviate this challenge, which include the use of compatibilizers [10], coupling agents [11], and mercerization treatment [9] among many others.

Composites are used to raise performance level, address traditional material design limitations and also enable the development of new product solutions. Composite materials are found in many of the products used in our daily lives such as cars, bath tubs and counter tops, boats and windmills. However, from all the available matrixes, reinforced polymer

matrix composites have many applications as a class of structural materials because of their ease of fabrication, relatively low prize and higher mechanical properties. Thus, the renewed interest in the natural fibers has resulted in a large number of modifications to bring it at par and even more superior to synthetic fibers. Because of such tremendous changes in the quality of natural fibers, they are fast emerging as a reinforcing material in composite development.

Polymer matrix composites can be reinforced with either particulate or fibrous materials from synthetic or natural sources and can be used in combined (hybrid) form. Commonly used particulate fillers include talc, calcium carbonate, kaolin and silica. While particulate reinforcement materials impact strength in all directions, fibers usually offer unidirectional reinforcement. However, their incorporation in polymers leads to increase in stiffness, higher resistance to distortion by heat, low shrinkage and coefficient of thermal expansion as well as high resistance to permeation of gases and liquids [12].

Epoxy Resins are thermosetting resins, which cure by internally generated heat. Epoxy systems consist of two parts, resin and hardener. When mixed together, the resin and hardener activate, causing a chemical reaction, which cures (hardens) the material. They can be blended with various reinforcements to develop new composite materials for special applications.

Various research has been carried out to improve the mechanical properties of polymers for better service application due to its low tensile and impact strength by reinforcing them with various particles and fibers. Chandramohan et al. carried out studies on natural fiber reinforced polymer composite for automobile accessories [13]. The tensile loading condition showed a brittle-like failure due to less fiber pull out leading to reduction on the tensile strength. In this research, attention was based on producing hybrid polymer composite material having improved tensile, bending/flexural and wear properties for interior automobile accessories. Epoxy was reinforced with readily available biodegradable agro-wastes from palm kernel shell and cassava peel. The research was carried out to meet the need of increasing the percentage of biodegradable materials in automobile and to encourage sustainable environmentally acceptable production technology.

2. Materials and methods

2.1. Materials

Epoxy resin, Hardener, Sodium hydroxide (NaOH), Hydrochloric acid (HCl) and Distilled water were procured from chemical store in Akure, Ondo State, Nigeria. Palm kernel shell fiber was obtained from palm mill in Edo State, South–Western part of Nigeria while the cassava peel was procured from the farm plantation in Auchi, Edo State, Nigeria.

2.2. Methods

2.2.1. Processing of the agro-based reinforcement materials

Both agro wastes were washed in flowing water to remove the dirt before sun drying for 3 days. The dried materials were divided into two parts; one part was subjected to chemical treated and the other part was left untreated. The treatment was carried out with 1 M solution that was obtained from the mixture of HCl and NaOH in 75:25% (representing; 0.75 M HCl + 0.25 M NaOH) proportions before the treatment. The fibers were completely submerged in a beaker containing 500 ml of the reagent prepared by mixing 357 ml of 0.75 M HCl and 125 ml of 0.25 M NaOH solution. This was then placed in the shaker water bath maintained at 50 °C for 4 h. The treated fibers were then removed from the beakers and washed with tap water and rinsed with distilled water until test from litmus paper indicated pH of 7.0 which signify neutral status. The treated fibers were made to dry at room temperature in the laboratory for 5 days. The average length of the PKSF used was 10 mm. The treated and untreated PKSF were as shown in Fig. 1(a and b). These same treatment procedures were repeated for the cassava peel before they were pulverized into particles using laboratory ball mill. Treated and untreated ball milled cassava peels were further processed by sieving in sieve shaker to obtained particle size of <75 μm. The treated and untreated cassava peel particles were as shown in Fig. 2(a and b).

2.2.2. Composite development

The matrix and the reinforcement materials were weighed into various weight fractions using the electronic weighing balance. The samples for the hybrid untreated PKSF/PCP were weighed for each sample with 2, 4, 6, 8, and 10% weight fractions as shown in Table 2. The same procedure was repeated for treated samples. The volume of the epoxy resin and the hardener used for each sample varies with changes in the percentage of the particulate cassava peel and decreases as the volume of the particles increases. A constant percentage of the palm kernel shell fiber was used.

2.2.2.1. Compounding of the samples. Manual mixing of the samples was used to attain homogeneous mixture. The pulverized cassava peel and the fiber were mixed with the measured epoxy and hardener which was stirred vigorously using the glass rod stirrer for 2 min to attain homogeneity. The mixed materials were quickly poured into the mould to prevent premature curing and well filled with accurate measurement to avoid non-uniform dimensions. The homogenously mixed sample compositions were poured into the tensile, flexural and wear molds to form the required shapes and allowed to stay for the required curing time. Three samples were produced for each representative composition as shown in Fig. 3(a–c).

2.3. Mechanical tests

2.3.1. Measurement of tensile properties

Tensile test was carried out in accordance to American Standard Testing and Measurement Method D412 (ASTM D412-16) [14] on Instron Universal testing machine to determine the ultimate tensile strength and young's modulus of elasticity and elongation at break of the samples. Composite samples with 3 mm thick and of gauge length 150 mm were used. Three identical samples were tested for each weight fraction from where the average values were used as the representative values.

2.3.2. Measurement of flexural strength at peak

Three-point bend tests were performed in accordance to ASTM D790-17 [15] to measure the flexural strength at peak of the developed composites using Instron Universal testing machine. The samples were of 150 × 50 × 3 mm. Three samples were tested for each weight fraction used and the average values were taken to represent the actual values.

2.4. Measurement of wear property

The abrasive wear test was carried out using Taber abrasion testing machine according to ASTM F732-17 [16]. It involves mounting a specimen to a turntable platform that rotates at a fixed speed of 1000 rpm for 20 min under the influence of applied specific pressure which is lowered onto the specimen surface. A rub-wear action (sliding rotation) is produced on the surface of the test piece and the resulting abrasion marks form a pattern of crossed arcs in a circular band. The samples were measured using an analytical weighing balance to take the initial weight of the samples before and after mounting. The difference between the initial and the final values was noted and recorded against each samples. The amount of wear is determined by the weight loss. The average values from each sample were used as the representative values.

Fig. 1. (a) Treated palm kernel shell fiber (b) Untreated palm kernel shell fiber.
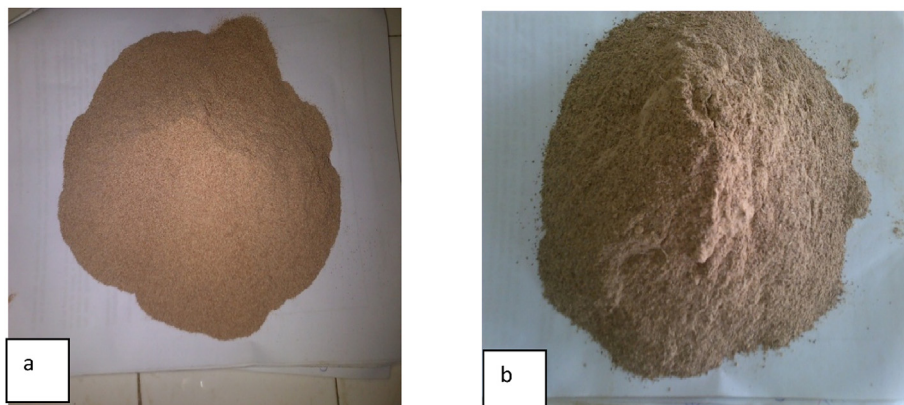

Fig. 2. (a) Treated pulverized cassava peel (b) Untreated pulverized cassava peel. The elemental composition of the particulate cassava peel as revealed by X-Ray Fluorescence was as shown in Table 1.

Table 1
X-ray fluorescence analysis of the treated cassava peel.

Elements	Conc. Value	Conc. Error	Unit
K	0.6233	±0.0081	wt.%
Ca	1.3909	±0.0051	wt.%
Ti	0.0879	±0.0100	wt.%
Cr	2.9	±1	Ppm
Mn	16	±24	Ppm
Fe	2.4431	±0.0112	wt.%
Ni	18	±3	Ppm
Cu	11	±1	Ppm
Zn	89	±12	Ppm
Ga	20	±10	Ppm
As	3	±2	Ppm
Rb	17	±4	Ppm
Sr	20	±14	Ppm
Se	15	±3	Ppm
Zr	112	±29	Ppm
V	92	±12	Ppm

From the analysis, Fe and Ca that dominate the constituents are the most likely elements that aid the enhancement of the developed composites.

Table 2
Proportion of the Constituents added in grams.

Weight Fraction (%)	PCP (g)	PKSF (g)	Hardener (g)	Epoxy Resin (g)
2	9	4.5	145.5	291
4	18	4.5	142.5	285
6	27	4.5	139.5	279
8	36	4.5	136.5	273
10	45	4.5	133.5	267
Control	–	–	150.0	300

where; ρc-density of composite; W_f-weight fraction of the composition; ρepoxy-density of epoxy; ρPKSF-density of palm kernel fiber; ρPCP-density of pulverized cassava peel.

The experimental density was evaluated by weighing the test samples using a high precision electronic weighing balance. The measured samples in each case were divided by the volume of the respective samples.

The percent porosity of the composites was determined using equation (2).

$$\% \, Porosity = \{(\rho T - \rho EX) \div \rho T\} X 100 \qquad (2)$$

where; ρT-theoretical density (g/cm^3) and ρEX-experimental density (g/cm^3).

2.5. Density measurement

Density measurements were carried out to determine the porosity levels (void content) of the composites produced. This was achieved by comparing the experimental and theoretical densities. The theoretical density of the samples was evaluated by using the rule of mixtures as shown in equation (1).

$$\rho c = (Wf \, x \, \rho epoxy) + (Wf \, x \, \rho PKF) + (Wf \, x \, \rho PCP) \qquad (1)$$

2.6. Scanning Electron Microscopic examination of the composites Fractured Surfaces

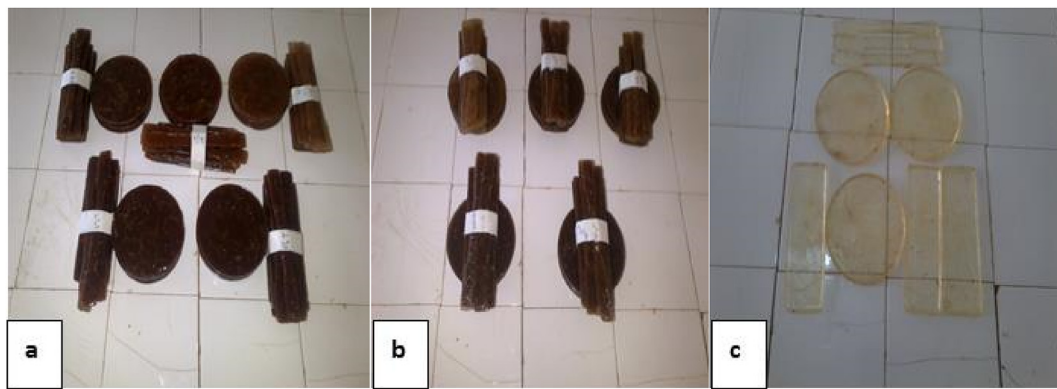The surface morphology of the samples was studied using an AURIGA

Fig. 3. Composites from (a)Treated Samples (b) Untreated Sample (c) Control sample.

Scanning Electron Microscopy (Carl Zeiss, Germany) with an accelerating voltage of 15 kV. The tensile fractured surfaces of the moulded samples were mounted on aluminium stubs and were sputter coated with gold using EMITECH K950X sputter coater before being subjected to Scanning Electron Microscopic analysis.

3. Results and discussion

3.1. Tensile properties

3.1.1. Ultimate tensile strength

The response of the materials to ultimate tensile strength was as shown in Fig. 4 where it was seen that 6 wt.% reinforcement from treated hybrid composites gave optimum value close to the control sample. While the control sample was with an ultimate tensile strength of 24.6 MPa, the best from the composite samples was with a value of about 23.59 MPa with a marginal difference. Since tensile strength happened to be a property that has to do with direction, and these reinforcements are randomly dispersed, this observed behavior is likely to ensue. However, it was noticed from the results that the tensile strengths were higher for the treated composites than the untreated composite samples in all the weight fractions considered with the exception of 2 wt.% composition. This suggests that at low weight fractions, untreated PKSF/PCP hybrid reinforcement produced better tensile strength than the treated ones. However, above this quantity, the treated agro-wastes offered better tensile strengths values. The trend of results for untreated reinforced composites showed that the tensile strength decreases as the reinforcement content increases whereas the strength increases as the reinforcement content increases for the treated samples up to 6 wt.% before experiencing a decrease in the property. The general representation of the results revealed that composites developed from treated agro-based
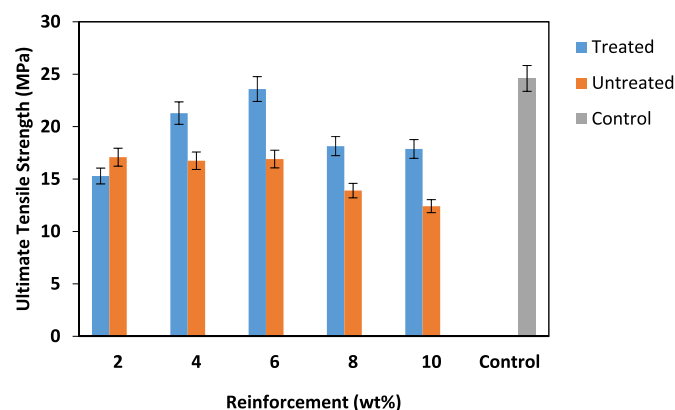
materials possess better tensile strength than those developed from untreated ones. This implies that there is potential in treating agro-based reinforcement materials with alkaline solutions which is in agreement with previous works by Ref. [17]. Plant fibers in general are hydrophilic in nature as they are derived from ligno-cellulose, which contain strongly polarized hydroxyl groups, therefore, making their rate of water absorption to be high. This high-water absorption rate leads to the degradation of the fibers and the fiber–matrix interface resulting in a loss of mechanical performance. Also, Vasile et al. [18] concluded in their report that the loss in the mechanical properties was due to the plasticity of the matrix by water and degradation of the fiber/matrix interfacial bond due to moisture swelling of the matrix. Since the lignin keeps the water in fibers [19], therefore, to reduce the water absorption rate of composite materials the lignin content of the fiber has to be reduced and this could be achieved by the pre-treatment of the fiber with chemicals that can help in reducing the lignin content.

3.1.2. Young's modulus of elasticity

Fig. 5 presents the behavior of the materials in terms of modulus. Similar trend to the tensile strength was observed but with enhanced results compared to the control sample. Contrary to what was obtained in tensile strength results in Fig. 4, all the composites were seen to possess better modulus than the control sample. The sample with best modulus was 6 wt.% PKSF/PCP treated reinforced epoxy composite with a value of about 427.97 MPa followed by composite sample from 2 wt.% untreated PKSF/PCP with a value of 406.12 MPa. Similar to the response of this weight fraction in Fig. 4, sample from 2 wt.% untreated PKSF/PCP hybrid reinforced epoxy composite was the best among the untreated hybrid reinforced composites. This also corroborate the point that low weight fraction from untreated agro-waste promote better tensile properties compared to the treated ones. The results revealed that, fiber surface modification by this treatment might likely favor higher loading of the reinforcements for improved properties. The treated PKSF/PCP reinforced composites gave better results in all the variations considered except at 2 wt.% that happened to be the best from untreated PKSF/PCP reinforced composites. The feat was due to the surface modification that has taking place as a result of the chemical treatment in which the lignin that serves as the matrix in the untreated PKSF and PCP have been washed, thereby, exposing the cellulose as a rough surface. This was in agreement with the submissions of [20,21]. The rough surfaces aid proper bonding between the treated agro-based materials and the epoxy matrix. Also, as shown in Table 1, the presence of Iron (Fe) and Calcium (Ca) in higher proportion in the PCP contributed to the enhancement observed. The modulus from the control sample was 333.22 MPa and when compared with composites with the best results, culminated to 28 and 22% enhancement, respectively. This observed enhancement may be due to the inclusion of the reinforcements that tend to stand as obstacle to the movement of dislocation as the load is being transfer from the matrix to the reinforcements in form of particulate and short fiber. These
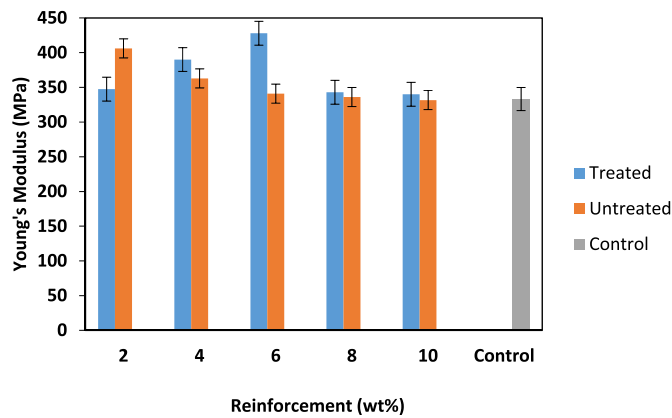

Fig. 4. Plots of the Variation of Ultimate Tensile Strength of Epoxy Resin and its Composites.

Fig. 5. Plots of the Variation of Young's Modulus Epoxy Resin and its Composites.

Fig. 6. Plots of the Variation of Flexural Strength at Peak of Epoxy Resin and its Composites.

reinforcements obstruct the dislocation movement, thereby, increasing the straining and stiffness of the materials before fracture and failure contrary to the control where there is nothing to aid or support the load bearing as well as distribution. Study on the effect of fiber length and fiber content on mechanical properties of short basalt fiber reinforced polymer matrix composites showed that fiber size and fiber content influence the mechanical properties of the composites [22]. Considering the emerging potentials in this research for the agro-based reinforcement materials, it is therefore, expedient to adopt the use of these materials as alternative to the more toxic, exorbitant and non-environmentally friendly synthetic materials that are commonly used as reinforcement in epoxy and allied matrixes. The formation of hybrid from fiber and particles are utilized to take advantage of the best properties of each, and minimize their weaknesses. In, this research fixed amount of PKS fiber was used with varied particulate cassava peel content. The PCP was to compliment the PKSF strength and toughness with stiffness. PKSF has been established as a potential reinforcement material for the enhancement of the mechanical properties of composites [23].

3.2. Flexural strength at peak

The flexural test measures the force required to bend a beam under three-point loading conditions. The data is often used to select materials for parts that will support loads without flexing. The results were as shown in Fig. 6 from where it can be seen that all the composites possess better flexural strengths than the control. The observed trend was similar to Fig. 5 in which all the developed composites were observed to possess better property than the control. Though, there was improvement in the flexural strength in Fig. 6, where it was discovered that the initial increase from 2 to 6 wt.% was followed by rapid decrease from 8 to 10 for both treated and untreated PKSF/PCP hybrid reinforced composites. From the results, 6 wt.% treated PKSF/PCP reinforced composite possess the best result with a value of about 74.16 MPa followed by 4 wt.% treated PKSF/PCP reinforced composites with a value of about 66.41 MPa compared to the control sample with a value of 38.69 MPa. The reasons for this performance was stated in the discussion of Fig. 5. The percentage enhancements were 92% and 72%, respectively which implies an improvement over the tensile properties as obtained in Figs. 4 and 5. These agro-based materials show a promising performance if employed for engineering applications.

3.3. Wear behavior of the composites

The wear behavior of the different materials in comparison with the control was as shown in Fig. 7. Abrasion wear test was carried out to determine the influence of the reinforcements on the wear loss at material surfaces. The results revealed that these reinforcements led to
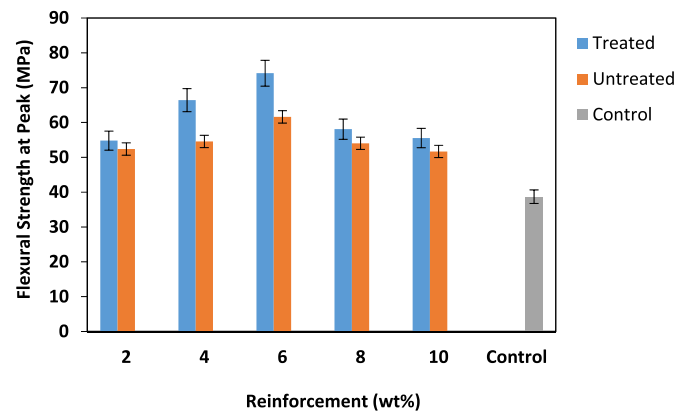
differences in the abrasive nature of the developed composites with respect to epoxy resin that serves as control. All the developed composites have higher wear resistance than the control sample. It implies that in every areas of application for epoxy material where abrasive wear at the surface are prevalent, the addition of these agro-based materials which are organic materials can aid the improvement of the wear resistance. The development of durable, low cost and environmentally friendly materials are currently been sought for globally. Presently, cassava peels have not been employed in composite development; they are only being used as either manure or animal feeds. But in most cases, they remain waste materials. Common trend in a reversed order was observed for the treated and untreated hybrid reinforced composites with best emerging from the two extremes. It was observed that untreated PKSF/PCP reinforced composites possess better wear resistance than the treated PKSF/PCP reinforced composites except at 10 wt.% that gave the overall best wear resistance with a value of about 0.0142 g. The best from untreated PKSF/PCP reinforced composite was with a value of about 0.0153 g from 2 wt.%. Samples from 6 wt.% treated and untreated PKSF/PCP reinforced composites possess the least wear resistance among the composites. However, sample from 6 wt.% treated PKSF/PCP reinforced composites with a value of 0.037 g still compete favorably with the control sample with wear capacity of 0.1324 g which culminated to 96% enhancement. The presence of Fe and Ca in the PCP might be part the reasons for the enhancement in the abrasive wear resistance of the developed composites.

3.4. porosity of the composites

The amount of porosities in the developed composites was determined and presented in Fig. 8. Since polymer composite developments are associated with the presence of voids and pores, there is need to estimate the number of pores that are present in any developed composites so as to be able to deduce the reason(s) for any observed potentials in the developed composites. From the results in Fig. 8, the untreated PKSF/PCP reinforced composites were seen to possess more voids or pores than the composites from treated ones. This may be due to the influence of the chemical treatments on the agro-based reinforcement materials which might have restructured their surfaces for effective interfacial bonding with improved bonding strength. Proper bonding will lead to reduction in the amount of porosity in the composites. The results showed that the porosity reduces as the untreated PKSF/PCP content increases while it increases as the treated PKSF/PCP increases. However, the highest porosity from treated PKSF/PCP reinforced composites was almost the same with least from the untreated PKSF/PCP reinforced composites.

The porosity content of the samples can help in determining the mechanical properties of composite materials. The higher the porosity, the weak the mechanical properties, and from the mechanical properties
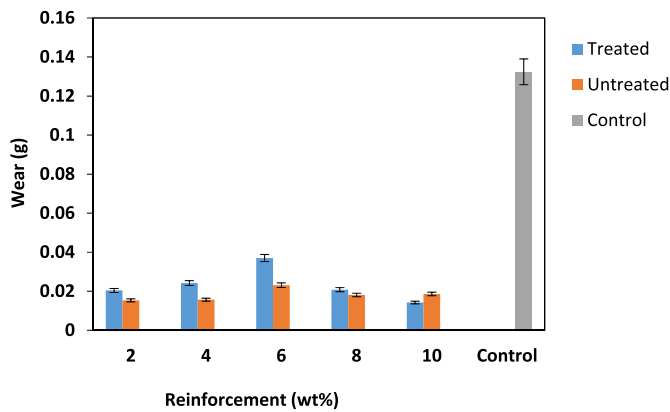
Fig. 7. Plots of the Variation of Wear Characteristics of Epoxy Resin and its Composites.
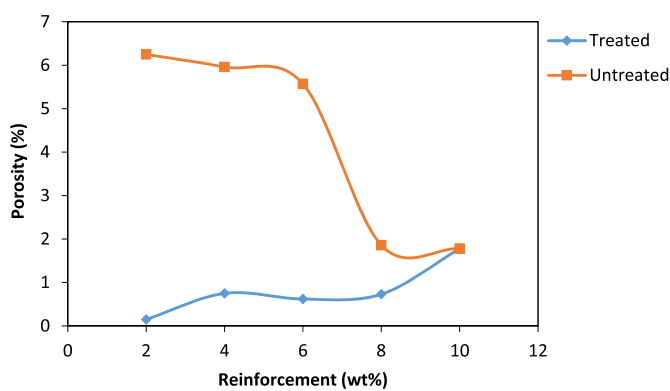

Fig. 8. Plots of the porosity content of the composites.

examined in Figs. 4–6, it was seen that, the untreated PKSF/PCP reinforced composites possess the least values in most of the results. This is an indication that, their higher porosity content as observed in Fig. 5 was responsible. From the results 2 and 6 wt.% treated PKSF/PCP reinforced composites possess the least porosity content with values of 0.15 and 0.62%, respectively.

3.5. SEM images of the composites

Figs. 9 and 10 showed the images of the fractured surfaces of the developed composites from both treated and untreated PKSF/PCP reinforced composites. From the plates it was observed that both the fibers and the particulates were well dispersed and bonded within the epoxy matrix. However, the treated PKSF/PCP reinforced sample revealed a more blend of the material compare to the untreated ones. There are more particles, the whitish part, at the fractured surface and de-bonding at the interface between the untreated fiber and the epoxy matrix for the untreated PKSF/PCP reinforced composites. These are the evidence of poor and weak interfacial reaction that usually cause weak bonding strength at the interface and, hence, poor load transfer at the interface. Untreated agro-based materials are usually with smooth polymeric material called lignin that bonds the cellulose and hemicellulose together to form natural composites. This smooth surface in most cases, will not allow proper bonding between the matrix and the agro-based materials when developing composite. Therefore, surface modification is usually essential in which chemical treatments or the use of compatibilizer is commonly adopted to improve the strength at the interfacial level. The sound blend obtained in Fig. 9 revealed the reason for the good mechanical and porosity properties obtained in Figs. 4–6 and 8 compared to the untreated PKSF/PCP reinforced composites.
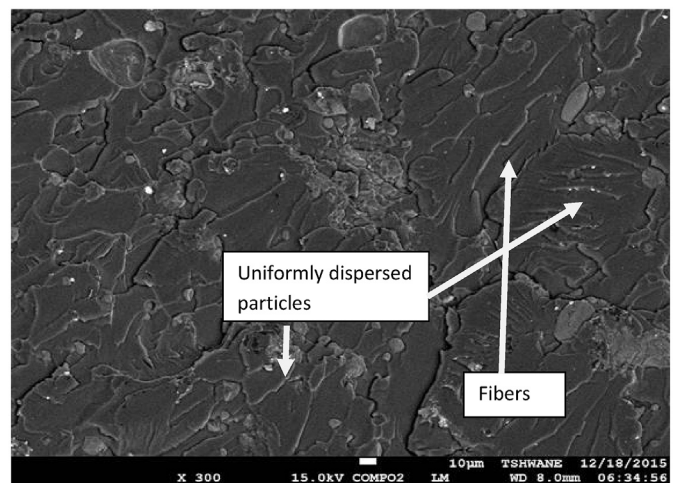

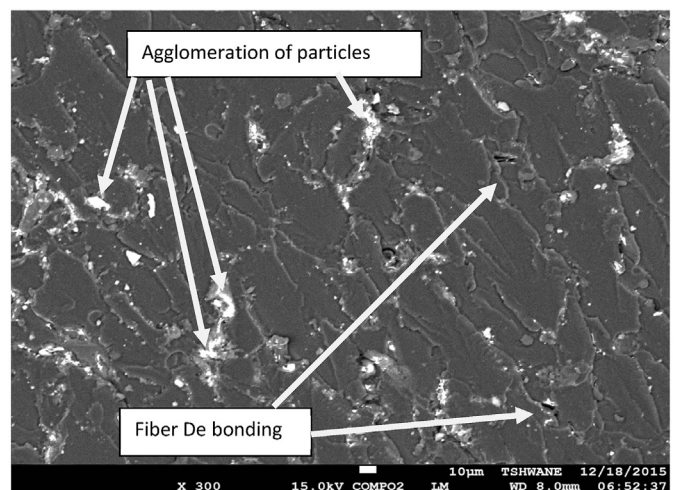**Fig. 9.** Fracture surface of treated PKSF/PCP reinforced sample.

Fig. 10. Fracture surface of untreated PKSF/PCP reinforced sample.

4. Conclusion

i. The results of the study revealed that chemical treatment for the modification of the agro-based reinforcement's surfaces for effective interfacial adhesion was a very good means of enhancing the mechanical properties with less porosity content. Their inclusion into the epoxy matrix brought about some enhancement in the stiffness of the developed composites which made them have better resistance to deformation under such loads (Young's Modulus, Flexural Strength at Peak and Wear).

ii. The use of these agro-based materials in both treated and untreated conditions is imperative since they yielded high and improved mechanical and wear properties. And, as they are environmentally friendly organic materials that are biodegradable, they remain best alternative materials for low technology applications.

iii. Though much has been done with palm kernel shell fiber but not much has been done with particulate cassava peel for the development of composite materials. The analysis of its elemental composition, therefore, encourages the use of the material as another good source of agro-waste that has the potential for engineering applications.

iv. Reinforcement content with optimum value was 6 wt.% from chemically treated PKSF/PCP hybrid reinforced composites.

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