



## MECHANICAL PROPERTIES AND WEAR BEHAVIOUR OF KAOLINITE CLAY PARTICLES REINFORCED EPOXY MATRIX COMPOSITES

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### ABSTRACT

*Epoxy matrix composites reinforced with fine clay particles were developed by hand lay-up open mould casting technique. The clay used in this study was pulverized and processed into fine particles through sedimentation process. The composites were developed by mixing the epoxy matrix and hardener with various weight fractions of fine clay particles (2, 4, 6, 8 and 10 wt%) in an open test moulds. In order to accomplish a uniform blend of the constituents; manual mixing of the blend was carried out for 3 min. The test specimens were left to cure for 24 hours in the moulds and for additional 30 days at room temperature of  $25 \pm 2$  °C and were thereafter detached from the moulds. The developed composites test specimens were subjected to mechanical tests (flexural, tensile and impact) in accordance with ASTM standards and performed at room temperature. Structural characteristics of the clay particles were determined using X-ray diffractometer (XRD). The morphologies of the composites were determined utilizing scanning electron microscope (SEM). There was progressive enhancement in the mechanical properties of epoxy composites containing 2-6 wt.% fine clay particles while drastic decrease in the mechanical properties were noticed in the epoxy/clay composites reinforced with 8-10 wt.% fine clay particles. The SEM images revealed homogeneous particles distributions within the epoxy matrix at lower fine clay particles weight fractions (2 wt. % and 6 wt.%).*

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## 1. INTRODUCTION

Polymer matrix composites are turning into a fundamental piece of materials used in recent time for various engineering applications because of their low weight, high fatigue strength and easy production technique. Composites are utilized as materials for making airplane structures, golf clubs, electronic parts, clinical hardware, space vehicles and building (Pandey *et al.*, 2005;

Autar, 2006). Composite materials are inherent materials created from two or additional basic constituents with substantial diverse physical or chemical properties which remain detached and discrete at macroscopic and microscopic level within the refined formation. Composite materials consist of robust load carrying material acknowledged as reinforcement entrenched in fragile material well-known as matrix.

Reinforcements impart strength and stiffness, assisting in supporting structural load. These can be particulate, whiskers or fibers and are generally added to the matrices to enhance material properties such as modulus, tensile strength and fracture toughness of the matrices (Sushree, 2012; Daramola *et al.*, 2017a). Polymer composites can be grouped as macro composites, micro-composites and nano-composites according to fillers size, (Ajayan *et al.*, 2003). Capacity of a polymeric material to protect a surface is exceptionally subject to its capacity to adhere to a surface and limit admittance to corrosion sites. A solid relationship occurs amid corrosion protection and adhesive strength (Daramola *et al.*, 2017b).

Polymer Matrix Composites (PMCs) are lightweight; they have high specific strength properties and are usually low cost alternative to the conventional metals or alloys. Polymers usually have advantages over other materials such as metals and ceramics. They are extensively utilized in numerous technical applications because of their unique merits such as ease of production, light weight, ductility and corrosion resistance. Nevertheless, they have poorer stiffness and strength properties likened to that of metals and ceramics. The industrial significance of polymers and their growing usage results to the incessant request for enhancement in their properties to meet the essential conditions. Polymer properties are enhanced while upholding their light weight and ductile nature. The incessant reinforcing fibers of innovative composites are accountable for their high strength and modulus (Nadiir *et al.*, 2014; Jordan *et al.*, 2005).

Clay is a weathering creation made by

fragmentation and chemical disintegration of igneous rocks with fine texture of particle size less than 0.002 mm (2 micron). Finest part of clay is comparable to colloid, with fine grain size, stickness, and plasticity features when wet. Clay and clay minerals are very essential engineering minerals; they have been employed as raw materials for hundreds of industrial applications owing to its copious availability and affordable cost. Clay minerals can be classified into four subcategories: Kaolinite, Smetite, Mica and Chlorite (Burhan and Cifti, 2010). Kaolinite is the most important member of the subgroup, which occurred in residual weathered material and a common constituent of soil. Properties such as plasticity, bond strength, shrinkage, retention of forms, opacity and viscosity makes kaolinite suitable for use in paper coating, pottery, porcelain and sanitary ware production (Folorunso *et al.*, 2014). Ikere clay has been used for production of furnaces lining, electrical porcelain and ceramic products. The clay particles can be blended with a polymer matrix to produce a biological/mineral composite (Hwu *et al.*, 2002). Clays are used in agricultural, engineering and construction applications, environmental remediation, geology, pharmaceuticals, food processing, electrical applications and many other industrial applications (Murray, 2007).

Matrix materials in case of polymer matrix composites can be categorized based on their chemical structure which comprise of the thermoplastic and thermosetting polymers. Amid the thermoset materials, epoxy resins displays unique chemical features such as nonappearance of by-products or volatiles during curing reactions, low shrinkage up on

curing, curing over an extensive range of temperature and the control of degree of cross-linking. Depending on the chemical structure of the curing agents and curing conditions, the properties of cured epoxy resins will vary. Epoxy resins are flexible with outstanding chemical and heat resistance, elevated adhesive strength, worthy impact resistance, great strength and hardness, and elevated electrical insulation (Sandler *et al.*, 2003, Liu *et al.*, 2002). Currently, epoxy resins are extensively utilized in numerous engineering and structural applications such as aircraft, aerospace engineering, sporting goods, automotive and military aircraft industries. Despite the numerous advantages portrayed by epoxy resins, they are delicate and brittle. To enhance epoxy resins manufacturing and product performances and to lessen cost, several fillers are blended with the resins during production process (Parkhe and Sanjay, 2014).

Centrifugal processing method is generally utilized for separating solid liquid or liquid-liquid in numerous industries owing to the great gravitational force effect on the particles. In this method, if the applied centrifugal force generated by the angular velocity of a rotating basket is greater than capillary force, liquid in the capillary tubes is impulsively detached from the filter cake.

The purpose of this research is to produce fine ceramic particles from natural clay to reinforce epoxy resin to enhance its mechanical properties and wear behaviour for engineering applications.

## 2. Materials and Methods

### 2.1 Materials

Clay particles were utilized as reinforcement in this study; the clay was obtained from Ikere - Ekiti clay deposit, Ekiti State, South western Nigeria. Epoxy resin and hardener were used as matrix which was procured from Pascal scientific laboratory, Akure, Ondo State, Nigeria. Silicon oil which was used as lubricant was procured from Hysil chemicals venture limited Lagos Nigeria.

### 2.2 Methods

#### 2.2.1 Preparation of Clay

The clay samples as obtained were milled with the aid of laboratory ball-mill and immersed in water for 3 days. The saturated clay specimen were blended dynamically in a helical direction with the support of ceramic blunger. The clay slurry were detached from the blunger and placed in a translucent vessel and allowed to settle. Three diverse clay strata were detected, the layers are colloids, fine and coarse particle sizes, two layers were successfully separated, (fine and coarse particle sizes). The clay samples in their diverse classes were dried in the sun for 4 weeks and were thereafter pulverized using laboratory mortar and pestle and the pulverized particles were used for the composites development.

#### 2.2.2 Particles Size Analysis

The clay particles size analysis was carried out with the aid of particle size analyzer. About 1 g of the fine clay particles was sprinkled in de-ionized water in the sample distribution unit of the instrument, dynamically blended for about three minutes at speed of 2100 rpm, and sonicated for 55 seconds. The ultrasonic waves were utilized to halt or curtail any particle clustering that may be existing in the

suspension. The measurements of the intensities and particles diameters were taken and recorded for plotting the graph using origin software.

### 2.2.3 Clay Particles X-Ray Diffraction (XRD) Analysis

X-ray diffraction (XRD) measurements were carried out on the clay powder. Structural information of the samples was examined utilizing a Rigaku Multiflex powder X-ray diffractometer with the wavelength set at 0.154 nm utilizing a Cu  $\alpha$  source, a voltage/applied current of 40 kV and 30 mA respectively. The samples were scanned from 10° to 80° span using a step of 0.5°  $\theta$ /minute.

### 2.3.4 Epoxy/Clay Composites Development

The materials used for this research were weighed into numerous weight fractions with the aid of meter electronic weighing balance. The clay samples were weighed based on a predetermined proportion of 2, 4, 6, 8 and 10 % weight fraction. The volume of the epoxy resin and hardener used for each sample varies with change in the percentage clay. The composite samples were produced using open mould casting method by dispersing the clay particle into the epoxy matrix. The mixture

were stirred properly to ensure homogeneity and the mixture were poured into the metallic mould. Tensile mould of gauge length 90mm x 5mm x 3mm of a dumb-bell shape and flexural mould of 150mm x 50mm x 3 mm was utilized for the fabrication of tensile and flexural test samples respectively. Wear test and impact test moulds were used to produce their test samples. The mixture were poured into the metallic mould and allowed to cure for 24 hours. The process were repeated for the five different weights percent, the developed composites were separated from the mould and allowed to cure further at room temperature for 27 days. The formulation table used in the development of the composites is as shown in Table 1.

## 2.4 Testing and Structural Characterization of Composite Samples

### 2.4.1 Ultimate Tensile Test

The ultimate tensile test of the composites was evaluated in agreement with ASTM D638-10 procedures (American Society of Testing and Materials). The test was performed utilizing an Instron universal testing machine, USA model; Instron-series 3369 operated at a crosshead speed of 0.3mm/mm and a strain

Table 1: Formulation Table for the Epoxy/Clay Composites Development

Fine Clay Particles Weight Fraction (%)	Fine Clay Particles Weight Fraction (g)	Weight of Hardener (g)	Weight of Epoxy (g)
0	0	150	300
2	9	147	294
4	18	144	288
6	27	141	282
8	36	138	276
10	45	135	270

rate of  $10^{-3}$ /S at room temperature of 27 °C and relative humidity of 40 %. Three repeated test were carried out for each composition of the composites developed.

#### 2.4.2 Flexural Test

Flexural strength was evaluated with the aid of a universal testing machine (Torse's) utilizing three-point bending configuration according to ASTM D790 at a crosshead speed of 10 mm/min. The width and thickness of the specimens were measured. The flexural base jig, support span, was adjusted to allow for the specimens to fit in as per ASTM D790, with each end point being 63 mm from the center of the specimen. The machine has a built in encoder to capture the required data. The specimens were positioned in place with the loading nose placed at the central point of the specimen. The machine was reset and the program ran at a cross head speed of 2 mm/min until the specimen failed. Once the specimen failed the machine was returned to its starting position and reset with the same procedure followed for the remaining specimens. The equation used to calculate the flexural stress for the composite materials was obtained from ASTM D790.

#### 2.4.3 Impact Test

Epoxy matrix and epoxy/clay composites were tested on a Charpy V- Notch impact testing machine (Instron CEAST 9050) in agreement with ISO 179. Three repeated test were carried out for each of the composites samples to generate a reliable data and the average data were calculated for each sample.

#### 2.4.4 Abrasive Wear Test

The wear tests were evaluated in accordance with ASTM D1044-13 standard using Taber abrasive tester model TSC-AO16. This method determines the volume of material eliminated by abrasion, and is generally stated in milligrams. The weight of the developed composite samples for each composition were determined and reported to be the initial weight using a four decimal place analytical weighing balance. The test was carried out at room temperature, and operating parameter of 500 g load at 50 rpm. The samples were cleaned and weighed to determine the wear rate values. The formula for calculating the weight loss is shown in equation 1

$$L = A - B \quad (1)$$

where  $L$  is weight loss;  $A$  is weight (mass) of specimen before abrasion and  $B$  is weight (mass) of specimen after abrasion

#### 2.4.5 Scanning Electron Microscopic Examination

The fractured surface of the samples were assessed using AURIGA Scanning Electron Microscope (SEM) (Carl Zeiss, Germany) with an accelerating voltage of 15 kV. The samples were first fracture and the fractured surfaces were fixed on aluminum stubs and were sputter coated with gold using EMITECH K950X sputter coater before being exposed to scanning electron microscope for surface morphological analysis.

### 3. Results and Discussion

#### 3.1 Clay particle sedimentation process

The clay particles sedimentation process is

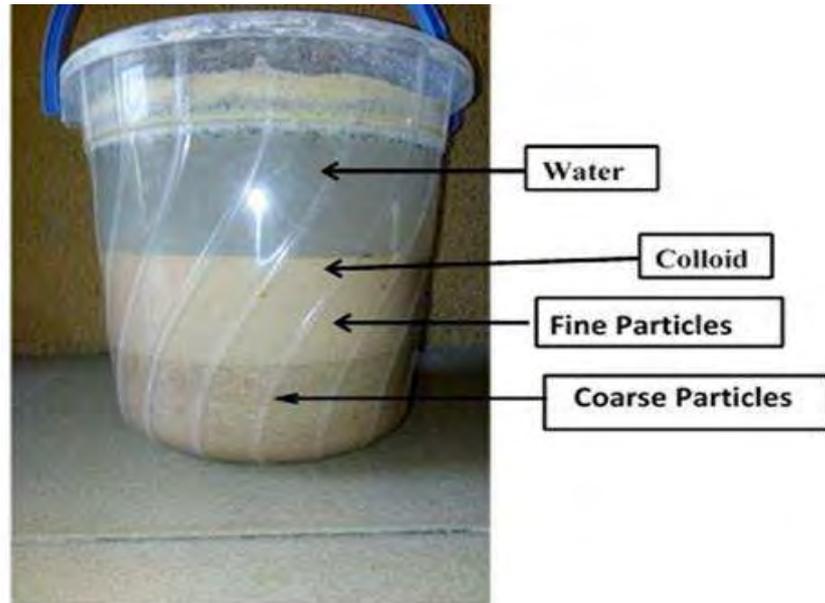


Figure 1: Clay Particles Sedimentation Process

presented in Figure 1, the process revealed three major layers of clay particles which are: colloid, fine and coarse particle sizes. The fine particles was used for the development of the Epoxy matrix composite in this research.

### 3.2 Clay Particles Size Analysis

Figure 2 showed the particle size analysis result of the fine clay particles. The average diameter of the particles was found to be  $1.9807\mu\text{m}$  and was utilized for the development the epoxy-clay composites in the present work.

### 3.3 X-Ray Diffraction (XRD) of Clay Particles

The XRD analysis of the clay particles is presented in Figure 3. Structural information of the samples was evaluated with the aid of a Rigaku Multiflex powder X-ray diffractometer with the wavelength set at  $0.154\text{ nm}$  utilizing a  $\text{Cu } \alpha$  source, a voltage of  $40\text{ kV}$  and applied current of  $30\text{ mA}$ . The samples were scanned from  $10^\circ$  to  $80^\circ$  span

using a step of  $0.5^\circ \theta/ \text{minute}$ . The XRD analysis revealed the sample as Kaolinite Clay (Aluminium Silicate Hydroxide) with chemical formula  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$  which is an inorganic mineral

### 3.4 Ultimate Tensile Strength

Figure 4 displays the disparity in the ultimate tensile strength of Epoxy matrix and Epoxy/Clay composites. It was discovered from the results that the ultimate tensile strength of composites increased progressively from  $2\text{ wt.}\%$  to  $6\text{ wt.}\%$  and start decreasing as from  $8\text{ wt.}\%$ . The optimum results were obtained at  $6\text{ wt}\%$  fine clay particles loading with a value of  $63.47\text{ MPa}$  while composite with  $8\text{ wt}\%$  particles loading has a value of  $55.41\text{ MPa}$ . The reason for this pattern of results is that, there was homogeneous particles distribution at lesser weight fraction ( $2\text{-}6\text{ wt.}\%$ ) of clay particles loading as displayed in the SEM image in Figure 8(a and b), which led to useful enhancement of ultimate tensile strength of

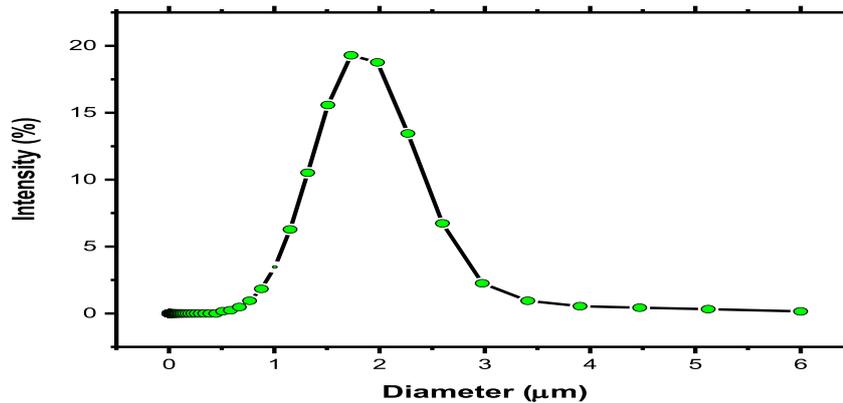


Figure 2: Mean particle size plot of the kaolinitic clay

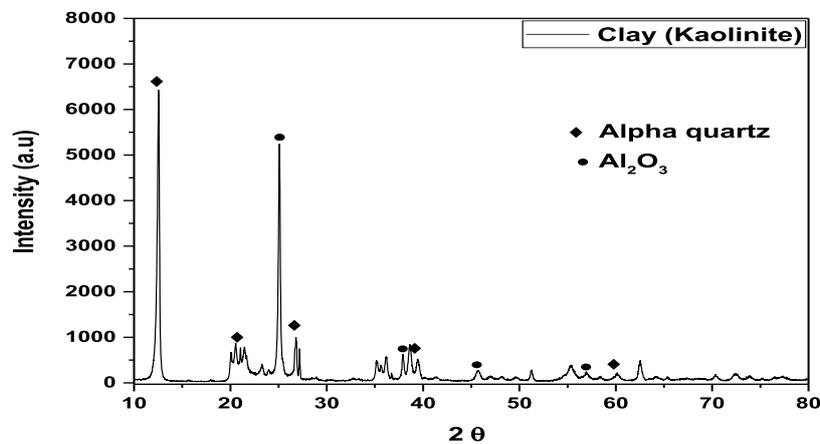


Figure 3: X-Ray Diffraction of Clay powder

the composites as a result of chain inter dispersion and entanglement between epoxy and clay particles. Moreover, the increase in tensile strength at lower weight fractions (2-6 wt.%) may be ascribed to the development of exfoliated structure for epoxy composites with 2- 6wt% of clay particles. In the exfoliated structure, individual silicate layers in this size are distributed consistently in the polymer matrix with great aspect ratio. The great aspect ratio of clay particles may also enhance the tensile strength by improving the filler contact surface area on the polymer matrix, As the particles weight fraction increases (from 8-10 wt.%) the clay particles begin to cluster and the size of the epoxy

chains is elevated appropriately and the inter-layer and the interfacial stress transfer efficacy diminished, the fragile interfacial area between the particles and the matrix was enhanced, which led to interfacial detachment at the particles-matrix interface thereby causing reduction in the ultimate tensile strength of the composites (Yang, 2004, Jamil *et al.*, 2006, Lou *et al.*; 2007).

### 3.5 Flexural strength at Peak

Flexural strength at peak is the ability of a material to withstand bending force perpendicular to its longitudinal axis. The effect of fine clay particles inclusion on the

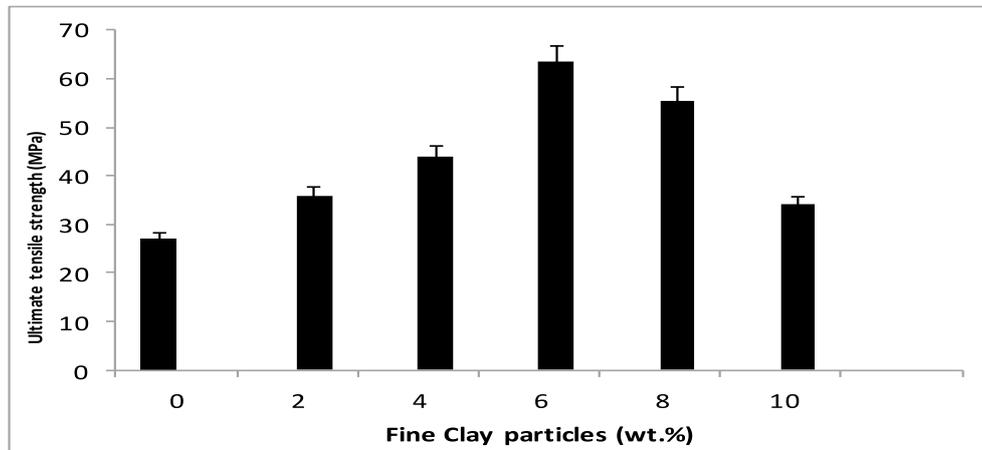


Figure 4: Variation of the Ultimate Tensile Strength of Epoxy/Fine Clay Composites

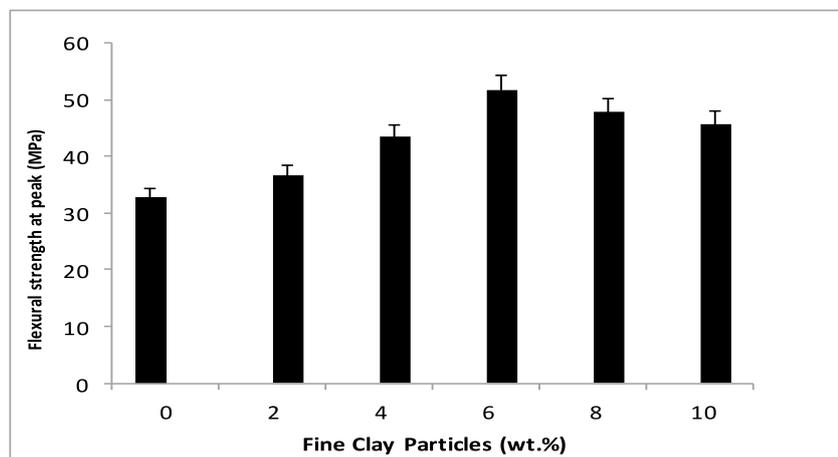


Figure 5: Variation of Flexural Strength at peak of Epoxy/Fine Clay Composites

flexural strength of epoxy composites is displayed in figure 5. A related behaviour to that of the tensile strength was noticed. The inclusion of clay up to 6 wt% enhanced the flexural strength at peak with a value of 51.69 MPa. Above 6wt% of clay particles loading, the flexural strength begins to reduce. The exfoliated structure formed and the smaller particle size of the reinforcement in the composites containing 2 – 6 wt% of fine clay particles is considered to be liable for the experimental inclination. The interfacial

bonding between the clay particles and the epoxy matrix is usually moderate at lower particles weight fraction, thus enlarging the surface area of matrix/clay interface. This leads to useful stress transfer from the matrix to the clay, therefore, causing enhanced flexural strength. The reduction in flexural strength above 6wt% of fine clay particles was possibly due to the occurrence of clustered fine clay particles, which probably behaved as stress concentration spots and triggered a reduction in the flexural strength of the

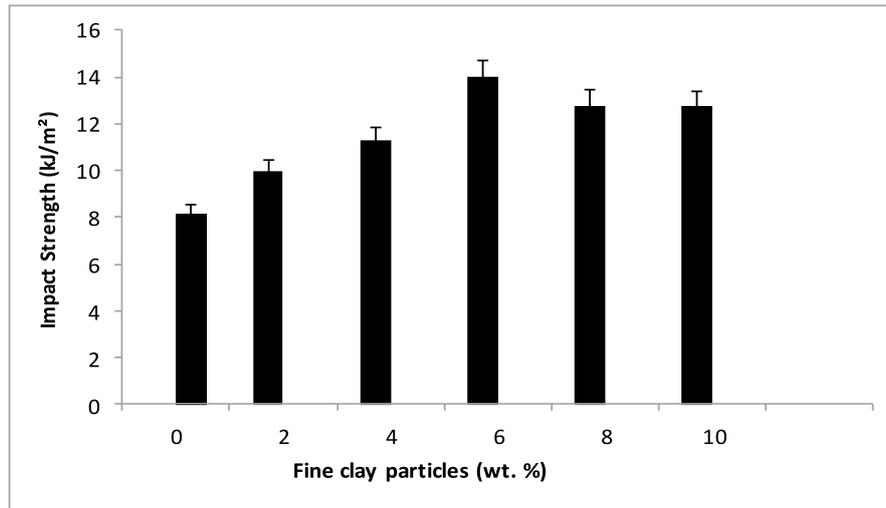


Figure 6: Variation of Impact Strength of Epoxy/Fine Clay Composites

composites. Chow *et al.* (2004) examined the influence of organoclay on the flexural property of injection-moulded polyamide 6/polypropylene nanocomposites; the outcomes of the research revealed that the flexural strength was enhanced as the weight fractions of the organoclay particles increases progressively up to 4wt%. This increase in flexural strength might be as a result of the uniform distribution of organoclay particles within the epoxy matrix.

### 3.6 Impact Strength

Figure 6 revealed the impact strength of epoxy composites reinforced with clay particles which increased with the clay particles up to 6 wt%. Above 6 wt% clay particles weight fraction, the impact strength begin to decrease with increase in the fine clay particles loading in the composites. The optimum impact strength was achieved at 6 wt% of fine clay particles weight fraction with a value of 13.98 kJ/m<sup>2</sup>. This showed that the clay has additional useful strengthening effect particularly for 6 wt% of clay particles. The enhancement in the

impact strength may be associated to the molted silicate strata of clay in the epoxy matrix as revealed in the SEM images in Figure 8 (a and b). The trend observed in the present research is similar to the work of Rozman *et al.*, (1998) on the effect of rubber and wood particles on the impact property of high density polyethylene matrix composites. Liu *et al.*, (2004) and Sanadi *et al.*, (1997) also reported in their work that there is a weakening influence of the particles on the impact strength of the matrix as a result of sudden reduction in discontinuity elongation because the particles clusters and bonds the fracture and enhances the resistance of fracture procreation at higher particles weight fraction.

### 3.7 Abrasion Wear Result

Figure 7 displays the precise wear behaviour of fine clay particles reinforced epoxy matrix composites. It was discovered that 6 wt.% fine clay particles reinforced epoxy matrix composites showed the best abrasion result with a value of 0.042 mg followed by 4 wt.%

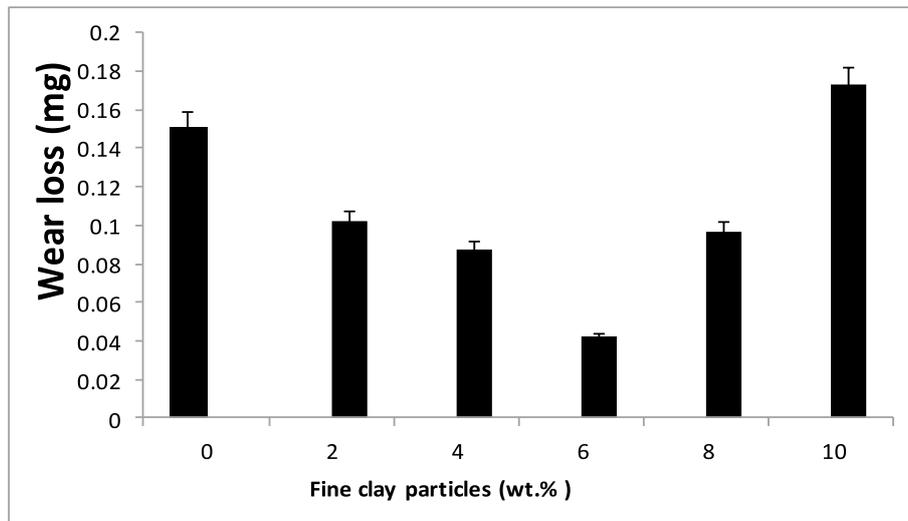


Figure 7: Variation of Wear index of Epoxy/Fine Clay Composites

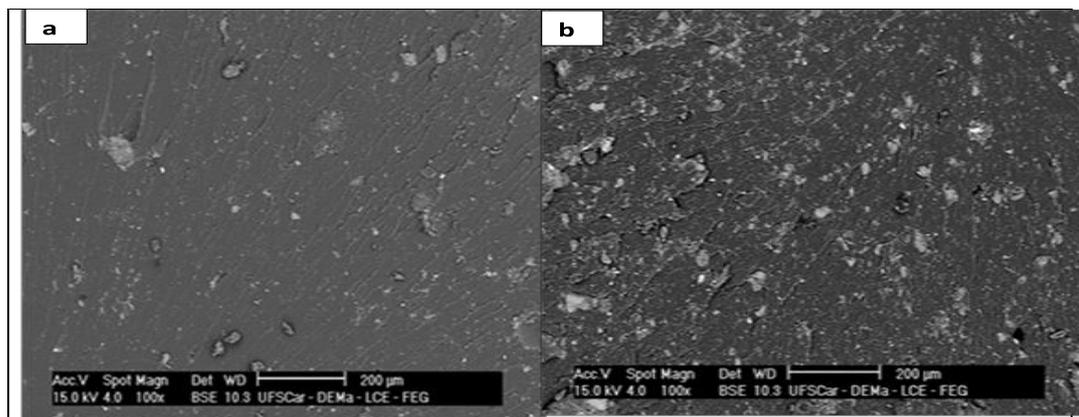


Figure 8: SEM Images of (a) 2 wt% of Epoxy/Fine Clay Composite  
(b) SEM Image of 6 wt% of Epoxy/Fine Clay Composite

and 2 wt.% clay particles reinforced epoxy matrix composites with values of 0.087 mg and 0.1023 mg respectively. Conversely, the additional inclusion of fine clay particles to the epoxy matrix has serious effect on the precise wear behaviour of the composite. It was observed that the exfoliated structure in the epoxy composites with 4 and 6 wt.% fine clay particles decreased the precise wear property of the composites. The optimum wear characteristic of the epoxy matrix

composites was attained at 6 wt.% fine clay particles loading.

### 3.8 Examination of the Surface Morphology of Clay /Epoxy Composite

The surface morphologies of the epoxy matrix composites with the least and optimum mechanical properties were examined. Figure 8(a, b) revealed the SEM images of epoxy matrix composites containing 2 wt.% and 6

wt.% of clay particles respectively taken at a magnification of 100X. For the 2 wt.% clay particles reinforced epoxy composite sample as shown in Figure 8a, the cleavage surface showed fractured pieces with smooth white fracture borders, nevertheless, it is obvious that the inclusion of 6 wt.% fine clay particles in the epoxy matrix composites intensified the coarseness of the fracture surfaces as shown in Figure 8b.

## CONCLUSIONS

The influence of diverse weight fractions (2, 4, 6, 8, 10 wt.%) of kaolinite clay particles on the mechanical and wear behaviour of epoxy matrix composites has been studied. The following conclusions were drawn:

- i. Fine clay particles produced by sedimentation process were successfully utilized for the production of epoxy clay composites.
- ii. It was observed that the addition of clay particles to epoxy resin causes an enhancement in the mechanical properties (ultimate tensile strength, flexural strength and impact strength) up to 6 wt. % fine clay content.
- iii. The XRD analysis of the fine clay particles revealed the sample as Kaolinite Clay with chemical formula  $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$  (Aluminum Silicate Hydroxide)
- iv. The scanning electron microscopic analysis of the fractured surfaces of the developed composites revealed that fine clay particles, dispersed uniformly in the epoxy matrix as

observed from the SEM images (Figure 8) of epoxy/clay composites reinforced with 2 wt.% and 6 wt.% clay particles

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