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## ENHANCEMENT OF ELECTRICAL RESISTIVITY PROPERTY OF NATURAL CLAY/EPOXY COMPOSITE FOR ELECTRICAL APPLICATION

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**Abstract:** Properties of particulate inorganic clay reinforced epoxy resin for electrical applications were investigated in this research. Ikere clay particle of 38  $\mu\text{m}$  size was utilized to develop the clay particulate reinforced epoxy composites having 2, 4, 6, 8, 10, 15, 20 and 25 wt. % of the clay particle as reinforcements. Unreinforced epoxy was produced for control experimentation. The effect of reinforcing epoxy with clay on the mechanical, wear and electrical characterization were studied. Scanning Electron Microscopic analysis was carried out on the fracture surfaces of the developed composite samples. The result shows that Ikere clay particulate reinforced epoxy composite developed with 15 wt. % reinforcement has the best impact strength, maximum resistivity and least conductivity while 10 wt. % reinforced composite possess the best hardness and wear resistance, respectively. The results demonstrate the potential of using Ikere clay particulate as a reliable cost effective and technically efficient reinforcement material for electronic casing application.

**Keywords:** Epoxy, electrical resistivity, inorganic reinforcement, Ikere clay

### 1. INTRODUCTION

The use of polymer and polymer matrix composites has found wide application in our modern day world. This is as a result of the combination of properties which these materials possess. Of the new areas of applications for these materials are Inorganic light emitting diodes (OLEDs) which are an emerging technology for displays and lighting applications which are gaining more and more attention because of their simple structure, low weight and low cost processing potentials [1]. Also, polymer light emitting diodes (PLEDs) are very interesting for the possibility to be fabricated on flexible substrates by solution processes compatible with roll-to-roll printing and coating techniques [2]. Others are sensor applications [3] and Conducting Polymers (CPs) which have been widely used in advanced technologies. They are utilized for technological devices such as biosensors [4], photovoltaic cells [5] and super capacitors [6].

Some of the properties of polymer matrix composites include lightweight, excellent specific strength, high modulus, good fracture and fatigue properties as well as corrosion resistance [7]. The commercial importance of polymers and their increasing use has led to the continuous demand for improvement in their properties to meet the necessary conditions. By the composite technology, polymer properties are improved while maintaining their light weight and ductile nature [8]. A variety of new advanced composite materials are now available that provide great advantages over conventional materials for electronic packaging and thermal control including extremely high thermal conductivities (more than twice that of copper); In addition, composites are in a state of continual development that will provide even greater benefits. Epoxy resins belong to a class of polymer under the aegis of thermoset [9]. Epoxy resins are one of the most common thermosets which are widely used as adhesives in various applications: in the aerospace industry, construction composite materials, surface coatings, materials in machine building including automotive and ship industries, packaging materials for electronic devices, as well as in medicine or home fields [10]. Among the most valuable properties of epoxies are, good adhesion to various substrates, high modulus, high temperature performance, low shrinkage and good corrosion resistance [11].

However, when compared to other light materials like aluminum, they have low mechanical strength and thermal resistance. Hence, filling of epoxy resin for improved mechanical, wear and thermal resistance properties is imperative [12]. Fillers play a key role in polymer industry. Polymer matrix composites with fillers have the potential to replace traditional materials. One of the advantages of using fillers is that it can reduce the overall cost involved in development of composites because of reduced resin requirement. It also helps in improving toughness, mechanical and tribological properties [13]. The formation of light polymer foams with potential applications of such foams in the field of electrically and thermally conductive materials, as well as electromagnetic interference (EMI) shielding materials that can be developed by incorporating conductive nanofillers such as carbon nanotubes (CNTs) and graphene nanoplatelets (GNPs) within the porous matrix prior to foaming was made possible [14-15]. Selection of appropriate filler depends on end use, compatibility with other constituent materials and dimension of the particles. Some inorganic filler includes calcium carbonate, sulphate and metal powders. Higher specific gravity and incompatibility with polymers are the known limitations of inorganic filler materials. Inorganic fillers when used in composite laminates can account for 40 to 65 % by weight and perform a function similar to silica fume in concrete. Kaolin (hydrous aluminum silicate) is the second most

commonly used filler known throughout the industry as clay [16]. Clay are been used due to its unique physical and chemical properties. Hence, this research aimed at determining the effect of using varying weight percentages of Ikere clay particle with a specific micron meter size on the mechanical, wear and electrical properties of epoxy reinforced composites.

## 2. MATERIALS AND METHOD

### — Materials

The materials used for this work are raw clay sample collected from Ikere-Ekiti, Ekiti State in South western part of Nigeria which serves as the reinforcement while kerapoxy and hardener were used as the matrix.

### — Preparation of Ikere-Ekiti clay

Raw Ikere-Ekiti clay gotten from its deposit was soaked in water for 3 days to dissolve the clay and also form slurry. The slurries formed were then sieved to remove dirt and other foreign substances. These were allowed to settle down for 2 days after which the clear liquids were decanted to remove deleterious particles. The settled fine clay was sun dried for 5 days with bigger lumps being formed after drying. The clay was pulverized into smaller size and ground into finer particle size using a ball mill. The fine particle clay was sieved using a 38  $\mu\text{m}$  mesh sieve size which was used to produce the composite samples.

### — Composite development

The composites were produced through open-cast moulding technique using 2:1 of the epoxy and hardener, respectively. A homogenous mixture of the clay particle, the epoxy resin and the hardener was achieved via manual mixing with a glass rod stirrer for 2 minutes in a polymeric container. The sieved clay particle was dispersed into the epoxy matrix in predetermined proportions: 2, 4, 6, 8, 10, 15, 20 and 25 wt. %. Prior to the introduction of the homogeneous mixture into the respective moulds, the moulds were coated with a releasing agent to facilitate the ease of removal of test samples. The samples were allowed to cure in air at room temperature which ranged between  $24 \pm 2$  °C. The developed composite samples were separated from the mould after curing and then, allowed to cure further at room temperature for 27 days. Plate 1 shows the Ikere-Ekiti clay that was used for the developed composite samples. Control sample was produced without the addition of the clay particle.



Plate 1. Ikere-Ekiti Clay particle (38  $\mu\text{m}$ )

### — Mechanical testing

Mechanical properties of the composites produced were assessed through the evaluation of the impact, hardness and tensile properties.

#### ≡ Impact test

The strength of the composites developed were evaluated on a Hounsfield balanced impact testing machine. The notched composite samples were placed in cantilever position and pendulum swings around to break the sample, three repeated tests were carried out for each of the composition of the composites samples to generate a reliable data for the computation of the average impact strength values.

#### ≡ Hardness test

Hardness of the developed composites were been evaluated using Rockwell hardness tester "C" scale. Flat surfaces of the sample were subjected to a direct load of 60Kgf for 10 seconds for the determination of the hardness value. Several hardness indents were made on each sample while readings within the margin of  $\pm 2\%$  were taken for the computation of the average hardness values.

#### ≡ Tensile test

Tensile properties of the developed composite samples were evaluated using an Instron Universal testing machine at room temperature operated at a strain rate of  $10^{-3}/\text{s}$ . For reliability of values generated, three repeated tests were performed for each composition of the developed composites. The samples preparation, testing procedure and basis for determination of the tensile properties were in accordance with the specification of ASTM D3038M-08 [17] standard.

### — EDXRF and SEM examination

EDXRF was used to determine the elemental composition of Ikere-Ekiti clay while the fractured surfaces of the developed composite were examined using Phenom ProX scanning electron microscope (SEM) with energy dispersive spectroscopy (EDS) operated at 15 kV. The developed composite samples were coated with gold to make them conductive prior to SEM observation using Quorum coating machine (Q150RES).

### — Wear behavior

The wear behaviors of the developed composites were evaluated in accordance with ASTM D1044-13 standard using Taber abrasion machine. The samples were placed on a turntable platform of the wear machine and gripped at a constant

load of 500g lowered onto the sample surface. In operation, the turntable rotates at 150rpm with the sample which drives the abrasive wheels in contact with its surface. The initial weight and final weight of the different samples were measured using digital weighing balance, the wear index were calculated using the relation in equation 1.

$$\text{Wear Index} = \frac{(\text{Initial weight} - \text{Final weight})}{\text{Time of test cycle}} \times 1000 \quad (1)$$

#### — Electrical characterization

Electrical properties of some selected composite samples were done using two point probe system coupled with Keithley source meter. The two point probe system setup was controlled with a computer interface using Labview with a DC voltage from 0.0 to 0.5 V and Keithley 2400 series source meter and multimeters. The Keithley 2400 series source meter is used for I-V characteristic and sheet resistance measurement. The operation was carried out using sample of dimension 2 x 2 cm connected to the source meter in which voltage was introduced to determine the output current passing through the sample(s). The voltage was measured in volts while the current was measured in Amperes. The resistance (R) was determined using the principle of ohms law as shown in equation 2.

$$V = IR \quad (2)$$

Where; V- voltage in Volts; I- current in Amperes, and R- Resistance in Ohms.

Further calculations were made to determine the resistivity ( $\rho$ ) and conductivity ( $\sigma$ ) using equation 3 and equation 4, respectively. The resistivity and conductivity were displayed in a bar chart.

$$\rho = \frac{RA}{L} \quad (3)$$

where  $\rho$  - Resistivity in  $\Omega \cdot m$ ; R - Resistance in  $\Omega$ ; A - Cross-sectional area in  $m^2$ , and L- Length in m.

$$\sigma = \rho^{-1} \quad (4)$$

where  $\sigma$  - Conductivity in S/m.

### 3. RESULTS

Table 1 and Figure 1 show the representative elemental composition and the EDX pattern of Ikere-Ekiti clay used for the developed composites, respectively. The analysis shows the percentages of the principle minerals and the element present. The major minerals that are present in the clay are silicon and aluminum which makes the clay to be classified as kaolin clay [18]. The EDX pattern in Figure 2 shows the peaks of elements present in the clay.

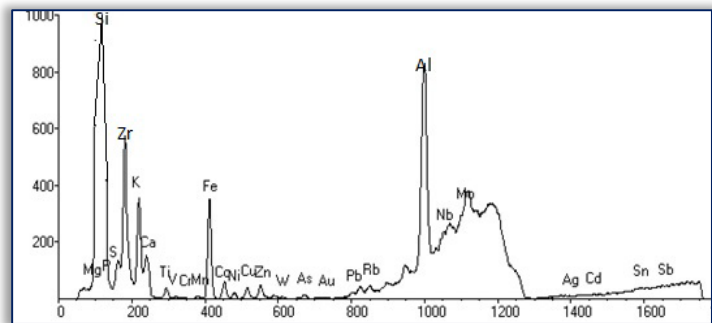
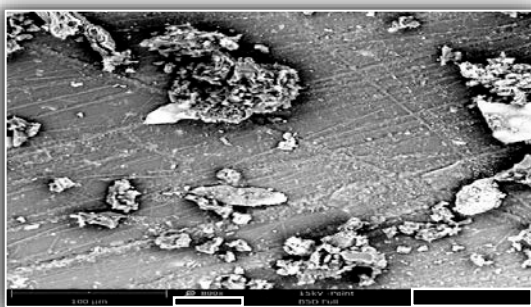


Figure 1. EDX pattern of Ikere-Ekiti clay

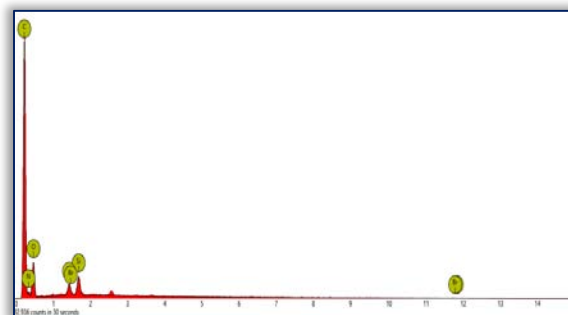
Table 1. Elemental composition of Ikere-Ekiti clay

|         |         |         |        |        |        |        |         |        |
|---------|---------|---------|--------|--------|--------|--------|---------|--------|
| Element | Al      | Si      | P      | S      | K      | Ca     | Ti      | V      |
| Wt. %   | 30.4995 | 45.4987 | 0.2015 | 0.7305 | 0.9010 | 0.1094 | 0.1198  | 0.0078 |
| Element | Cr      | Mn      | Co     | Fe     | Ni     | Cu     | Zn      | W      |
| Wt.%    | 0.0021  | 0.0284  | 0.0136 | 1.6722 | 0.0636 | 0.0540 | 0.0896  | 0.0032 |
| Element | Rb      | Nb      | Mo     | Cd     | Sn     | Sb     | Zr      |        |
| Wt%     | 0.0041  | 0.0166  | 0.1623 | 0.0001 | 1.5451 | 1.4588 | 15.2103 |        |

To investigate the homogeneity of the samples with different weight content of Ikere-Ekiti clay particles, the fractographic observation through the use of SEM/EDS was conducted. The samples with 10 and 15 wt. % of Ikere-Ekiti clay are shown in Figures 2 and 3, respectively. The sizes of the clay particle were similar with each other and likewise the elements that were present as shown by the EDS. The clay particles were found uniformly dispersed in the developed composites.



(a)



(b)

Figure 2. SEM characterization of the 10 wt. % Ikere Ekiti particle clay reinforced epoxy composite showing (a) secondary electron imaging of the fractured surface, and (b) EDS profile of the surface morphology

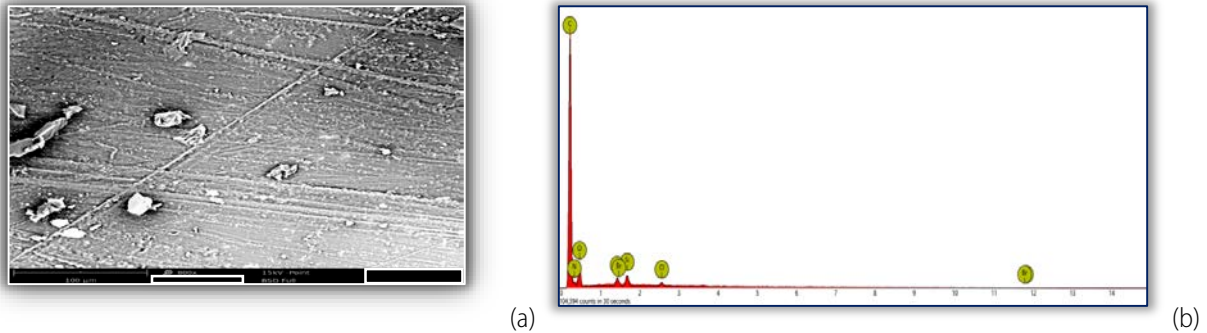


Figure 3. SEM characterization of the 15 wt. % Ikere Ekiti particle clay reinforced epoxy composite showing (a) secondary electron imaging of the fractured surface, and (b) EDS profile of the surface morphology.

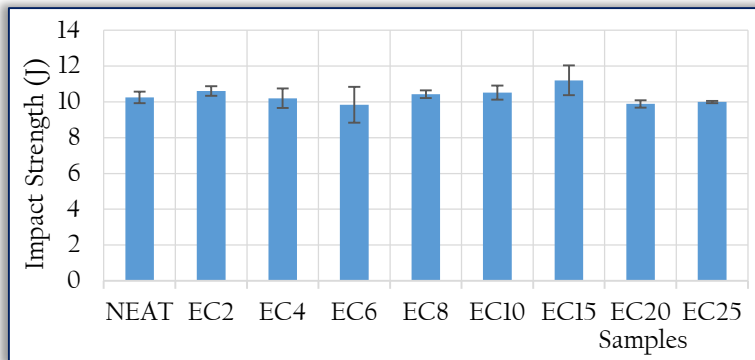


Figure 4. Impact strength of the developed composites and the neat sample

Figure 4 shows the impact strength of the different compositions of the developed composites. From the result, it was observed that most of the developed composites possessed better impact strength than the neat sample. Although, it has been established by researchers that the introduction of rigid fillers into polymer matrices practically leads to embrittlement of the composites once the critical filler loading is exceeded [19], sample EC15 which is made up of 15 wt. % clay particles possessed the best impact strength with a

value of 11.20 J which suggests that the plateau state of filler loading is likely to be at 15 wt. % for the composites. For the materials to be toughened, the interparticle distance must be suitable to allow the interparticle matrix ligament lie in the plane stress state as this allows for easy plastic yield. This plastic yield increases with decreasing particle size and increasing filler content until the critical content is reached [19]. This improvement can be attributed to the combination of a very high strength of interfacial adhesion between the epoxy matrixes and the clay particles and, the absence of localized stress concentration in the resultant composite, hence a significant enhancement in the impact strength of the material. The neat sample was with a value of 10.25 J. However, since significance decrease in the impact strength of the developed composite was observed after 15 wt.%, it may be attributed to low elasticity of the material as the clay particle increases and also coagulation of the clay particle in the matrix after the maximum energy peak has been attained. SEM characterization of the 15 wt. % Ikere Ekiti particle clay reinforced epoxy composite showed in Figure 3 (a) from the secondary electron imaging of the fractured surface revealed that the particles were well dispersed and wetted due to proper interfacial adhesion. This actually confirmed the reason for the observed improved property.

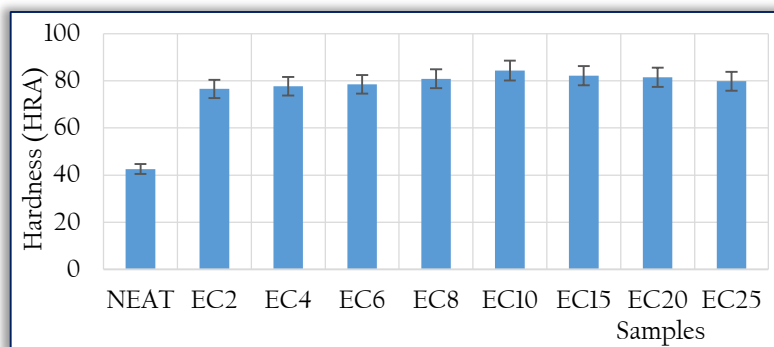


Figure 5. Rockwell hardness results of the developed composites and the neat sample

Hardness properties of the developed composites were shown in Figure 5 where it was seen that all the developed composites possessed better enhancement than the neat sample. The hardness increases from 2-10 followed by a decrease as clay content increases in weight percent. This result was in agreement with the work of Oladele and Adewole [20] in which the hardness increases as the filler content increases from 2-8 % for cowbone ash filled polyester matrix. Therefore, optimum hardness value was obtained at EC10 which is made up of 10 wt. % addition of the clay particle with a value of 84.35 HRA. All the developed composite samples have better hardness properties than that of the neat sample which has a value of 42.55 HRA. The increase in hardness value of the developed composites from 2-10 wt% can be linked to good surface adhesion of the clay particle and the epoxy resin as shown in Figure 2 (a) while the gradual decrease after 10 wt% clay particle addition as the clay particle content increases was attributed to poor interfacial

bonding between the clay particle and the matrix at higher clay particle content. Usually, the presence of a more flexible matrix causes the resultant composites to exhibit lower hardness [21-22]. The presence of inorganic fillers in the matrix brings about filler loading which resulted in abilities of these materials to effectively preclude rapid crack propagation via crack pinning mechanism. Additional energy is required by the cracks to break away from the pinned positions, hence improved in hardness of the materials [19]. Hardness is a measure of the resistance of material to surface indentation which means that the sample with the highest hardness value has the best resistance to surface indentation.

The tensile properties of the developed composites as shown in the stress-strain curve in Figure 6 indicate that the unreinforced epoxy resin which serves as the neat sample has the best ultimate tensile strength as also revealed in Figure 7 compared to the reinforced composites. It was realized from Figure 6 that the neat sample had high strength with low ductility and toughness. Samples EC2 and EC8 which were made up of 2 and 8 wt. % clay particles, respectively possessed good combination of strength, ductility and toughness while samples EC4 and EC6 made up of 4 and 6 wt. % clay particle, respectively exhibit low strength and toughness with high ductility compared to other developed composites. This indicates that, developed samples EC2, EC4, EC6 and EC8 which is made up of 2, 4, 6 and 8 wt. % of clay particles, respectively can serve as materials with good strain behavior due to good matrix/reinforcement interface bonding [23] with samples EC4 and EC6 possessing better ductility.

From Figure 7, it was observed that sample with the highest UTS from the developed composites was EC25 which was made up of 25 wt. % clay particle. The effective stress transfer between the reinforcement and matrix determine the strength of the composite while its toughness/brittleness is controlled by adhesion. The various trends on the effect of particle loading on composite strength and toughness have been observed due to the relationship between the three factors which cannot be separated [24]. The good tensile strength at higher weight percent for the clay particle reinforcement could be attributed to better dispersion of the reinforcement in the epoxy resin, better wettability, absence of void and good interface bonding [23, 25]. However, this strengthening mechanism becomes ineffective because the strength of adhesion between the interfaces of the epoxy matrix and the clay particle has been reduced. Owing to this fact, the stress transfer within the composite becomes ineffective as the applied stress at this stage is principally bore by the epoxy matrix (the poorly bonded particles at this stage begin to debond from the epoxy matrix upon the application

of stress) resulting in a composite with decreased tensile strength. This result was in agreement with the work of Oladele et al., [19] in which it was observed that as the surface area of the particle is decreased as observed with the EMSBs filled with 75  $\mu\text{m}$  MSA, strengthening mechanism becomes ineffective.

The wear test results for the developed composites are presented in Figure 8. It was observed that the wear index (a measure of wear rate) increases as the clay

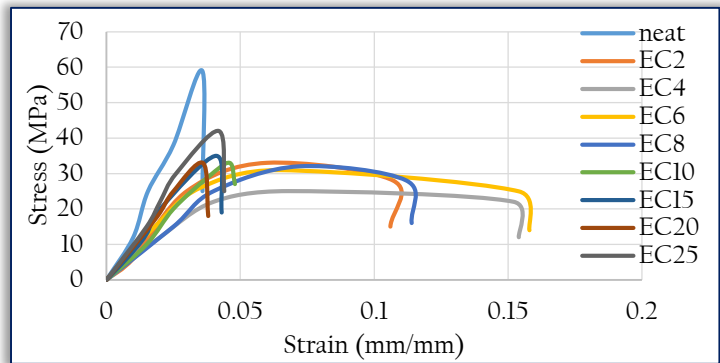


Figure 6. Stress-Strain plots of the developed composites and the neat sample

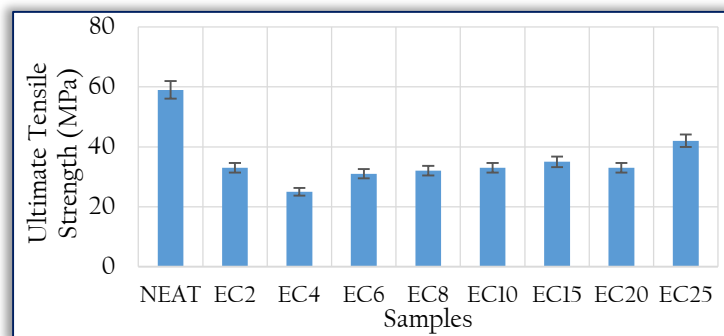


Figure 7. Ultimate tensile strength of the developed composites and the neat sample

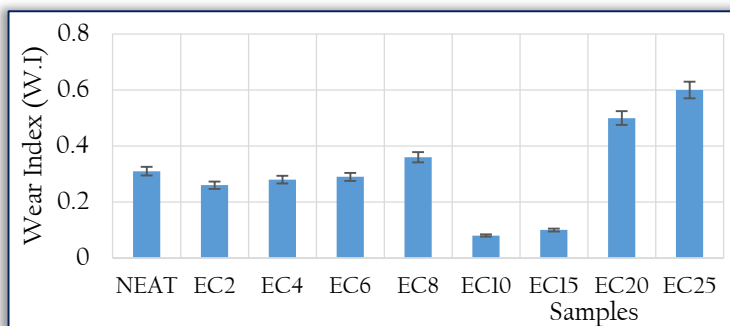


Figure 8. Wear index of the developed clay particle reinforced epoxy composites

particle increases from 2-8 before experiencing a decrease at 10 - 15 clay particle additions. Whereas composite sample EC10 which is made up of 10 wt. % clay particle shows a better wear resistance of 0.08 W.I compared to the neat sample which has a value of about 0.31 W.I was followed by sample EC15 made up of addition of 15 wt. % clay particle with a

value of 0.10 W.I. This can be attributed to the reinforcement being a ceramic material that is hard and brittle in nature as well as good interfacial bond occurring between the reinforcement and the matrix within this range as shown in Figures 2-3 (a). However, at higher addition of the clay particle to the matrix, the developed composites shows poor wear resistance compared to the neat at higher rate to what was obtained at lower reinforcement content. In the case of composites reinforced with hard particles, interfacial bonding between matrix and particle should be strong. When the reinforcement is not well bonded to the matrix, the reinforcement element will not contribute to the wear resistance and can even increase the wear rate of the composites. Since the matrix phases are generally softer than that of the reinforcement phases, the extent of debonding of the reinforcement phase can play a critical role in the wear behavior of the composite [26].

The voltage applied to the electric current which flows through the developed composites sample predicts the behavior of the composites. The resistivity of sample EC10 and EC15 which is made up of 10 and 15 wt. % clay particle was compared with the neat sample. It was observed from Figure 9 that the resistivity increases with increase in the reinforcement compared to the unreinforced which is the neat. The neat sample has the minimum resistivity and sample EC15 which is made up of 15 wt. % clay particle has the maximum resistivity, which indicates that the neat sample readily allows flow of electric current while sample made up of 15 wt. % clay particle has strong opposing flow of electric current.

Electrical conductivity of the neat, EC10 and EC15 samples was presented in Figure 10. It was observed that sample EC15 with addition of 15 wt. % clay particle reinforcement in the matrix shows the reciprocal of the composite electrical resistivity. This implies that, sample with 15 wt. % clay particle developed composite has high resistance to the flow of current compared to the neat with low resistance to the flow of current.

#### 4. CONCLUSION

It was observed from the outcome of the research that, 38  $\mu\text{m}$  particulate from Ikere clay deposit was a potential inorganic reinforcement materials that can be incorporated into epoxy resin for the development of polymer composites for electronic application due to the presence of some elements such as Si and Al that form refractory materials in high proportions, thereby, leading to the enhancement of the mechanical, wear and electrical properties of the developed composites.

The addition of clay particles within 10–15 wt. % gave the optimum reinforcement content for improved properties. Optimum performance in terms of wear and hardness properties with wear index of 84.35 and hardness value of 84.35 HRA were obtained from 10 wt. % clay particulate reinforced composite while 15 wt. % clay particulate reinforced composite has better impact strength of 11.19 J as well as electrical resistivity property needed for insulating application.

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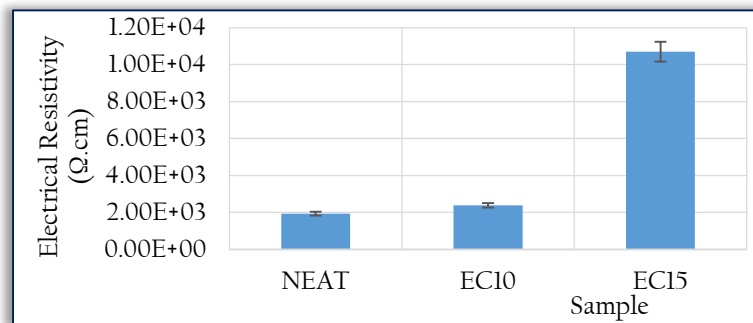


Figure 9. Electrical resistivity of neat, EC10 and EC15 samples

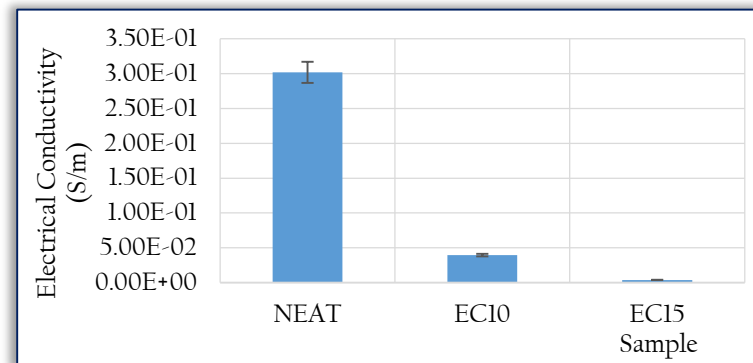


Figure10. Electrical conductivity of neat, EC10 and EC15 samples

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