

**MECHANICAL AND WEAR BEHAVIOUR OF EPOXY REINFORCED  
WITH ISAN-EKITI CLAY PARTICLES COMPOSITES**

**BY**

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**APPROVAL PAGE**

This project report has been approved as meeting the requirements for the award of Bachelor of Engineering (B.Eng.) Degree in Materials and Metallurgical Engineering Federal University Oye-Ekiti, Ekiti State.

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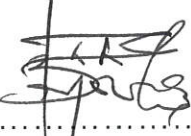
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## CERTIFICATION

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## DEDICATION

**This project is dedicated to God Almighty.**

## ACKNOWLEDGMENT

My profound gratitude is expressed to the Almighty God for giving me life, knowledge and resources to complete this study.

I express my unalloyed regards, indebtedness and deep appreciation to my project supervisor and all the lecturers in Materials and Metallurgical Engineering Department Federal University Oye Ekiti, for scholarly criticisms, meticulous teachings and sagacious advice throughout the study.

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## ABSTRACT

This study is about the production and characterization of epoxy-clay composite. Composites were produced at 2%, 4%, 6%, 8% and 10% volume fraction of Clay fillers using 150 micron under size, sieve size and the epoxy was cast neat at 0% Clay which served as the control.

The Mechanical properties of the composites such as tensile properties (Young's modulus, tensile strength, energy at break and extension at break), flexural strength, wear behaviour, hardness and water absorption test were experimentally determined in the engineering laboratory using universal testing machine, flexural testing machine, taber abrasive tester and vickers hardness testing machine respectively. Results of the mechanical property tests showed low gain tensile strength and Young's modulus. The tensile properties showed: that at 4% Clay the highest tensile strength of 17.08MPa was obtained, the cast neat epoxy (control 0% Clay) had the best Young's modulus of 590.50MPa and energy at break improved at 2% and 6% as volume fraction of clay increased. There was gain in extension at break, at 8% as the energy of clay fillers increased. The hardness values of the composites were higher as compare with cast neat epoxy. The hardness of the composites increased from 2% - 8% as the clay filler increases and drop at 10% which is even higher than the neat. The wear behaviour of the cast neat epoxy, 2%, 10% decreases, while the wear behaviour increased at 8% as the clay fillers increases.

It was deduced from the study that epoxy-clay composite is suitable for engineering applications in areas where light weight and resistance to breakage are required such as in producing electronic casing, handle of kitchen utensil, sole of shoes, insulators, tables and slabs.



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## CHAPTER ONE

### INTRODUCTION

#### 1.1 Background Information

Most times engineers are confronted with the duty of developing a new material that has light weight, low cost and good mechanical properties. A likely alternative to this duty is to use a low density particulate material like clay in a polymer matrix to form a polymer composite.

Polymer/clay composites are new class of composite materials, in which clay as a layered silicate is distributed in micro scale size in a polymer matrix (Sinha Ray and Okamoto, 2003). Recently, Polymer clay composites have attracted major academic and industrial attention. This attention stems from the fact that micro sized-layer-filled polymers can display vivid improvements in mechanical and thermal properties at low clay contents because of the strong synergistic effects between the polymer and the silicate platelets on both the molecular and metric scales. The likely properties enhancements of Polymer clay composites have led to increased application in several fields such as the packaging industry (bottles, containers, and plastic films), automobile industry (exterior and interior body parts and fuel tanks), coating industry (paints, wire enamel coatings, etc.), aerospace industry (body parts of airplane and exterior surface coatings) and electronic industry (packaging material and exterior parts of electronic devices).

Epoxy resins have been widely used as impregnating materials, adhesives, or matrices for composites because of their good electric insulating, good chemical resistance, low shrinkage during cure, good thermal characteristics, and ease in processing. However, the major problems with the epoxy resins for engineering applications are their low stiffness and strength when compared with metals. One effective method for offsetting these deficiencies of pure

epoxy is incorporation of reinforcing fillers. Montmorillonite (MMT) clay has been well documented as the best reinforcement materials for polymer composites due to its high aspect ratio, low cost, and the fact that it consists of layered silicates which can be inserted individually in size by polymer chains (Gu and Liang, 2003).

The dispersion of clay particles in a polymer matrix results in the formation of three forms of composite materials (Giannelis *et al*; 1999).

- i. The first form is conventional phase separated composites in which the polymer and the inorganic host remain immiscible resulting in poor mechanical properties of the composite material.
- ii. The second form is intercalated polymer clay composites, which are formed by the insertion of one or more molecular chains of polymer into the interlayer or gallery space.
- iii. The last form is exfoliated or delaminated polymer-clay composites, which are formed when the clay layers are individually dispersed in the continuous polymer matrix. Exfoliated polymer-clay composites are especially desirable for improved properties because of the large aspect ratio and homogeneous dispersion of clay and huge interfacial area (and consequently strong interaction) between polymer chains and clay layers.

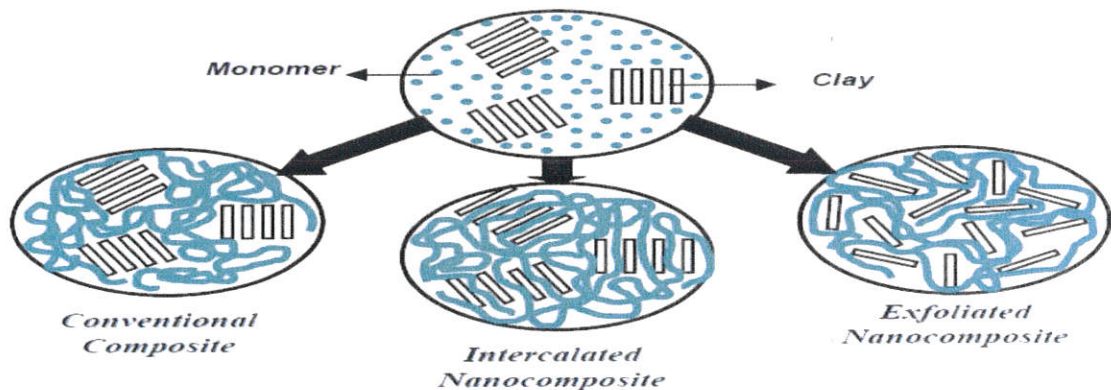


Figure 1.1: The three structures of polymer-clay composites system

A composite material is a microscopic or macroscopic mixture of two or more distinct materials with an identifiable interface between them. In a composite material the constituents do not dissolve or combine completely in one another. Normally the components in a composite material can be physically identified and they reveal interface between one another. A particulate composite comprises of a matrix reinforced with a dispersed phase in form of particles. Soft particles like coir dust, rice husk flour, baggase ash, sawdust, rice husk ash, and clay can be dispersed in a harder matrix to improve machinability and reduce coefficient of friction (Lake, 2002; Jiquao *et al*; 2010).

Unique benefit of a composite is that two or more materials could be combined to take advantage of the good characteristics of each of them.

Composites are gaining a wide range of applications in engineering because of the following benefits: weight savings, corrosion resistance, easy manufacturing, low temperature processing, possibility of producing novel shapes, reduced parts and long fatigue life (Nowosielki *et al*; 2006; Ranganathathiah, 2010).

Composites can be made of two or more components, the matrix and the dispersed phase. The properties of a composite material depend on the following: properties of the matrix; properties and distribution of reinforcement, nature of bonding at the interface and volume fractions occupied by the constituents (Lake, 2002).

A matrix is a material in which the reinforcements or other components of a composite system are inserted. It can be made of metal, ceramic or polymer (Askeland, 1994). The function of the matrix is to bind the reinforcements together by virtue of its cohesive and adhesive characteristics, to transfer load to and between the reinforcements and to protect the reinforcements from environment and handling. The matrix is often the weak connection in a

composite when observed from a structural perspective. Chemical treatment of reinforcement materials or filler boosts interfacial adhesion between the matrix and fillers leading to better mechanical properties of the composites (Gauthier *et al*; 1998). It is in view of these expected roles of matrix materials that epoxy resin was used in this study.

A polymer matrix composite is a composite made by the combination of a Polymer (resin) matrix and a fibrous reinforcing phase (Ezuanmustapha *et al*; 2005). Polymer composites are gaining importance as substitute for metals in applications in the aerospace, automotive, marine, sporting goods and electronics industries due to their light weight and corrosion resistance. Polymer matrix can be classified as thermoplastics and thermoset. Thermoplastics include low density polyethylene, high density polyethylene, nylon, polypropylene and polyester while epoxy resin is an example of a thermoset.

Epoxy resin is currently of much research interest due to its superior properties over polyester resin. Some of the properties of the epoxy resin identified by researchers are low cure shrinkage, better resistance to moisture, better mechanical properties, processing flexibility and better handling.

Epoxy resins are presently used more than all other matrices in advanced composite materials for structural applications in the United States of America (USA) Air Force and Navy. The dispersed phase of a composite refers to the reinforcement or fillers added in the matrix and the role of reinforcement in a composite material is to increase the mechanical properties of the neat resin system (Askeland, 1994).

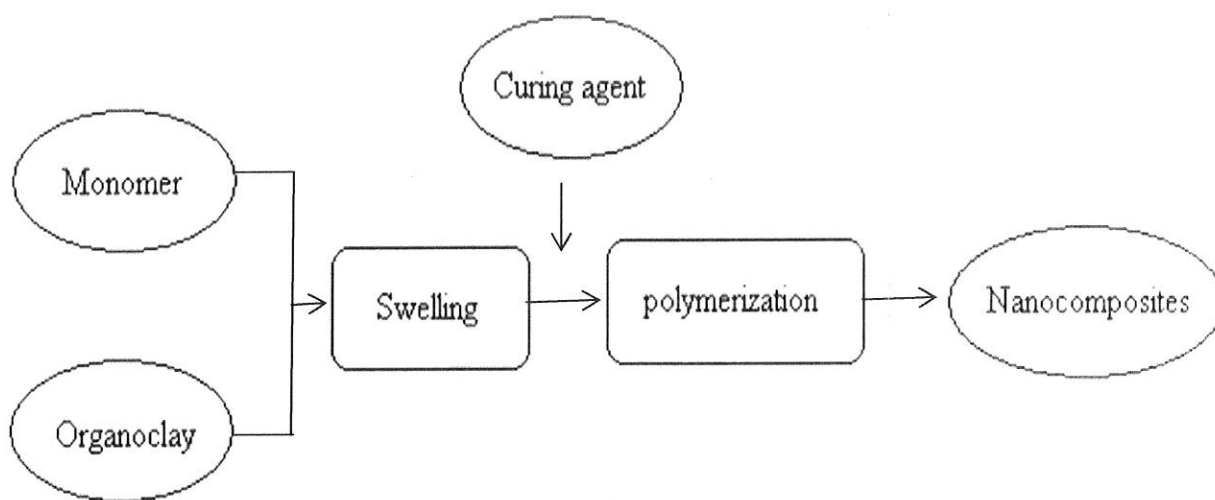
Filler materials are generally the inert materials which are used in composite materials to reduce cost absorb thermal stresses, improve mechanical properties to some extent and in some cases to improve processing (Singla and Chawla, 2010). Fillers which increase bulk volume and hence



reduce cost are known as extender fillers while those that improve mechanical properties particularly tensile strength are termed reinforcing fillers (Igwe and Onuegbu, 2010).

Most significant factors for success in preparing epoxy/clay composites are types of epoxy resin and curing agent/hardener used. Epoxy/clay composites based on diglycidyl ether of bisphenol A (DEGBA) resin have been synthesized using a wide range of curing agents including triethylenetetramine (TETA) (Mohan *et al*; 2005), diaminodiphenyl methane (DDM) (Nigam *et al*; 2004), diaminodiphenylsulfone (DDS) (Wang *et al*; 2006), and diethyltoluenediamine (DETDA) (Liu *et al*; 2004).

The epoxy/clay composites using a kind of epoxy resin of DEGBA and polyaminoamide as hardener were prepared by using an in situ polymerization method. The effect of clay addition to the neat epoxy on the tensile strength, flexural strength, hardness, water absorption and specific wear abrasion was investigated in this work.



#### In situ polymerization

**Figure 1.2: Flowcharts presenting the different steps in preparing polymer layered silicate composites.**

## **1.2 Statement of the problem**

Epoxy-clay composites are widely studied, and their formation mechanism and performances have been reported (Wang *et al.*; 1999). However, much is still unknown concerning the relationships between materials, synthesis, structure, wear, and properties of epoxy-clay composites, especially for mechanical/wear behaviour of epoxy reinforced with Isan-Ekiti clay. For example, exfoliated composites are the objective of most researchers, because they provide dramatic improvements in mechanical and physical properties. However, it is extremely difficult to synthesize completely exfoliated composites. Organoclays are not uniformly distributed, and agglomerates of organoclays are observed in epoxy resins. As a result, the biggest task is how to exfoliate platelets of clay in epoxy resins. The scientific challenges encountered in this project can be summarized as follows:

- i. How to disperse organoclays in uncured epoxy resins?
- ii. How to understand the formation of exfoliated composites?
- iii. How to understand and predict the improved properties of epoxy-clay composites?

## **1.3 Objectives of the Study**

The overall objective of this project is to characterize composite materials produced from different compositions of epoxy and clay. The specific objectives of the study are to:

- i. Prepare epoxy-clay composite using Isan-Ekiti clay particles of various size particles;
- ii. Examine the effect of clay volume fraction on some mechanical properties of epoxy-clay composite; and
- iii. Ascertain the suitability of the composite for engineering applications.

#### **1.4 Justification of the Study**

This study is helpful in understanding the potentials of clay as filler in composite production and the behaviour of Epoxy resins. The study is useful to engineers and researchers in the composite industry because it will help to suggest ways of improving the mechanical/wear behaviour of the epoxy-clay composite. Composite production can offer employment opportunity to unemployed youths due to low energy and machinery requirements for production. The knowledge of mechanical and wear properties of particle filled composites is vital in describing the behaviours at the interface and the effect of forces on the composites. Proper understanding of the mechanical and wear properties of composites will help to ascertain the engineering application of composite in structures, industries, electronics, oil and gas, and other industrial production.

#### **1.5 Scope and Limitations of the Study**

Experimental method was used in this study involving composite production as well as the determination of the mechanical/wear properties of the composite material. The composites were produced at 0%, 2%, 4%, 6% 8% and 10% volume fraction of clay fillers, 150 micron sieve size was used for the study. The clay used in the study was sourced from Isan-Ekiti, Ekiti State, Nigeria. Limitations faced in the research were sourcing the epoxy resin and getting the characterization equipment.

## CHAPTER TWO

### LITERATURE REVIEW

#### 2.1 Overview of Composites

The encyclopedia of life support defined composite material as a multiphase material in which the phase distribution and geometry have been controlled in order to optimize one or more properties. The intention of producing a composite material is to make a material that combines the best properties of the components whilst eliminating any poor properties. Composites are also materials that comprise strong load carrying materials known as reinforcement embedded in a weaker material known as matrix (Askeland, 1994).

Reinforcement provides strength and rigidity, helping to support structural load. The matrix or binder (organic or inorganic) maintains the position and orientation of the reinforcement.

The use of composites started many centuries ago. The Book of Exodus in the Christian Bible recorded that straws were used to produce rigidity and strength in mud walls. Historical examples include the use of bamboos as a reinforcing material in **mud walls** in houses by Egyptians (15000BC) and laminated metals in the forging of swords (1800AD). In the 20th century, modern composites were used in the 1930's, where glass fibers reinforced resins. Boats and aircrafts were built out of these glass composites commonly called fiberglass.

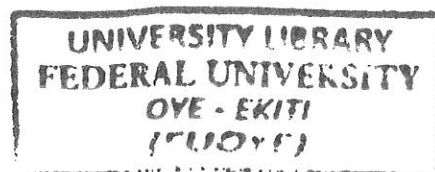
Since the 1970's the application of composite materials has widely increased due to development of new fibers such as carbon, boron and aramids and new composite systems with matrices made of metal and ceramics.

Singla and Chawla, (2010) produced epoxy-fly ash composites at high filler loading using fly wheel ash particles. They found that as the Fly wheel ash particles loading increased

the compressive strength of the composites increased while the impact strength reduced. Kulkurni and Krishore, (2003) studied the mechanical properties of fiber epoxy composites. The result of the study showed that the addition of fly ash particles to epoxy matrix led to reduction of the density and increase in the modulus of the composites. According to Cattaleeya *et al*; (2008) rice husk ash is not considered good reinforcing filler in rubber composites due to the large particle size and low reactive functional group at the filler surface. The reinforcing effect of rice husk ash is not as good as silica and carbon black, but is only comparable to calcium carbonate ( $\text{CaCO}_3$ ). Cattaleeya *et al*; (2008) were of the view that chemical surface treatment of rice husk ash prepared under special condition is effective in reinforcement only at low filler loading.

Hathapamit, (2003) compared the suitability of silica particles and rice husk ash particles for embedding composites in electronic devices. In his study he found that silica filled epoxy composites had better tensile strength than the rice husk ash filled epoxy composites but the mixing viscosity, water absorption and coefficient of thermal expansion were better than the silica filled composites. Osureminda and Abode, (2010) reinforced rubber products with baggase ash fillers and succeeded in improving the tensile strength, abrasion resistance and hardness of rubber composites with increasing filler loading while the elongation at break and compressive strength decreased.

According to Hanseung *et al*; (2008) rice husk flour is not good reinforcing filler for polymer composites but can be utilized as biodegradable filler in polymeric materials to minimize environmental pollution. They found from the study of mechanical properties of polypropylene composites that addition of rice husk flour decreased the mechanical properties of



the composites due to poor interfacial bonding between the matrix and the filler and holes formed in the microstructures because of pulling of filler particles in the matrix.

Ahmadi *et al;* (2007) found that replacement of cement with rice husk ash up to 20% volume in the matrix will improve the mechanical properties of the concrete. The researchers posited that using pozzolans like rice husk ash will reduce the utilization of cement and lower the cost of buildings.

Yunfu *et al;* (2008) were of the view that particle size, particle matrix interface adhesion and particle loading affect mechanical properties of composites. They found that introduction of rigid fillers like  $Al_2O_3$ , glass,  $CaCO_3$  into epoxy matrix normally result to decrease in mechanical properties. Yunfu, *et al;* further stated that coupling agents should be used in filler composites to improve adhesion and that tensile modulus is not affected by adhesion but by filler loading. The researchers found that using nano fillers increased the stiffness of composites.

The findings of Sapuan *et al;* (2005) showed that fiber treatment improved interfacial bonding between fiber and epoxy matrix and led to better mechanical properties of the spathe-fibre reinforced composite laminates.

Arukalam and Madufor, (2011) studied the effect of filler loading on some mechanical properties of calcite-filled low density polyethylene composite. They found that as the filler loading increased tensile strength, tensile modulus and elongation at break (ductility) of the composites reduced. There were gains in hardness of the composites because calcite obtained from snail shell is harder than the low density polyethylene matrix. Srinavasa and Bharath, (2011) investigated the hardness and impact properties of Areca fiber epoxy-reinforced composites. They treated areca fibers extracted from areca husk with potassium hydroxide to get better interfacial bonding between the fiber and the epoxy matrix. Mechanical properties of the

composites increased as the fiber volume fraction and composite post curing time increased. They concluded that mechanical properties of fiber reinforced composites depend on the nature of matrix material, the distribution and orientation of reinforcing fibers and the nature of the fiber matrix interfaces and interphase region.

According to Srinavasa and Bharath, Arecafiber, – epoxy reinforced composite is a good material in fabrication of lightweight materials used in automobile body building, office furniture, packaging industry and partition panel.

Egwaikhade *et al*; (2007) developed rubber composites using palm kernel shell husk. The study proved that palm kernel husk is potential filler for natural rubber compounds because addition of palm kernel shell husk fillers improved some mechanical properties.

Due to the continuous decrease of mechanical properties at higher filler loadings associated with untreated fibers and fillers most researchers have advocated the coating of fillers with coupling agents. According to the report from handbook of USA Department of Defense (2002) coupling agents are substances that are used in small quantities to treat a surface so that bonding occurs between it and other surfaces. Coupling agents include bonding agents and surfactants (surface reactive agents), compatibilizers and dispersing agents. Organic coupling agents produce stronger adhesion and include isocyanides, anhydrides, silanes and anhydride modified co-polymers.

Gauthier *et al*; (1998) reported that adhesion can be improved using coupling agents like maleic anhydride to incorporate hydroxyl groups on the matrix through hydrophilization and consequently enhancing wetting effect of the resin on the fillers.

Ranganathathiah, (2010) did a comparative study of mechanical properties of composites formed from epoxy matrix modified with amine containing silicone and unmodified epoxy

matrix using fly ash and calcium aluminosilicate as fillers. He discovered that the fillers interacted favorably with the modified matrix and mechanical properties were improved at higher loads.

Ezuanmustapha *et al*; (2005) incorporated 4wt% of a coupling agent matrix anhydride modified polypropylene into composites prepared with rice husk and polypropylene matrix. It was observed that the addition of MAPP coupling agent increased the flexural strength of the composites but decreased the flexural modulus and impact strength. Razman *et al*; (2003) studied the effect of chemical modification of rice husk and reports that with chemical modification the reinforcing effect can be increased to an acceptable limit.

## **2.2 CLAY STRUCTURE**

### **2.2.1 Formation of Clays**

Clays form at the earth's surface, either in contact with air or with covering water bodies. The majority of clay formed is through the process of weathering, either sub-aerial or sub-aquatic. Sedimentation and burial changes the clay from one structure to other structures. Some clay is produce by hydrothermal processes, that is, water/rock interaction at temperature of 100-250 °C. The high quality clays used in industrial processes are produced by the hydrothermal method (Velde, 1992: Chapman and Hall, 198).

### **2.2.2 Structure of Clays**

Clay mineral is the broad term given to a group of minerals based on the general structure formed with the individual particles. Another term often used to describe the clay mineral family is layered silicate. This name gives an indication of the overall structure of the clays. Clays consist of a number of layers stacked one upon the other and within each layer is either two or



three sheets. The number of sheets is dependent on the group of clay minerals the materials falls into. The kaolin group consists of two sheets, while all other groups consist of three sheets. The sheets within the layers are based primarily on Silicon or Aluminium. The clay family can be divided into the following groups; kaolin, mica, smectite and talc.

Clay has been used as filler in polymers for many years, such as kaolin, bentonite and attapulgus (Seymour and Deanin, 1987); however it plays little functional role, and mainly serves economic considerations. It was not until the early 1990's that clay received great attention from the polymer industry, when Toyota's researchers discovered the possibility of constructing a nanostructure with a polymer and an organoclay (Okada *et al*; 1990 and Kojima *et al*; 1993)

### **2.2.3 Applications of Clay**

From prehistoric times, clay has been indispensable in architecture, in industry, and in agriculture. As a building material, it is used in the form of brick, either sun-dried (adobe) or fired. Clays are also of great industrial importance, e.g., in the manufacture of tile for wall and floor coverings, of porcelain, china, and earthenware, and of pipe for drainage and sewage. Highly absorbent, bentonite is much used in foundry work for facing the molds and preparing the molding sands for casting metals. The less absorbent bentonites are used chiefly in the oil industry, e.g., as filtering and deodorizing agents in the refining of petroleum and, mixed with other materials, as drilling muds to protect the cutting bit while drilling. Other uses are in the making of fillers, sizing, and dressing in construction, in clarifying water and wine, in purifying sewage, and in the paper, ceramics, plastics, and rubber industries.

## 2.3 Epoxy

Epoxy is also known as polyepoxide. It is a thermosetting polymer formed from the reaction of an epoxide resin with a polyamine hardener. Epoxy is a copolymer because it is formed from two different chemicals referred to as resin and hardener.

An epoxy resin contains oxirane or ethoxyline groups. Epoxy resins are mostly produced by the reaction between bisphenol A with epichlorohydrin in the presence of a basic catalyst.

Examples of commercial epoxy resins are diglycidylether of bisphenol A (DGEBA), glycidylethers of novolac resins and phenoxy epoxy resin. Properties of epoxy resins are: low viscosity, low shrinkage, low glass transition temperature, good adhesion to other materials, good chemical and environmental resistance, good insulating properties, and ability to be processed under variety conditions and good mechanical properties (May, 1988; Singla and Chawla, 2010 and Turner, 2007). The structure and reaction of a typical epoxy resin is shown in figure 2.1.

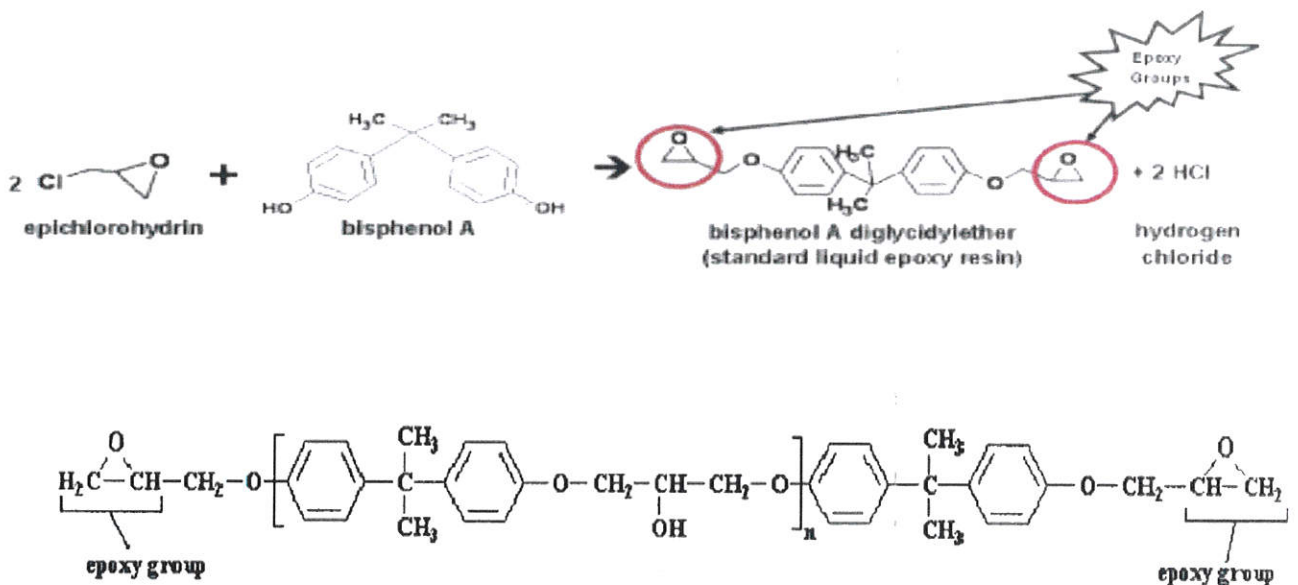


Figure 2.1: Reaction and structure of a typical epoxy resin diglycidylether of bisphenol A,  $n$  is the degree of polymerization and ranges from 0 to 25. (Nnaji, 2012).

### **2.3.1 Curing of Epoxy Resins**

Curing process is a chemical reaction in which the epoxide groups in epoxy resins react with curing hardener in order to convert epoxy resin into a hard, infusible and rigid material.

Curing process normally involves an exothermic reaction. Epoxy resin cure quickly and easily at practically any temperature from 5 – 150 °C depending on the choice of curing agent. Curing agents are amines, polyamides, phenolic resins, anhydrides and isocyanates (Pascault and Williams, 2010).

### **2.3.2 Engineering Applications of Epoxy**

Epoxy resins were first commercialized in 1946 and are widely used in industry in the following areas:

- i. Protective coatings: Epoxy resins can be applied in corrosion protection of steel pipes and fittings used in oil and gas industry (wikipedia.org.Epoxy).
- ii. Structural applications: Epoxy has been applied in the production of composites, molds, industrial tools, laminates, casings, fixtures, automobile bumper and other industrial production aids.
- iii. Electrical systems and electronics: Epoxy has excellent insulating abilities that protect electrical components from short circuiting, dust and moisture. This has resulted to usage in motors, generators, transformers, switch gear and insulators. It is equally used in making integrated circuits, hybrid circuits and printed circuit boards.
- iv. Singla and Chawla, (2009) reported that epoxy can be applied in production of military aircraft and commercial aircraft.
- v. Consumer and Marine Applications: Epoxy is suitable for production of shoe, furniture, boats and ship component.

- vi. Adhesives: Epoxy adhesives are used for construction of aircrafts, bicycles, golf clubs, snow board and other applications where high strength bond is required (sunilbhangletripod.com: wikipedia.org.Epoxy).

#### **2.4 Flexural Strength (MPa)**

Flexural strength is the ability of an engineering material to resist bending or twisting under load. It is known as modulus of rupture, bend strength or fracture strength. Flexural strength is a mechanical parameter for brittle materials. Flexural strength represents the highest stress experienced within the material at its moment of rupture (Hogkinson, 2000).

#### **2.5 Hardness**

Hardness is the resistance of a material to localized plastic deformation; it indicates wear resistance and resistance to scratching, abrasion and indentation (Askeland, 1994). Hardness testing can be done with Rockwell, Vickers, Brinell sclerescoper, durometer, rebound and barcol hardness tester.

#### **2.6 Tensile Properties**

According to Liu, (1999) tensile properties indicate how materials will react to forces applied on tension. Tensile properties are determined by performing a tensile test. Tensile test is a simple uniaxial test that consists of slowly pulling a sample of material in tension until it breaks. These properties can be found from a tensile test: modulus of elasticity, elongation at break, tensile strength, tensile stress and tensile strain.

### 2.6.1 Stress (MPa)

Tensile stress of the material is defined as the force per unit area as the material is stretched (Liu, 1999). The area used in finding tensile stress is the original undeformed cross sectional area because the cross sectional area of a material may change if the material deforms on stretching.

$$\text{Tensile stress } (\sigma) = \frac{\text{Load of force}}{\text{Original crosssectional area}} = \frac{P}{A} \quad (2.1)$$

### 2.6.2 Strain

According to Onwuka, (2001) Strain is the non-dimensional measure of deformation of a material with respect to a given length dimension of that material. Tensile strain or engineering strain is the change in length of a sample of material divided by the original length or gauge length of the sample. Strain can be represented thus,

$$\begin{aligned} \text{Strain} &= \frac{\text{Change in length}}{\text{Original length}} = \frac{\text{Change in length}}{\text{Gauge length}} \\ &= \frac{L_f - L_o}{L_o} = \frac{\Delta L}{A_o} \end{aligned} \quad (2.2)$$

Where  $L_o$  = Original length

$\Delta L$  = Axial deformation or change in length

$L_f$  = final strength

$A$  = Original cross sectional area

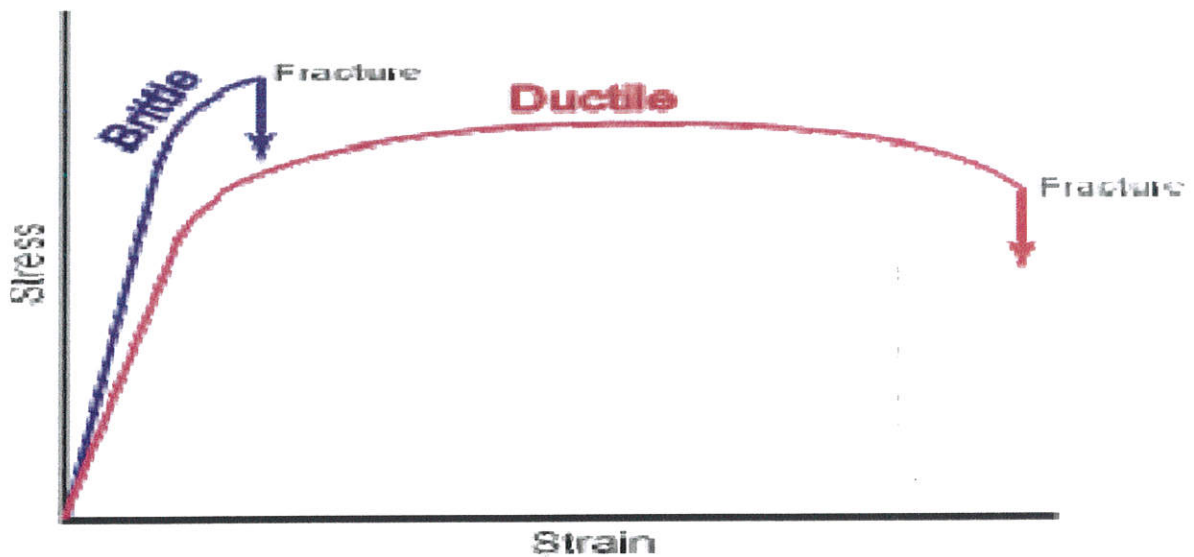


Figure 2.2: Stress- strain curve

### 2.6.3 Tensile Strength (MPa)

According to Askeland, (1994) Tensile strength or ultimate strength is the maximum amount of tensile stress that a material can absorb before breaking. It is the maximum tensile stress reached on a stress-strain diagram. Tensile strength of a material is affected by the preparation of the test specimen, the presence of surface defects (voids, porosity and inclusions), the temperature of the test environment and the nature of the material.

$$\text{Tensile strength} = \frac{\text{Maximum Tensile Force Applied}}{\text{Original Crosssectional Area.}} \quad (2.3)$$

### 2.6.4 Young's Modulus (MPa)

Young's modulus or modulus of elasticity (E) is the slope of the stress- strain curve (Askeland, 1994). According to Hooke's law it is a measure of the stiffness of the material. Stiffness is the property of a material to resist deformation in the elastic range or within the

proportional limit. Young's modulus (E) =  $\frac{\text{Stress}}{\text{Strain}}$  (2.4)

### 2.6.5 Elongation at Fracture (%)

Elongation at fracture is the strain on a sample when it fractures. It is usually expressed in percentage and is a measure of the ductility of the material. Elongation at fracture is the amount of uniaxial strain at fracture and is depicted as strain. Elongation at fracture is mostly calculated by removing fractured specimen from the grips, fitting the broken ends together and measuring the distance between gauge marks.

$$\% \text{ Elongation at fracture} = \frac{L_f - L_0}{L_0} \times 100$$

Or  $\frac{\text{Extension}}{\text{Original length}} \times 100$  (2.5)

$L_f$  = final length of tensile test specimen at rupture.  $L_0$  = initial length of test specimen.

### 2.7 Wear Behaviour Test

Wear test is carried out to predict the wear performance and to investigate the wear mechanism (Li, 2002). Two specific reasons why wear test is carried out:

- i. From a material point of view, the test is performed to evaluate the wear property of a material so as to determine whether the material is adequate for a specific wear application.
- ii. From a surface engineering point of view, wear test is carried out to evaluate the potential of using a certain surface engineering technology to reduce wear for a specific

application, and to investigate the effect of treatment conditions (processing parameters) on the wear performance, so that optimized surface treatment conditions can be realized.

$$\text{Wear index} = \frac{W_1 - W_2}{RPM} \times 100 \quad (2.6)$$

## 2.8 Water Absorption Test

The samples initial weights were taken with the aid of an electronic weighing scale. Each of the samples was immersed in a water container for four days. On removal from water, the surfaces of the specimens were cleaned dry and weighed immediately, and the new weights of the samples were recorded.

Water absorption is a measure of material ability to absorb moisture (water). The increase in weight was recorded as percentage gained and is expressed by;

$$Mt (\%) = \frac{W_w - W_d}{W_d} \times 100 \quad (2.7)$$

## 2.9 Adhesion and Cohesion

Adhesion is an attraction process between dissimilar molecular species which have been brought into direct contact such that the adhesive (binder or matrix) binds to the applied surface. Cohesion is an attraction process that occurs between similar molecules, primarily as the result of chemical bonds that have been formed between the individual components of the adhesive or bonding agent for example curing of cast neat epoxy.

Cohesion may be defined as the internal strength of an adhesive due to various interactions within that adhesive or binder that binds the mass together whereas adhesion is the bonding of one material to another namely an adhesive to a substrate, due to a number of



different possible interactions at the adhesive substrate interface, The strength of adhesion between two materials depends on the interactions between the two materials and the surface area which the materials are in contact. (Fraunhofer, 2010). Adhesion is necessary for achieving high level of mechanical properties.

The various types of adhesion are:

**Chemical adhesion:** It is a type of adhesion in which the cohesive strength of a matrix material is determined by chemical bonds within the matrix material, chemical bonds due to cross linking of polymer(s) within a resin based material, inter molecular interactions between adhesive (binder) molecules and mechanical bonds and interactions between the molecules in the adhesive (resin, binder)

Wetting is the ability of a liquid to form an interface with a solid surface (Fraunhofer, 2010). The two materials form a compound at the interface by Ionic or covalent bonds that results in strong bond between them. Such bonds are usually brittle except with nano particles.

**Dispersive adhesion:** This type of adhesion involves holding the surfaces of two materials together by Vander walls forces.

Diffusive adhesion involves merging or intermingling of the materials at the bonding interface by diffusion typically when the molecules of both materials are mobile and soluble. Example includes sintering of ceramics or powder metallurgy.

The types of adhesion explained above are the ones that affect mechanical properties of composites. Interfacial region is known as the adhesion region.

## 2.10 Calculation of Fiber Volume Fraction

The fiber volume fraction is mostly calculated using this formula

$$V_f = \frac{P_m W_f}{P_f W_m + P_m W_f} \quad (2.8)$$

Where  $V_f$  = volume fraction of fibers.

$W_f$  = weight of fibers

$W_m$  = weight of matrix

$P_f$  = density of fibers

$P_m$  = density of matrix

## CHAPTER THREE

### MATERIALS AND METHODS

#### 3.1 Materials

##### 3.1.1 Matrix Material

A standard epoxy resin of grade 3554A, chemically belonging to the epoxide family and curing hardener of grade 3554B were used as the matrix material. The resin and hardener were supplied by De Paragon Chuks Ventures (NIG) LTD, 195 Faulks Rd, chemical Zone Ariaria, Aba, Abia State. The epoxy resin has a density of 1.15g/cm<sup>3</sup>.

##### 3.1.2 Filler Material

###### Clay

The filler material used in this study is clay which was obtained from Isan-Ekiti, Ekiti State. The finely clay fillers were collected in large quantity and washed with water to remove impurities like dust, wood particles, fine sand particles and clay slurry. After washing, drying followed, drying with sun in order to remove moisture from it. After drying, the clay was crushed in order to obtain fine clay particles. Sieve Analysis was done on the fine clay particles, using Mechanical Sieve Shaker; 150 micron sieve size was used for the study. The chemical composition of Isan-Ekiti clay is shown in Table 3.1.

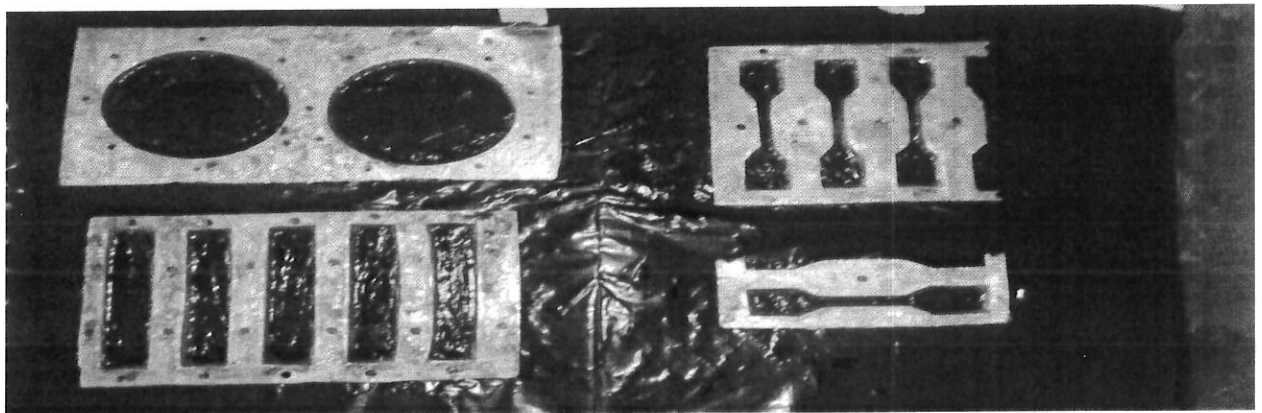
**Table 3.1: Chemical Composition of Isan-Ekiti Clay**

Chemical Compound	$Al_2O_3$	$SiO_2$	$Fe_2O_3$	MnO	CaO	MgO	$Na_2O$	$K_2O$	Loss on ignition (LOI)
Composition (%)	18.63	55.49	9.67	0.04	0.77	1.25	0.64	1.84	10.18

### 3.2.0 Methods

#### 3.2.1 Preparation of Composite Mould

Aluminium steel sheets were cut and formed into different sizes that served as moulds for the test samples. The testing techniques for the composite required that five sets of pattern (tensile, hardness, flexural, wear and water absorption) were produced. The patterns were made according to the required dimensions of the test samples. Flat wooden bars were used and served as mould control volume for other test piece samples. The molds were constructed to within  $\pm 1\text{mm}$  to give allowance for machining, and the surfaces were rubbed with wax releaser to ensure easy removal of the composite. Figure 3.2 below shows the mold used for composite production.



**Figure 3.1: Mold**

#### 3.2.2 Composite Fabrication

The composites were produced using facilities at Federal University of Technology Akure, (FUTA). Manual mixing method and hand lay-up technique were used for the composite production. The composites were prepared using 0%, 2%, 4%, 6%, 8% and 10% volume fraction of clay.

The matrix material (epoxy resin, grade 3554A and hardener grade 3554B) were prepared in the ratio of 2 parts of epoxy resin to 1 part of hardener (2:1). The measured volume of resin and hardener were mixed in a container and stirred at low speed for 5 minutes until the mixture

became uniform, tacky and exothermic reaction occurred. The clay filler were then added to the mixture of resin and hardener in the container and stirred at low speed for 5 minute. The matrix material and the filler (Composite) was poured into the mold and allowed to cure. To cast neat epoxy samples, the epoxy resin and hardener were poured into the mold and allowed to cure. The curing time was 24 hours at room temperature. Finally the composite plates were demolded and various mechanical property tests were carried out.

Tables 3.2 and 3.3 give the composition of the composites and dimensions for different mechanical property test.

**Table 3.2: Composition of the Composites.**

S/NO	Volume of Clay Filler (VF)	Volume of Epoxy Matrix (VM)
Sample 1	0%	100%
Sample 2	2%	98%
Sample 3	4%	96%
Sample 4	6%	94%
Sample 5	8%	92%
Sample 6	10%	90%

$$VF = 100 - VM \quad (3.1)$$

$$VC = VF + VM \quad (3.2)$$

Where VC is the volume of composite

**Table 3.3: Dimensions of specimen for different mechanical property tests**

S/NO	Test	Length (mm)	Width (mm)	Thickness (mm)
1	Tensile	40.00	10.00	4.70
2	Flexural	65.00	26.10	6.30
3	Hardness	40.00	10.00	4.70

### **3.3 Mechanical Property Tests**

#### **3.3.1 Tensile Testing of Composite Samples**

Tensile testing of the samples was done at Obafemi Awolowo University (OAU), Ile Ife. Universal Testing Machine (Instron Machine- Series 3369) was used in performing the tensile test.

The tensile test was conducted at room temperature according to ASTM D638 specification using an Instron-3369 universal testing machine at a cross-head speed of 5 mm/min. The dumbbell-shaped test specimen had a gauge length of 40mm, breadth of 10mm, and a thickness of 4.7mm for composites were made. At least five identical specimens were tested for each composites sample and averaged reported.

#### **3.3.2 Hardness Test of Composite Samples**

The hardness test was done at the Engineering Material Development Institute (EMDI), klm 4, Ondo Road, Akure, Ondo State. An automatic electric powered Vickers hardness testing machine was used to determine the hardness value of the cast neat epoxy and the composites. The procedure followed in performing the test were: switching on the machine, selecting the

desired load of 980.7mN, placing the surface of the specimen to be tested on the anvil of the machine and releasing the indenter of the machine from the lever until it touched the specimen making a green to be shown to indicate test zone specimen. The next thing done was pressing the test button and there was automatic indentation of the specimen by the conical shaped indenter of the Vickers tester. At the end of the indentation a red light showed “read” and instantly reading was directly done from the dial indicator. The value was reported as Vickers hardness value.

### **3.3.3 Flexural Strength Test of Composite Samples**

The flexural strength test was done at Obafemi Awolowo University (OAU), Ile Ife. Universal Testing Machine (Instron Machine- Series 3369) was used in performing the tensile test.

Each specimen of dimension 65mm by 26.1mm by 6.3mm was placed in the flexural testing machine. The three point flexing and loading arrangement was used in which fracture occurred at the middle. The specimen was flexed and flexural force that fractured the specimen at the middle was read from the scale of the machine. The flexural strength was calculated using equation 3.3.

$$F_s = \frac{FL}{bd^2} \quad (3.3)$$

Where F= flexural force (KN)

L = length in (mm)

B = breadth (mm)

D = thickness (mm)

F<sub>s</sub> = flexural strength.

### 3.3.4 Wear Behaviour Test of Composite Samples

The wear behaviour test was done at Federal University of Technology Akure, (FUTA). Taber Abraser Tester Machine was used in performing the wear behaviour test.

Taber tests involve mounting a flat specimen approximately 100mm square or round to a turntable platform that rotates on a vertical axis at a fixed speed.

Each specimen was placed on the Taber Abraser Tester Machine, the initial and final reading was taken. The wear behaviour test was calculated using equation 3.4

$$\text{Wear index} = \frac{W1-W2}{RPM} \times 100 \quad (3.4)$$

Where Rpm= 150

### 3.3.5 Water Absorption Test of Composite Samples

The samples initial weights were taken with the aid of an electronic weighing scale. Each of the samples was immersed in a water container for (78 Hours). On removal from water, the surfaces of the specimens were cleaned dry and weighed immediately, and the new weights of the samples were recorded.

Water absorption is a measure of material ability to absorb moisture (water). The increase in weight was recorded as percentage gained and is expressed by;

$$Mt (\%) = \frac{Ww - Wd}{Wd} \times 100\% \quad (3.5)$$





## CHAPTER FOUR

### 4.0

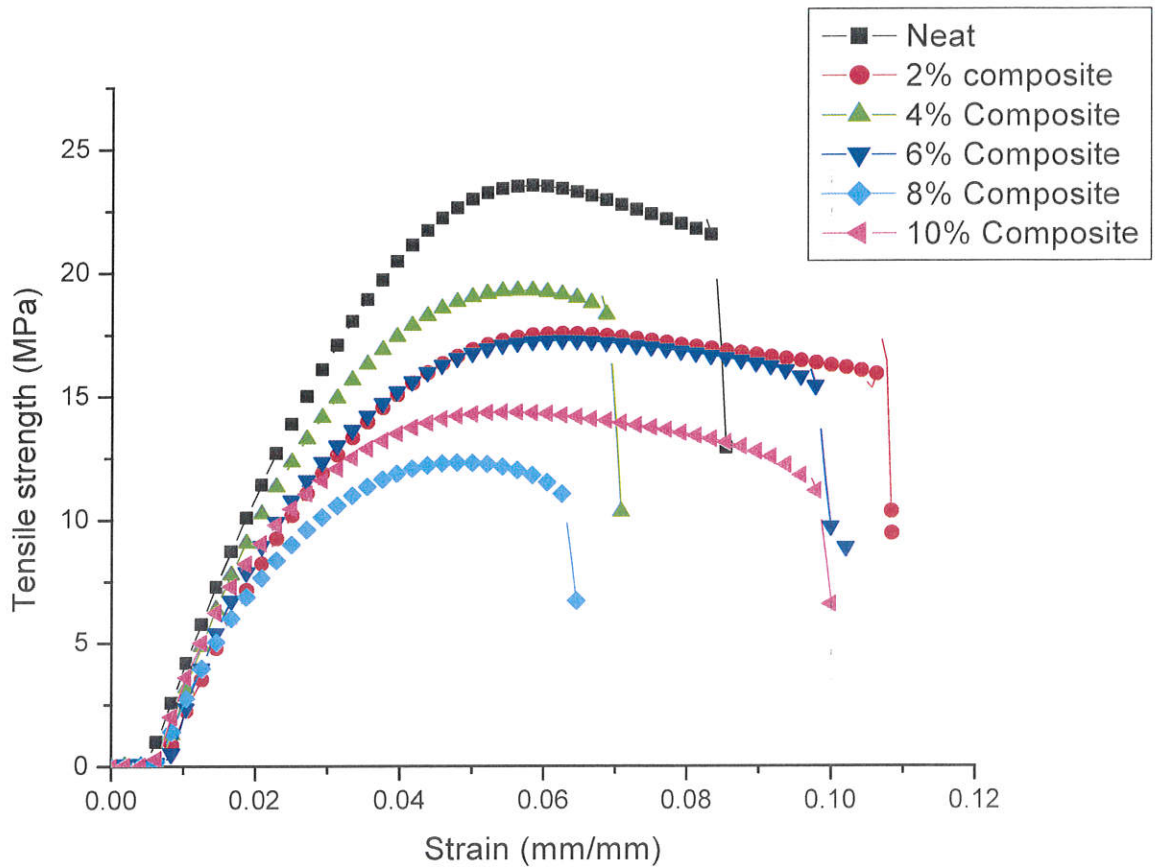
### RESULTS AND DISCUSSION

#### 4.1 Results

The results of the study are presented as follows, namely tensile test, flexural test, hardness test, wear behaviour test, water absorption test, Young's modulus, energy at break, extension at break,. Result of mechanical properties shows the behaviour of the composites and cast neat epoxy.

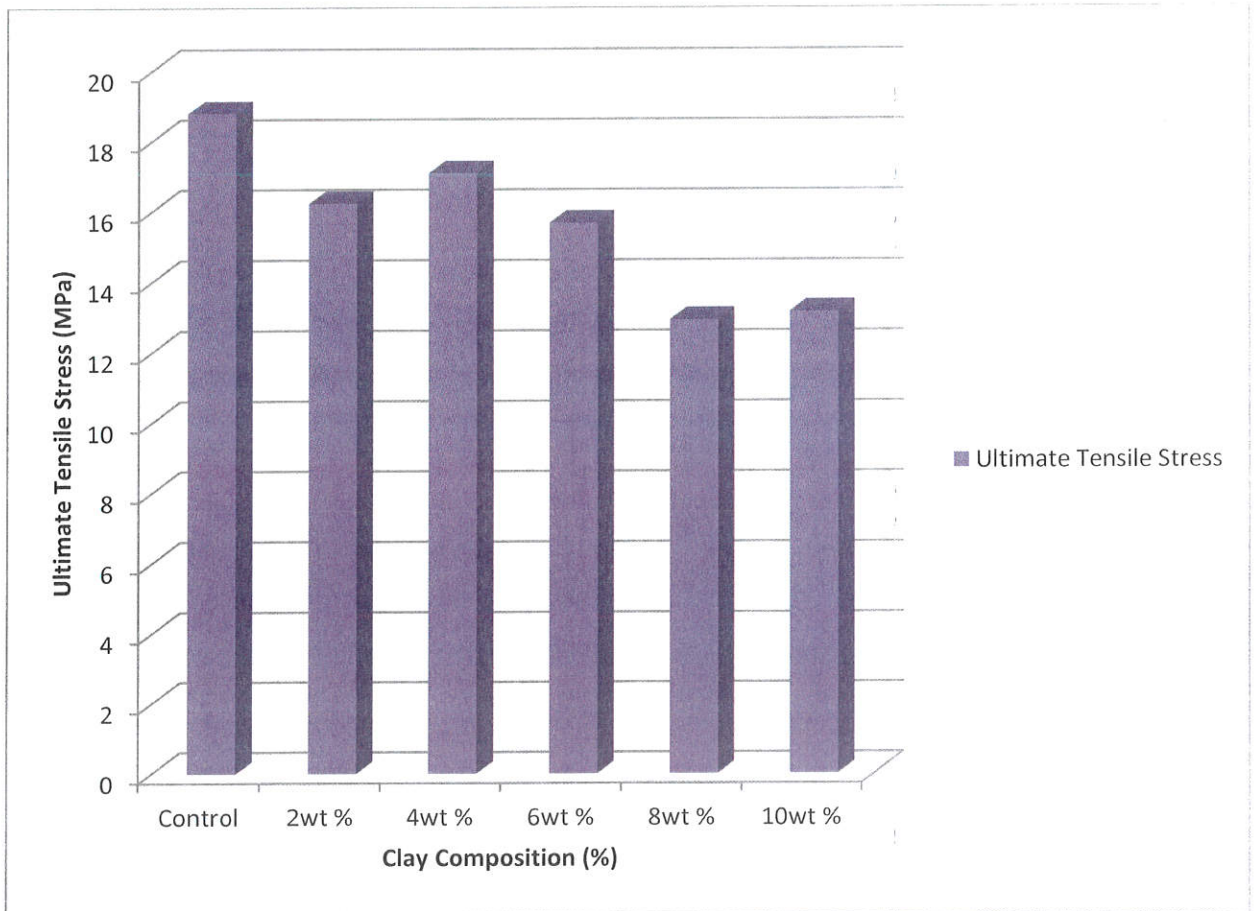
**Table 4.1: Tensile properties of neat and Clay reinforced Composites**

S/N	Material	Tensile Strength (MPa)	Young Modulus (MPa)	Energy at Break (J)	Extension at Break (J)
1	100% Epoxy 0% Clay	18.78512	590.50	1.78	2.89
2	98% Epoxy 2% Clay	16.21564	390.70	1.89	3.38
3	96% Epoxy 4% Clay	17.07848	485.39	1.29	2.42
4	94% Epoxy 6% Clay	15.66046	356.93	1.90	3.50
5	92% Epoxy 8% Clay	12.90602	282.49	1.75	5.00
6	90% Epoxy 10% Clay	13.14004	253.63	1.30	2.75



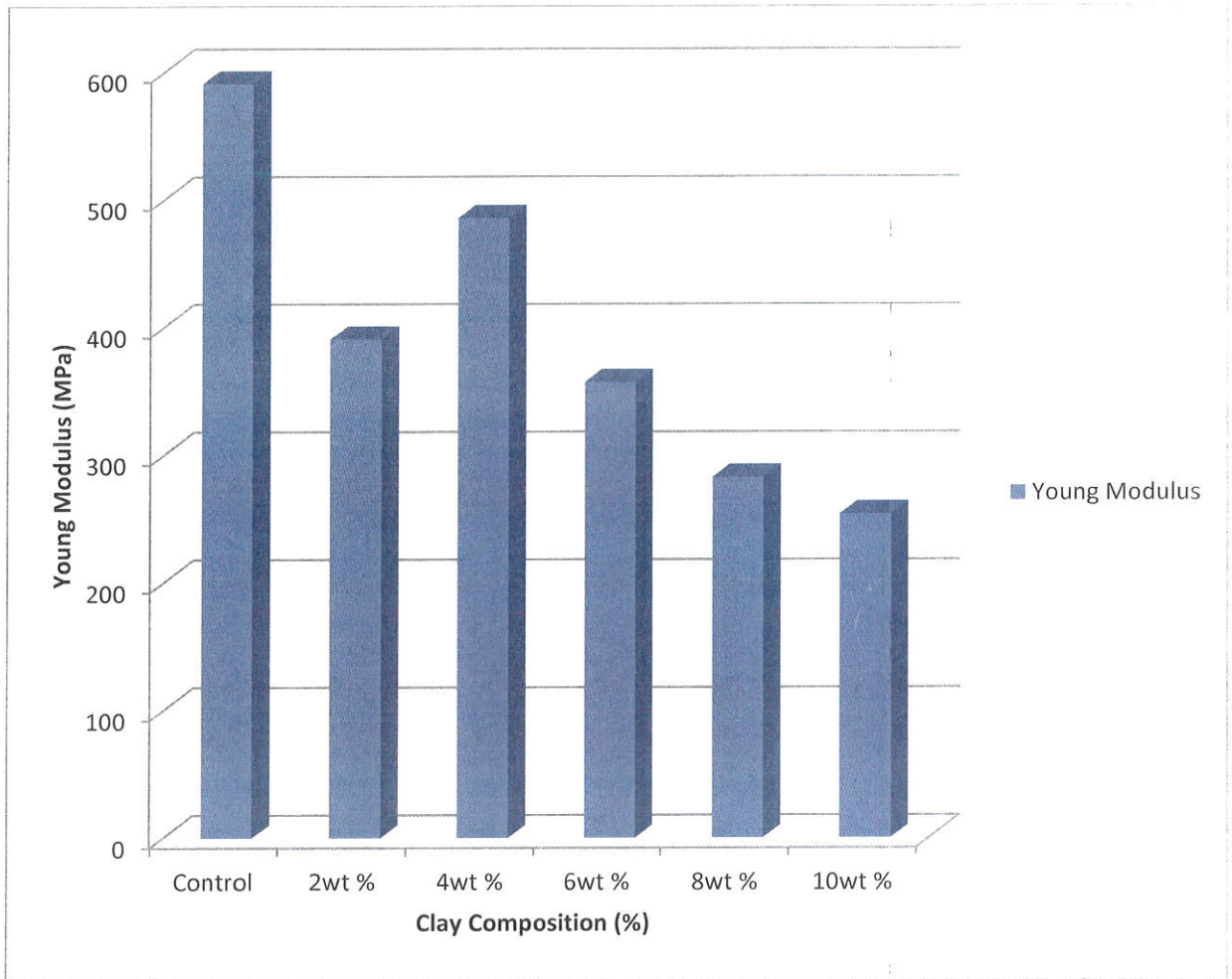
**Figure 4.1: Stress- Strain curves of Composites**

Figure 4.1 shows the stress-strain curves of the neat and reinforced composites. It was observed that the addition of clay particles<sup>6</sup> reduces the tensile properties of the composites when compared with the neat. This may be attributed to the formation of non-homogeneity observed in the composites produced. It was also observed that the tensile properties of the neat composites were better than that of the clay particle reinforced composites.



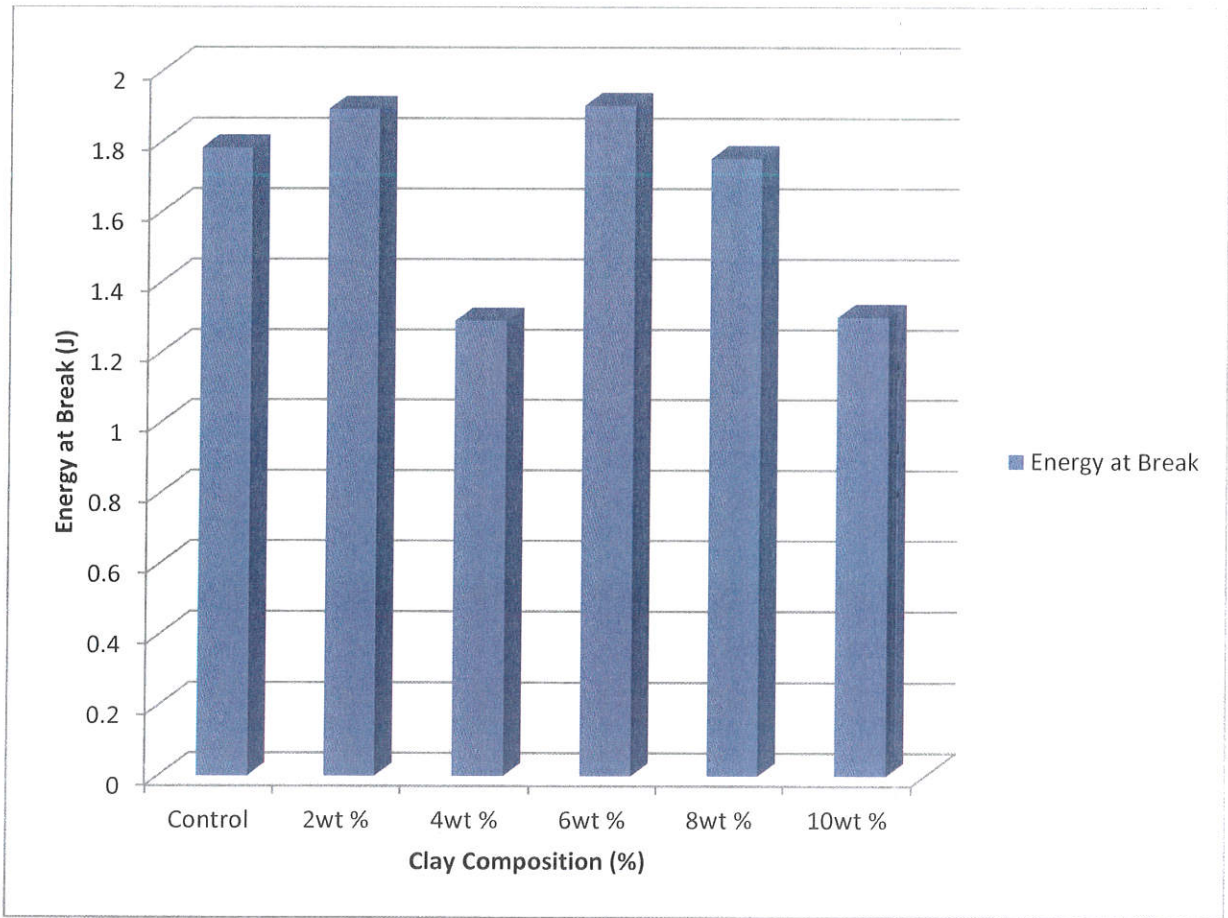
**Figure 4.2: Profile of UTS of neat and reinforced Composites**

Figure 4.2 reveals the ultimate tensile strength of the neat and clay particles reinforced composites. From Figure 4.2, the neat composite has the highest UTS (18.78 MPa); followed by composite reinforced with 4% clay particles with UTS value of 17.08 MPa , next is composite with 2 % clay particles composition with UTS value of 16.21 MPa, while the least was exhibited by composite reinforced with 8 % clay particles, as shown in Table 4.1.



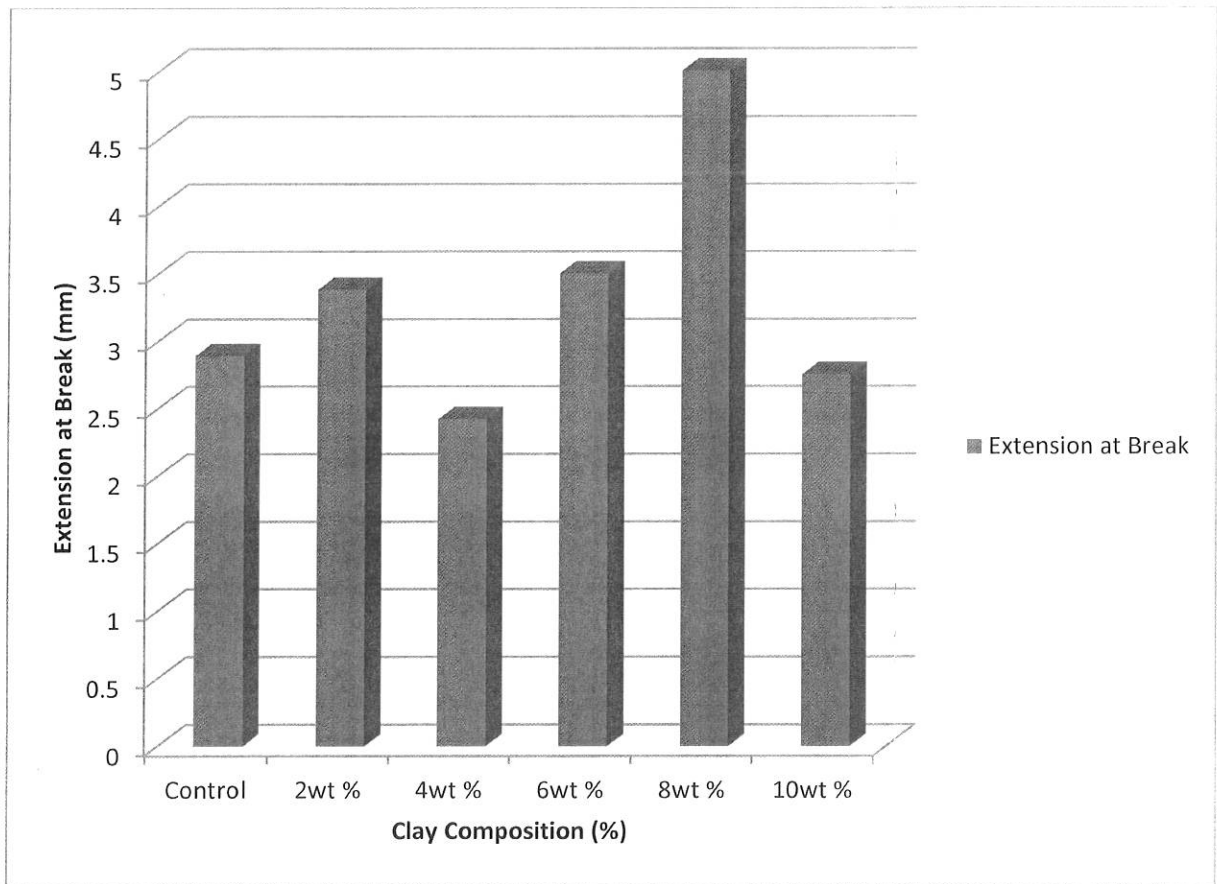
**Figure 4.3: Profile of Young's modulus of neat and reinforced composites**

Figure 4.3 shows the young's moduli of neat and clay particles reinforced composites. It was observed that the neat has the highest modulus (590.5 MPa), and this follow the same trend of the UTS; as shown in Table 4.1. The low values observed for the clay reinforced particles as compared with neat, may be attributed to various degree of immiscibility exhibited by the composites.



**Figure 4.4 Profile of Energy at Break of neat and reinforced composites**

Figure 4.4 shows the energy at break of neat and clay particles reinforced composites. It was observed that the addition of clay particles increased energy at break, composite of 6% clay particles content has the highest value of 1.9 J, followed by composite reinforced with 2% clay particles with value of 1.89J, next is neat with value of 1.78J, while the least was exhibited by composite reinforced with 4 engineering% clay particles (1.29 J), as shown in Table 4.1.



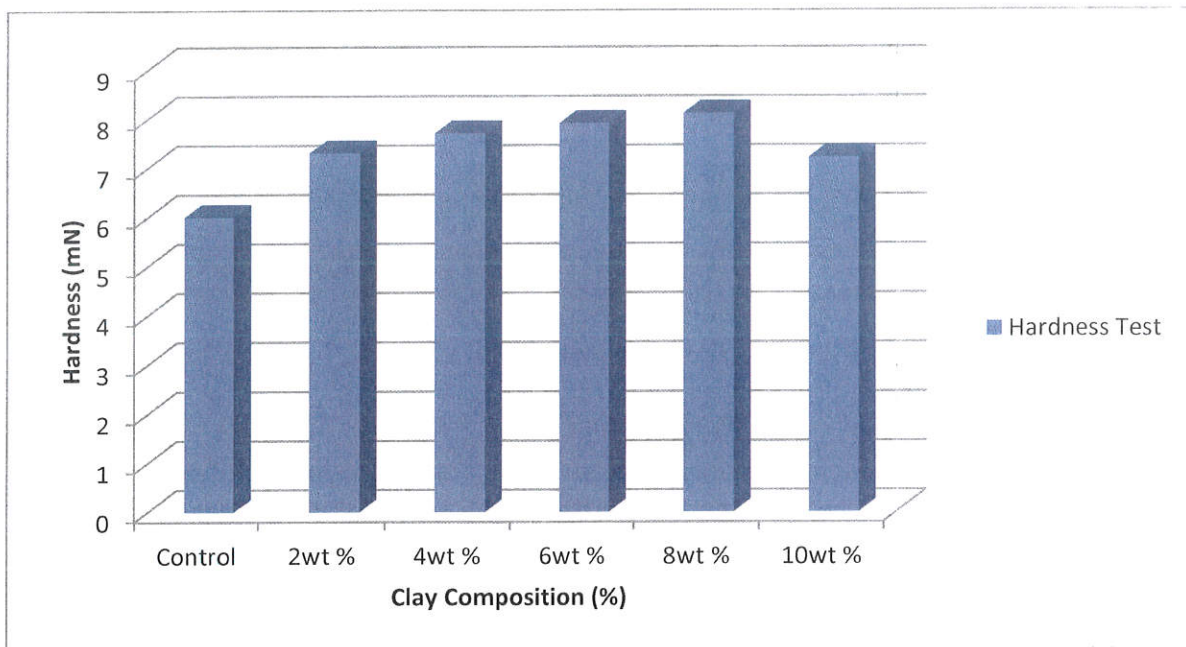
**Figure 4.5 Profile of Extension at Break of neat and reinforced composites**

Figure 4.5 reveals the extension at break of the neat and clay particles reinforced composites. From Figure 4.5, the 8% clay particle composite has the highest extension at break (5.00 mm); followed by composite reinforced with 6% clay particles with extension at break value of 3.50 mm, next is composite with 2 % clay particles composition with extension at break value of 3.38 mm, while the least was exhibited by composite reinforced with 4 % clay particles (2.42 mm), as shown in Table 4.1.



**Table 4.2: Hardness of neat and Clay reinforced Composites**

S/N	Material	Hardness Test (mN)
1	100% Epoxy 0% Clay	6.0
2	98% Epoxy 2% Clay	7.3
3	96% Epoxy 4% Clay	7.7
4	94% Epoxy 6% Clay	7.9
5	92% Epoxy 8% Clay	8.1
6	90% Epoxy 10% Clay	7.2



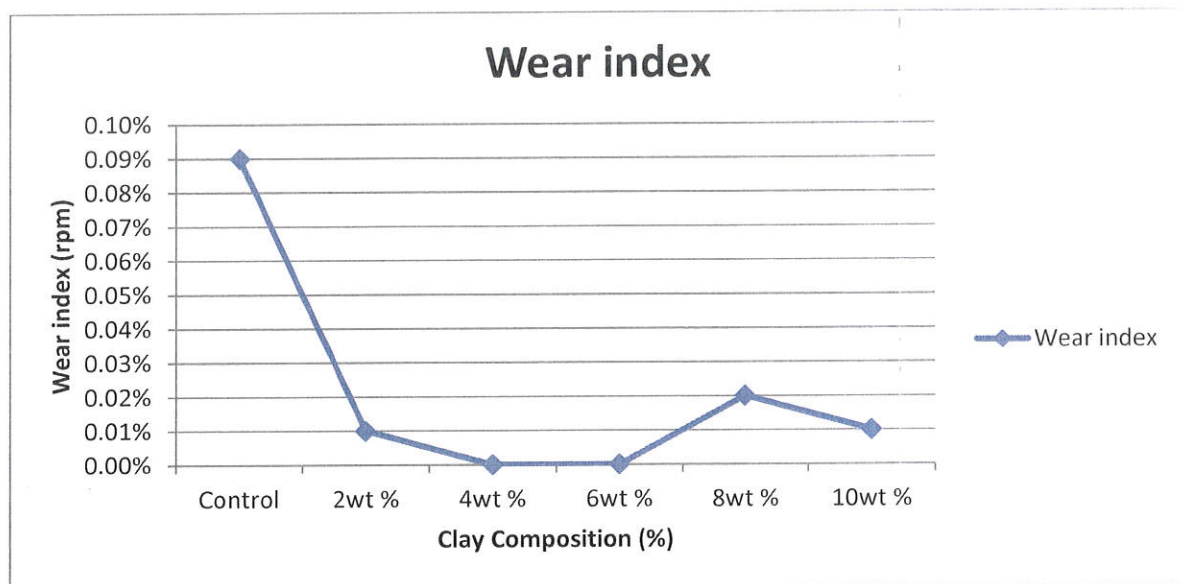
**Figure 4.6 Profile of Hardness of neat and reinforced composites**

Figure 4.6 shows the hardness of neat and clay particles reinforced composites. Generally, it was observed that the addition of clay particles increases the hardness of the epoxy-clay composite as compared with the neat. It was also observed as the clay content of the composites increases, the

hardness also increases up to 8% wt content, before a decline was observed which is even higher than the neat, as shown in Table 4.2.

**Table 4.3: Percentage of wear of neat and Clay reinforced Composites**

S/N	Material	Wear Behaviour (rpm)
1	100% Epoxy 0% Clay	0.09%
2	98% Epoxy 2% Clay	0.01%
3	96% Epoxy 4% Clay	0.00%
4	94% Epoxy 6% Clay	0.00%
5	92% Epoxy 8% Clay	0.02%
6	90% Epoxy 10% Clay	0.01%



**Figure 4.7 Profile of Wear Index of neat and reinforced composites**

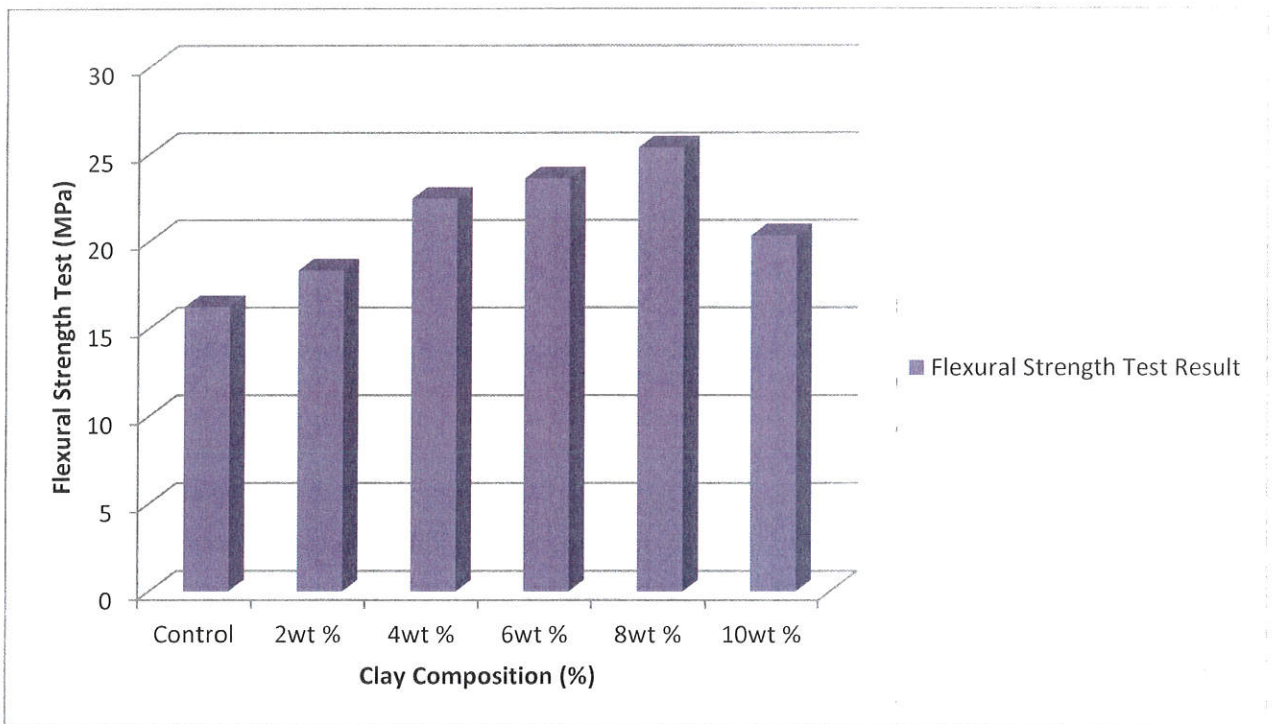
Figure 4.7 reveals the wear behaviour of the neat and clay particles reinforced composites. It was observed that the neat has the highest wear behaviour, as shown in Table 4.3. This implies that



the addition of clay particles reduces the wear behaviour of the composites, even it was observed that 4 and 6 weight percent clay particle composites exhibited zero tolerance to wear.

**Table 4.4: Flexural Strength of neat and Clay reinforced Composites**

S/N	Material	Flexural Strength (MPa)
1	100% Epoxy 0% Clay	16.19
2	98% Epoxy 2% Clay	18.28
3	96% Epoxy 4% Clay	22.39
4	94% Epoxy 6% Clay	23.55
5	92% Epoxy 8% Clay	25.32
6	90% Epoxy 10% Clay	20.28

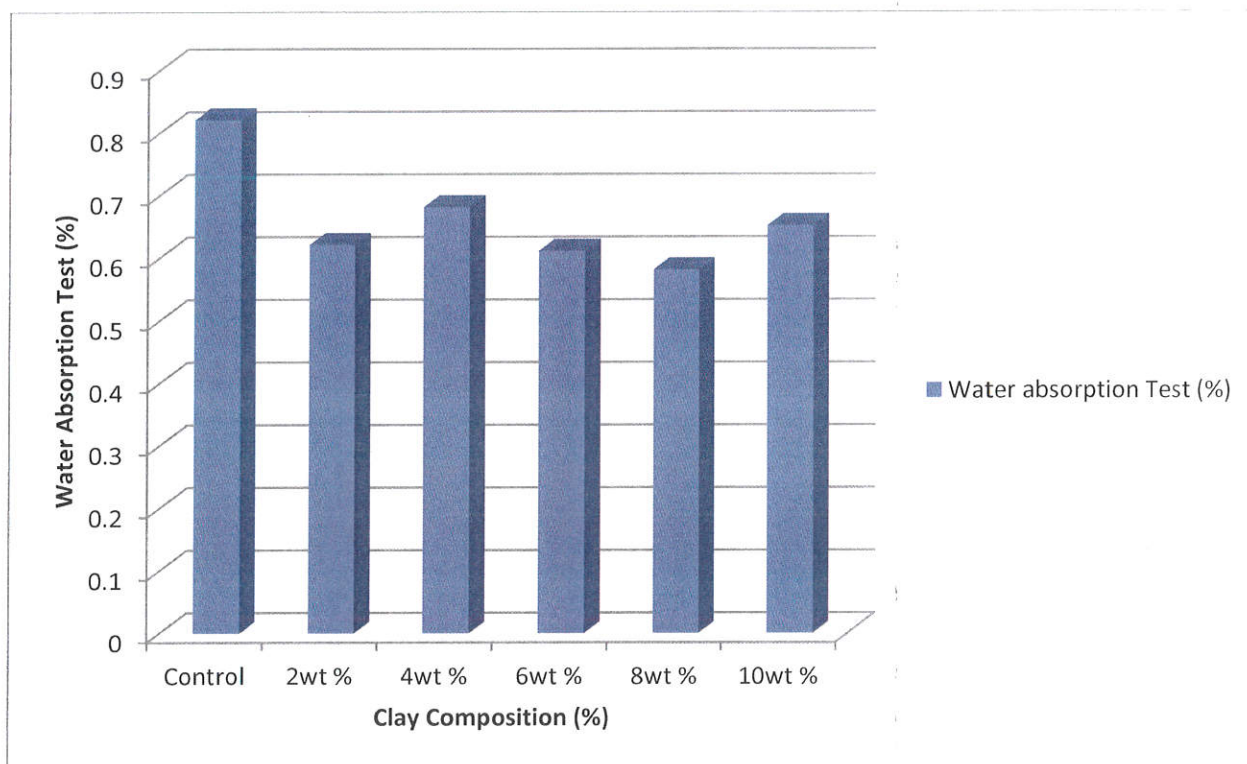


**Figure 4.8 Profile of Flexural Strength of neat and reinforced composites**

Figure 4.8 reveals the flexural strength of the neat and clay particles reinforced composites. Generally, it was observed that the addition of clay particles increases the flexural strength of the epoxy-clay composite as compared with the neat. It was also observed as the clay content of the composites increases, the flexural strength also increases up to 8% wt content, before a decline was observed. This shows that the maximum clay content that can improved the flexural strength of the composites is 8%, anything after it, there will be a decline of the flexural strength of the resulting composites, as shown in Table 4.4.

**Table 4.5: Water Absorption Test of neat and Clay reinforced Composites**

S/N	Material	Water Absorption (%)
1	100% Epoxy 0% Clay	0.82
2	98% Epoxy 2% Clay	0.62
3	96% Epoxy 4% Clay	0.68
4	94% Epoxy 6% Clay	0.61
5	92% Epoxy 8% Clay	0.58
6	90% Epoxy 10% Clay	0.65



**Figure 4.9 Profile of Water Absorption Test of neat and reinforced composites**

Figure 4.9 reveals the water absorption test of the neat and clay particles reinforced composites. From Figure 4.9, it was observed that the addition of clay particles to the composites decreases the percentage of water absorption of the composites, neat composite has the highest value of (0.82%); followed by composite with 4% clay particles with value of 0.68% , next is composite with 10% clay particles composition with value of 0.65%, while the least was exhibited by composite reinforced with 8 % clay particles (0.58 %), as shown in Table 4.5.

## CHAPTER FIVE

### 5.0 CONCLUSION AND RECOMMENDATIONS

#### 5.1 Conclusion

It was observed from the study that porous structure of clay, poor dispersion and poor interfacial adhesion (bonding) between the clay and the epoxy matrix caused decrease in tensile strength and young modulus as percentage of clay fillers increased. Due to these findings clay could be utilized for composite production in areas subjected to tension and abrasion as an extender filler to reduce cost. Clay is not a good reinforcement material.

Since the addition of clay particles improved the flexural strength, wear resistance, and hardness of the composites, hence epoxy-clay composite could be used in engineering applications such as production of electronic casing, handle of kitchen utensil, sole of shoes, insulators, tables and slabs.

## **5.2 Recommendations**

1. The government should assist engineering research in universities through the provision of some characterization equipment like Vickers Hardness Machine, Instron Universal Testing Machine and Wear Abrasive Machine.
2. The use of high volume fractions of clay for composite production should be investigated.
3. Coating of the surface of clay with coupling agents should be done to improve interfacial adhesion.
5. Mechanized stirring and dispersants should be used in the mixing of clay and epoxy to improve dispersion and prevent agglomeration of fillers.
6. In order to increase the hardness of the composite harder reinforcing materials like snail shell, Iron fillings, carbon fibers, periwinkle shell and palm kernel shell should be used together with clay.
7. The rheological properties of epoxy should be studied.

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## APPENDIX I

### Pre-treatment on Isan-Ekiti Clay



Plate 1: Washing of Clay

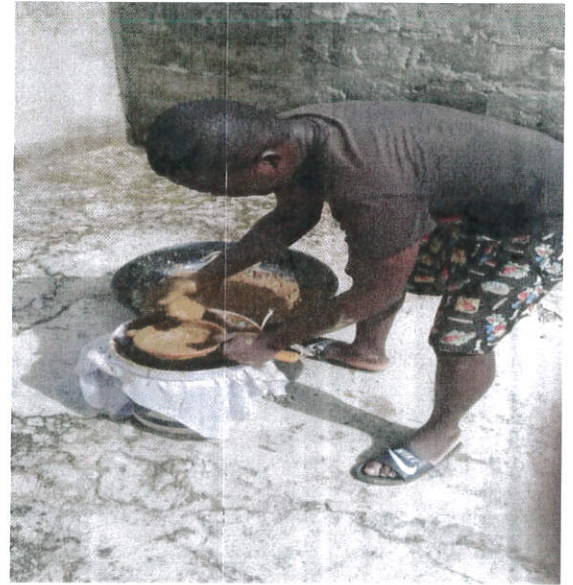


Plate 2: Washing & Sieving of Clay



Plate 3: Drying of Clay

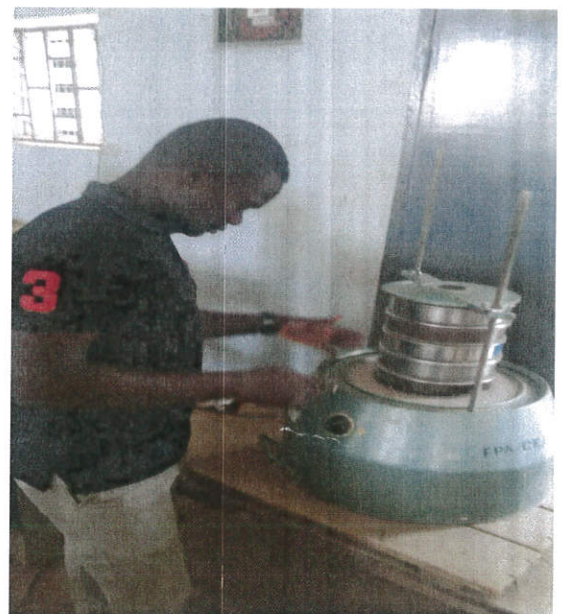


Plate 4: After grinding, Sieving

APPENDIX II

Composite Fabrication Procedures

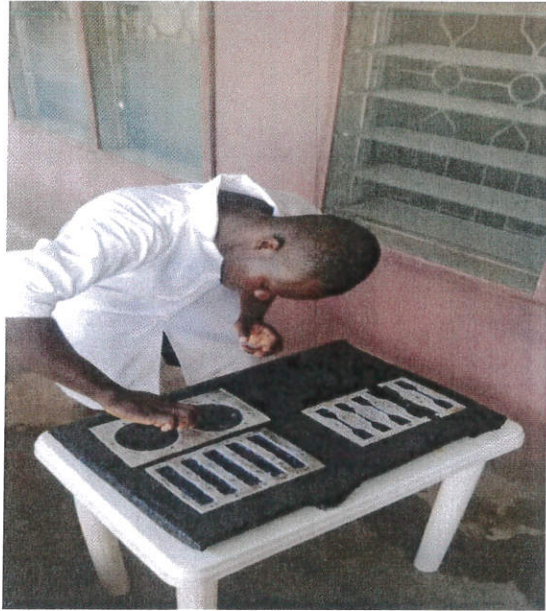


Plate 5: Preparing Mold for easy release

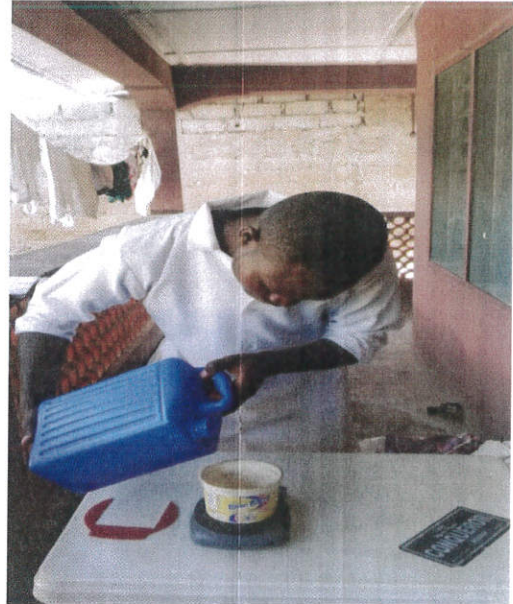


Plate 5: Measuring of the Epoxy

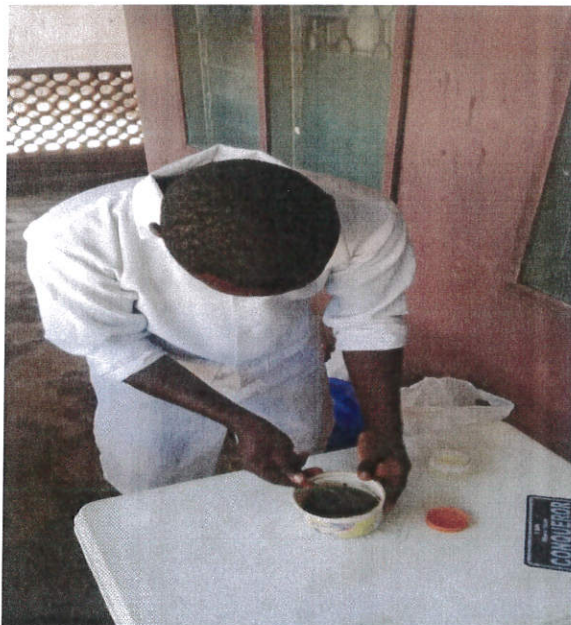


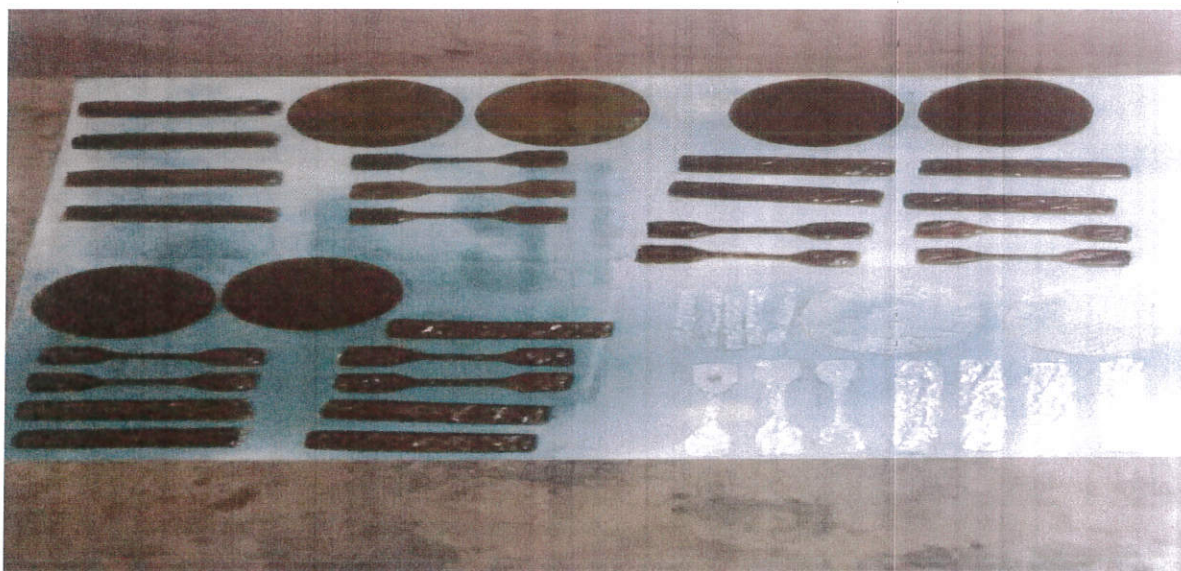
Plate 6: Mixing of the Composites



Plate 7: Pouring of Composites inside mold



**Plate 8: Curing of the composites**



**Plate 9: Hardness, Tensile, Flexural, Wear test specimen**

APPENDIX III

Machine used for testing of composites



Plate 10: Instron Machine- Series 3369



Plate 11: Vickers Hardness Machine

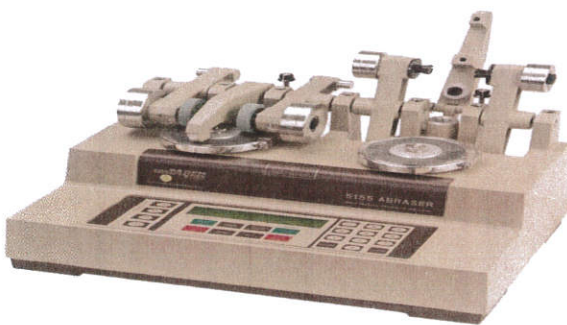


Plate 12: Taber Abraser Tester Machine

