CHARACTERIZATION OF THE MECHANICAL PROPERTIES OF UNSATURATED POLYESTER REINFORCED WITH VARYING FRACTION OF PARTICULATE GROUNDNUT SHELL

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CERTIFICATION

I certify that this work was carried out by AKEJU OLUWABEMI OLUWASEUN with Registration number MME/11/0422 under my supervision in the Department of Materials and Metallurgical Engineering, Federal University Oye-Ekiti. It is an original work for Bachelor degree (Engineering).

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DEDICATION

I dedicate this project to God Almighty and my loving parents.

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My sincere gratitude goes to the Almighty God for giving me life, knowledge and resources to complete this study.

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ABBREVIATIONS

AFM---------- Atomic Force microscope

ANOM---------- Analysis of Means

ANOVA---------- Analysis of Variance

ASTM---------- American Society for Testing and Materials

CONC---------- Concentration

DSC---------- Differntial Scanning Calorimetry

FTIR---------- Fourier Transform Infrared Spectroscopy

GFRP---------- Groundnut Fibre Reinforced polymer

GSF---------- Groundnut Shell Filler

RPM---------- Revolution per Minute

SEM---------- Scanning Electron Microscope

SIC---------- Silicon carbide

XRF----------X-ray Fluorescence

TGA---------- Thermogravimetric Analysis

XRD---------- X-ray Diffraction

ABSTRACT

The objective of this study is to produce and determine the mechanical properties as well as water absorption characteristics of unsaturated polyester-groundnut shell particle composite. The aim of the study with varying weight of groundnut shell particle is to find the composite with optimum mechanical properties. Groundnut shell of undersize particle of 300 microns was introduced at different percentages into the unsaturated polyester resin. Composites were produced at 2%, 4%, 6%, 8% and 10% volume fraction of groundnut shell particle fillers and the polyester was cast neat at 0% groundnut shell particle filler which served as the control.

Mechanical properties of the composites such as tensile properties (tensile stress, tensile strain, Young's modulus, tensile strength and percentage elongation at fracture), flexural strength and hardness were experimentally determined in the engineering laboratories using Double cell Universal Instron machine (Instron 3369) at a cross head speed of 5mm/min and a load cell capacity of 50KN, Rockwell hardness testing machine (HRA 60) and Taber abrasive machine at 150 RPM. Water absorption test was also carried out on the samples.

The tensile test specimen preparation and testing procedures were conducted in accordance with the ASTM.

Results of the mechanical property tests showed increase in hardness, flexural strength and Young's modulus. The tensile properties showed: that at 10%GSF the highest tensile strength of 783MPa compared to the control of 10.84155 MPa, the cast composite with 10wt% GSF had the best tensile modulus of 783MPa. Increasing the volume fraction of groundnut shell particle filler from 0% to 2%, 4%, 6% and 8% led to decrease in tensile strength from 10.84155MPa to 10.29971MPa of the reinforced composites. The wear property of the composite was enhanced at 4wt% and had the best wear index of 1.04 compared to the control of 0.251.

The polymer composites showed irregular pattern in their water absorption property with 2wt%GSF having the highest water absorption property and 4wt% having the poorest water absorption property.

It was deduced from the study that, the groundnut shell reinforced polyester composite will perform in engineering areas of application that require mild strength such as in window frames of automobiles, casing of electrical appliances, and floors of airplanes.

Also, Surface coating of groundnut shell particle filler could be used to improve its adhesion to the polyester matrix in order to enhance the mechanical properties of the composites for other engineering applications.

CHAPTER ONE

INTRODUCTION

1.1 Background to Study

Due to increased environmental consciousness throughout the world the application of natural fibers has drawn much attention in different engineering fields. The use of natural fibers as reinforcing materials in thermoplastics and thermoset matrix composites provides optimistic environmental profits with regard to ultimate disposability and better use of raw materials. The natural fibers are now believed to be as an option to synthetic fibers such as glass fiber, carbon fiber, etc. Owing to certain factors such as easy availability, light weight, high toughness, non-corrosive nature, low density, low cost, good thermal properties, reduced tool wear, less dermal and respiratory irritation, less abrasion to processing equipment and renewability, natural fibers are preferred over synthetic fibers and hence find wide applications in different industries for constructive parts such as in Aerospace and Automobiles.

Many authors have reviewed the latest developments in the application of natural fibres. The widespread investigations on the preparation and properties of thermoset and thermoplastic composites with the application of natural fibers such as bagasse, bamboo, pineapple, rice husk and groundnut shell have also been carried out. Natural fibres are used for variety of appliances such as packaging, low-cost housing and structures and the use of agricultural crop residues could progress rural agriculture based economy.

Composite materials are engineered materials produced from combination of two or more materials having compositional variations and depicting properties different from those of the individual materials of the composite (Rajput, 2013). Examples of composite materials are

Vehicle tyres, Cement concrete and plywood. According to Rajput (2013), the strength, heat resistance or stiffness of composite material is generally better than any of the individual components. Some of the limitations of composite materials are high cost of raw materials and fabrication, difficulties in reusing and disposing composites and difficulties in combining the reinforcement and matrix for formation of composite materials.

1.2 Statement of Problem

Groundnut shell over the years in Nigeria has been improperly disposed. The improper disposal of these shells could result in drainage blockage, dirty environment and subsequently health hazards. In an attempt to improve the conversion of wastes to wealth so as to reduce the unemployment rate of the nation, and to create a healthier and cleaner environment by reducing environmental pollution, wastes such as groundnut shells are used as natural particulates reinforcement in composite.

1.3 Aim

The aim of putting up this work is to characterize the mechanical properties of unsaturated polyester resin reinforced with varying weight fractions of particulate groundnut shell.

1.4 Objectives of the study

1. To assess the effect of filler loading (groundnut shell) on mechanical properties of unsaturated polyester.
2. To compare the individual properties of the composite produced with their economic value.
3. To develop a physical model from the experimental data

1.4 Justification of the study

This study is applicable in understanding the potentials of groundnut shell as reinforcement in unsaturated polyester resin matrix composite. The study is useful to scientists, researchers and engineers because it will help to suggest ways of improving the mechanical properties of the unsaturated polyester-groundnut shell composite. Composite production can offer employment opportunity to unemployed youths in the country due to low energy and machinery requirements for production. Proper understanding of the microstructure and mechanical 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 Significance of study

1. It contributes to the body of knowledge
2. The material Engineer has an additional option of using polyester and groundnut shell polymeric composites to replace some of the already existing materials used in the automobile sectors of the industry.

1.6 Scope and Limitation of the Study

Experimental approaches used in this study involve composite production and determination of the mechanical properties of the composite material. The composites were produced at 2%, 4%, 6%, 8% and 10% weight fraction of particulate groundnut shell fillers at the particle undersize of 300μm. The groundnut shell were washed, dried and milled before they were used to reinforce unsaturated polyester resin. The production of the composite was done through casting. Afterwards, the mechanical properties of the composite were determined.

CHAPTER TWO

2 LITERATURE REVIEW

2.1 Overview of Composite

Composite materials are man-made materials which are manufactured with an aim of replacing the conventional materials by overcoming their disadvantages. A composite material has two main constituents namely, matrix and reinforcements. The reinforcements or fibers are the main load carrying elements and it provides strength and rigidity to composite whereas, matrix gives the shape to composite, maintains fiber alignment and protects them against the environmental and possible damage.

The use of composites started many centuries ago. In the Christian Bible, the Book of Exodus 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 fibres reinforced resins. Boats and aircrafts were built out of these glass composites commonly called fibre glass.

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

2.2 Classification of composites

Composite materials are broadly classified in to three types based on the matrices namely, metal matrix composites (MMC), polymer matrix composites (PMC) and ceramic matrix composites (CMC).

2.2.1 Classification of composite based on the type of material used for the matrix

1. Metal matrix composite (MMCs)
2. Polymer matrix composite (PMCs)
3. Ceramic matrix composite (CMCs)

2.2.1.1 Metal matrix composite

Metal matrix composites are composites that have ductile metal as its matrix. Metals that are reinforced with hard ceramic particles or fibres have improved strength and stiffness, improved creep and fatigue resistance and increased hardness, wear and abrasion resistance (**Harris**, 1999). Also, these metals may be utilized at higher service temperatures than unreinforced metals or competing reinforced plastics (**Callister**, 2007). According to Harris, 1999, these properties offer potential for development in a range of pump and engine applications, including compressor bodies, vanes and rotors, piston sleeves and inserts, connecting rods, and so forth. These metal matrix composites have some advantages over the polymer-matrix composites. Some of the advantages include higher operating temperatures, non-flammability, and greater resistance to degradation by organic fluids. Metal-matrix composites are much more expensive than PMCs and this makes their (MMC) use is somehow restricted (Callister, 2007).

In metal matrix composite, super alloys, as well as alloys of aluminum, magnesium, titanium, and copper, are employed as matrix materials while the reinforcement may be in the form of particulates, both continuous and discontinuous fibres, and whiskers; concentrations normally range between 10 and 60 volume% (Callister, 2007). Some of the materials of the continuous fibre are carbon, silicon carbide, boron, aluminum oxide, and the refractory metals. On the other hand,

discontinuous reinforcements consist primarily of silicon carbide whiskers, chopped fibres of aluminum oxide and carbon, and particulates of silicon carbide and aluminum oxide.

According to Callister, 2007, some matrix–reinforcement combinations are highly reactive at elevated temperatures. Consequently, high-temperature processing or subjecting the MMC to elevated temperatures during service may cause composite degradation. The common way of resolving this is by applying a protective coating to the reinforcement or by modifying the matrix alloy composition.

2.2.1.2 Polymer matrix composite

Polymer-matrix composites (PMCs) consist of a polymer resin as the matrix with fibre as reinforcement. Polymers make ideal materials as they can be processed easily, possess lightweight, and desirable mechanical properties (Callister, 2007). There are two main kinds of polymers and they are thermosets and thermoplastics. Thermosets have qualities such as a well-bonded three-dimensional molecular structure after curing. They decompose instead of melting on heating. Merely changing the basic composition of the resin is enough to alter the conditions suitably for curing and determine its other characteristics. They can be retained in a partially cured condition too over prolonged periods of time, rendering Thermosets very flexible. Thus, they are most suited as matrix bases for advanced conditions fibre reinforced composites. Thermosets find wide ranging applications in the chopped fibre composites form particularly when a premixed or moulding compound with fibres of specific quality and aspect ratio happens to be starting material as in epoxy, polymer and phenolic polyamide resins.

Examples of polymer-matrix composites are:

a) Glass fibre reinforced polymer (GFRP) composites: optic fibre

Fibreglass reinforce polymer composite is simply a composite consisting of glass fibres, either continuous or discontinuous, contained within a polymer matrix; this type of composite is produced in the largest quantities (Callister, 2007).

According to Callister, 2007, glass fibre is popular as reinforcement because:

i. It is easily drawn into high-strength fibres from the molten state.

ii. It is readily available and may be fabricated into a glass-reinforced plastic economically using a wide variety of composite-manufacturing techniques.

iii. As a fibre it is relatively strong, and when embedded in a plastic matrix, it produces a composite having a very high specific strength.

iv. When coupled with the various plastics, it possesses a chemical inertness that renders the composite useful in a variety of corrosive environments.

The limitation of (GFRP) is that they are not stiff and they do not display the rigidity that is necessary for some applications (e.g., as structural members for airplanes and bridges). Most fibreglass materials are limited to service temperatures (180^0C – 200^0C) below at higher temperatures; most polymers begin to flow or to deteriorate. Service temperatures may be extended to approximately by using high-purity fused silica for the fibres and high-temperature polymers such as the polyimide resins (Callister, 2007).

According to Callister, 2007, the applications of fibreglass are in automotive and marine bodies, plastic pipes, storage containers, and industrial floorings.

b) **Carbon fibre-reinforced polymer (CFRP) composites**

Carbon is a high-performance fibre material that is the most commonly used reinforcement in advanced (i.e., non fibreglass) polymer-matrix composites.

The reasons for this are as follows:

i. Carbon fibres have the highest specific modulus and specific strength of all reinforcing fibre materials.

ii. They retain their high tensile modulus and high strength at elevated temperatures; high temperature oxidation, however, may be a problem.

iii. At room temperature, carbon fibres are not affected by moisture or a wide variety of solvents, acids, and bases.

iv. These fibres exhibit a diversity of physical and mechanical characteristics, allowing composites incorporating these fibres to have specific engineered properties.

v. Fibre and composite manufacturing processes have been developed that are relatively inexpensive and cost effective.

Carbon-reinforced polymer composites are now being used extensively in sports and recreational equipment (fishing rods, golf clubs), filament-wound rocket motor cases, pressure vessels, and aircraft structural components—both military and commercial, fixed wing and helicopters (e.g., as wing, body, stabilizer, and rudder components) (Callister, 2007)

c) Aramid fibre-reinforced polymer composites.

Aramid fibres are high-strength, high-modulus materials that were introduced in the early 1970s. According to Callister, 2007, aramid fibres are desirable for their outstanding strength to weight ratios, which are superior to metals.

The aramid fibres are most often used in composites having polymer matrices; common matrix materials are the epoxies and polyesters. Since the fibres are relatively flexible and somewhat ductile, they may be processed by most common textile operations. Typical applications of these aramid composites are in ballistic products (bulletproof vests and armor), sporting goods, tires, ropes, missile cases, pressure vessels, and as a replacement for asbestos in automotive brake and clutch linings, and gaskets.

2.2.1.3 Ceramic matrix composites (CMCs)

CMCs have ceramics as their matrix. Ceramics can be described as solid materials which exhibit very strong ionic bonding in general and in few cases covalent bonding. High melting points, good corrosion resistance, stability at elevated temperatures and high compressive strength, render ceramic-based matrix materials a favourite for applications requiring a structural material that does not give way at temperatures above 1500^0C. Naturally, ceramic matrices are the obvious choice for high temperature applications.

Ceramic-matrix composites may be fabricated using hot pressing, hot isostatic pressing, and liquid phase sintering techniques. Relative to applications, SiC whisker-reinforced aluminas are being utilized as cutting tool inserts for machining hard metal alloys; tool lives for these materials are greater than for cemented carbides (Callister, 2007).

2.2.2 Based on the reinforcement

2.2.2.1 Particulate reinforced composites

A particulate reinforced composite is a composite whose reinforcement is a particle with all the dimensions roughly equal. According to Richardson (1987), particulate fillers are employed to improve high temperature performance, reduce friction, increase wear resistance and to reduce shrinkage. The particles will also share the load with the matrix, but to a lesser extent than a fibre. A particulate reinforcement will therefore improve stiffness but will not generally strengthen.

2.2.2.2 Fibre reinforced composites

A fibre reinforced composite is a composite that contains reinforcement having lengths higher than cross sectional dimension. Fibrous reinforcement represents physical rather than a chemical means of changing a material to suit various engineering applications (Warner *et al.*, 1995). In fibre reinforced composite, the reinforcing fibre in a single layer composite may be short or long based on its overall dimensions. Composites with long fibres are called continuous fibre reinforcement and composite in which short or staple fibres are embedded in the matrix are called discontinuous fibre reinforcement (short fibre composites). In continuous fibre composites, fibres are oriented in one direction to produce enhanced strength properties. In short fibre composites, the length of short fibre is neither too high to allow individual fibres to entangle with each other nor too small for the fibres to loss their fibrous nature. The reinforcement is uniform in the case of composites containing well dispersed short fibres. There is a clear distinction between the behavior of short and long fibre composites.

2.2.2.3 Hybrid composites

Hybrid composites are composites incorporated with two or more different types of fillers especially fibres in a single matrix. Hybridisation is commonly used for improving the properties and for lowering the cost of conventional composites. According to Mallick (1988), There are different types of hybrid composites classified according to the way in which the component materials are incorporated. Hybrids are designated as

i. sandwich type
ii. interplay
iii. intraply and
iv. intimately mixed

In sandwich hybrids, one material is sandwiched between layers of another, whereas in interply, alternate layers of two or more materials are stacked in regular manner. Rows of two or more constituents are arranged in a regular or random manner in intraply hybrids while in intimately mixed type, these constituents are mixed as much as possible so that no concentration of either type is present in the composite material.

2.2.2.4 Laminates

Laminar composites are composites that are made from the two dimensional sheets or panels that have a preferred high strength (Rajput, 2013). A laminate is fabricated by stacking a number of laminae in the thickness direction. Generally three layers are arranged alternatively for better bonding between reinforcement and the polymer matrix, for example plywood and paper. These laminates can have unidirectional or bi-directional orientation of the fibre reinforcement according to the end use of the

composite. A hybrid laminate can also be fabricated by the use of different constituent materials or of the same material with different reinforcing pattern. In most of the applications of laminated composite, man-made fibres are used due to their good combination of physical, mechanical and thermal behavior.

2.3 Polymer Matrix Composite

In the recent years, there has been steady increase in the use of ceramic particle – reinforced matrix composite because of their advantages such as isotropic properties and the possibility of secondary processing facilitating fabrication of secondary components.

Polymer matrix composite is the material consisting of a polymer (resin) matrix with a fibrous reinforcing dispersed phase which can provide advantageous properties such as abrasion resistance, creep resistance, dimensional stability, exceptionally good stiffness-to-weight and strength to weight ratios over base polymer material. Polymer matrix Composites are very popular due to their low cost and simple fabrication methods.

2.3.1 Application of Polymer Matrix Composite

These materials have their wide range of disciplines. About 30% of all polymers matrix composite produced each year are used in the civil engineering and building industries. Polymer matrix offer many advantages over conventional materials including lightness, resilience to corrosion and ease of processing. They can be combined with fibres to form composites which have enhanced properties, enabling them to be used as structural members and units. Polymer composites can be used in many different forms ranging from structural composites in the construction industry to the high technology composites of the aerospace and space satellite industries. The following are some of them:

1. Polymer composites and alloys are used for many aeronautical and automobile applications due to their low weight to strength ratio (Nikhil, 2015).
2. Aluminium matrix composite materials are widely used engineering structures, marine application, automotive bumpers and sporting goods (Devi, 2012).

2.3.2 Limitation of Polymer Matrix Composite

Limitation of polymer based composite may be contradictory due to the fact that a variety of polymer matrices and reinforcement type combinations may exhibit completely different behaviour. But, generally;

1. Polymer matrix composite reinforced with groundnut shell, periwinkle shell in the form of particles, fibres, whiskers has a low working temperature.
2. High coefficient of thermal expansion- dimensional instability.
3. Sensitive to radiation and moisture.

2.3.3 Past Researches on Composites

Shehu *et al.*, (2013) carried out the research work on the effect of particle size on the properties of polyester and palm kernel shell particulate composites. The aim of the study was to develop polyester/ palm kernel shell particulate composites and to investigate the effect of particle size on the properties of the composites. The palm kernel shell particles were varied thus; 0, 10, 20, 30 and 40wt% at three different particle sizes; 75μm, 150μm and 300μm. Cobalt accelerator and Methyl-ethyl Ketone catalyst were utilized to initiate polymerization reaction and thus speed up the reaction. The effect of palm kernel shell particles and particle size on mechanical and physical properties of polyester was studied. The results showed a better interaction of polyester and palm kernel shell particles at 300μm sieve size with density, water

absorption, ultimate tensile strength and impact energy increasing upon increase in percent palm kernel shell particles with only hardness decreasing upon increase in percent palm kernel shell.

Sabo and Musa (2015) studied the use of non-conventional natural cellulosic fibres as reinforcement for unsaturated polyester composites. This research investigates the use of three natural cellulosic plant fibres in the production of fibre reinforced composite materials. These fibres included Dum palm plant, Luffa gourd and Baobab. Different parts of plants were retted in water for seven days to enable fibre extraction. The extraction was carried out with the use of a pointed metal object. The fibres obtained were subsequently dried and then treated with 5% and 20% concentrations of sodium hydroxide at 800C. Breaking load and extension of the bundle of fibres was determined using Instron universal testing machine. Morphology of the fibres was also studied using Scanning Electron Microscope (SEM). Composites were fabricated with the treated and the untreated fibres using unsaturated polyester resin. The properties of the composites; Tensile strength, Impact strength, Bending strength and Hardness were determined. Other properties tested include morphology by SEM and Atomic Force Microscope (AFM), Water absorption, Density, X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) and chemical resistance of the composites. Thermal property by Differential Scanning Calorimetry (DSC) and Thermogravimetric Analyser (TGA) were also determined. The experiment revealed that Dum palm fibres have the highest strength followed by Baobab and then Luffa gourd. The SEM micrographs of the treated fibres suggest that cellulosis, hemicellulosis, pectins and lignin have been removed from the surfaces hence a clean surface is revealed. On the assessment of tensile strength of both untreated and treated fibres, Luffa gourd and Dum palm fibre composites have higher tensile strength than Baobab fibre composite.

Impact strength assessment of treated and untreated fibres reveals that Baobab composite has the highest impact strength value followed by Luffa gourd and then Dum palm fibre composite. On the assessment of bending modulus, treatment has improved the property in Baobab and Luffa gourd composites, whereas it reduces in Dum palm composite. Moreover, hardness property assessment shows Luffa gourd and Dum palm composites improved with treatment while in Baobab composite, it reduces. In the chemical resistance assessment, all the composites were found to be resistant to some chemicals and not to others. Water absorption assessment shows no significant effect on all the composites, with low values. The densities of the composites were found to be the same. The FTIR analysis indicates a shift in positions of some peaks after the treatment, this suggest that in the surface of the treated fibre composites there is a change in chemical groups due to alkaline treatment hence a shift in positions as shown in the spectra. Morphological analysis of the composites (by SEM) shows a more uniform distribution of fibres in the matrix of treated fibre composites than the untreated ones. A more uniform distribution of fibres fortreated fibre composites is confirmed by the AFM micrograph compared to untreated fibre composites. Thermal analysis by DSC shows (nearly similar rate of decomposition) for treated and untreated fibre composites. In TGA analysis, (different peaks) were observed for both treated and untreated composites; the different rate of degradation observed between the untreated and treated fibre composites is marginal this may be due to the removal of pectins, lignins and hemicelluloses by the alkali treatment. Lastly, XRD analysis showed untreated samples to be more crystalline than the treated samples; this is because sodium hydroxides have opened the fibres internal structure, leading to better molecular chain re-arrangement. In conclusion, chemical treatment improves the mechanical, physical and thermal properties of the fibre reinforced composites as a result of better cohesion between the reinforcing fibres and the

matrix. Therefore, these natural fibre reinforced composites are recommended for light weight engineering applications.

Abiodun et al., (2014) studied the Property Evaluation of Sea shell Filler Reinforced Unsaturated Polyester Composite. The objective of this study is to determine the mechanical properties as well as water absorption characteristics of unsaturated polyester composite reinforced with varying weight fractions of sea shell with an aim of finding the composite with optimum mechanical properties. Ground sea shell of particle size 250 microns was introduced at different percentages into the unsaturated polyester resin. Mechanical tests which include: tensile, flexural, impact, hardness tests were carried out on the prepared composite samples. Water absorption test was also carried out on the samples. The tensile test specimen preparation and testing procedures were conducted in accordance with the ASTM. The results showed the flexural strength of the composite with 10wt% seashell particulate reinforcement to be largely improved also the hardness and impact properties were greatly improved at10wt% sea shell filler loading. This composite could therefore be considered for applications where flexural properties, impact and hardness properties are of utmost concern.

2.4 Groundnut shell

Groundnut botanically known as Arachis hypogeae belongs to Leguminosae family. It is the fourth largest oilseed produced in world and India is the second largest producer of groundnut after China. In India, groundnut is the largest oilseed in terms of production and accounted for about 7.5 million tons during 2009-10. A complete seed of groundnut is called as pod and outer layer of groundnut is called shell. Groundnut shell chemical composition is compared with some natural fibers. The hemicelluloses content of the fiber is found to be 18.7%, cellulose 35.7%, lignin 30.2% and ash content 5.9%. Lignin is

often called the cementing agent that binds individual fiber cells together. The lignin content of groundnut shell fiber is much greater than that of banana, baggase, rice husk, jute, hemp, kenaf and sisal fibers. The hemicellulose is accountable for substantial amount of moisture absorption. The hemi cellulose content of groundnut shell is less than wood, banana, baggase, rice husk and kenaf fibers. Pre-treated groundnut shell is used in this study to modify the surface properties to ensure interfacial interactions between the particles and the resin.

2.4.1 Past Researches on Groundnut Shell

Rufail *et al.,* 2015 made studied on the effect of cow bone and groundnut shell reinforced in epoxy resin on the mechanical properties and microstructure of the composite. Cow bone and groundnut shell particles from 0-25 wt% with 5 wt% interval. A hybrid of the cow bone and groundnut shell was also prepared. The mechanical properties of the developed composites were investigated. Optical microscopy was used to examine the microstructure of the composites. The results revealed that mechanical properties did not increase uniformly with additions in filler but exhibited maximum properties at specific percentages of filler additions. From the Microscopic evaluation, it was discovered that homogeneity decreases with increase in % filler, this could be due to poor interfacial bonding.

Pragatheeswaran R. and S. Senthil Kumaran (2014) studied the mechanical behaviour of groundnut shell powder/calcium carbonate /vinyl ester composite. In this study the effect of calcium carbonate on the mechanical properties of groundnut shell powder based composite was investigated. To meet this objective, groundnut shell powder and calcium carbonate (reinforced vinyl ester were prepared by hand lay-up process. The effects of calcium carbonate on tensile

and flexural properties of the composites were investigated. The test result shows that increase in calcium carbonate increases the tensile and flexural properties of composites.

Raju *et al.*, 2012, conducted an experimental Study on Optimization of Thermal Properties of Groundnut Shell Particle Reinforced Polymer Composites. The present work aims to elucidate the optimization of thermal properties such as thermal conductivity, linear thermal expansion and specific heat of groundnut shell particles reinforced polymer composite materials. The composite specimens were prepared with different weight percentages of randomly distributed groundnut shell particles in polymer matrix. The experiments were planned as per Taguchi *L9*orthogonal array. The analysis of means (ANOM) was performed to determine the optimal parameter levels and analysis of variance (ANOVA) was employed to identify the level of importance of the parameters on each of the properties. TGA and DSC analyses were also carried out to ascertain the thermal stability of these composites. The results revealed that using groundnut shell particles as reinforcement for polymer matrix could successfully develop beneficial composites and can be used for thermal applications.

CHAPTER THREE

3. EXPERIMENTAL METHODOLOGY

3.1 Mechanical properties

3.1.1 Hardness

Hardness can be defined as the resistance of a material to localized plastic deformation; it indicates wear resistance and resistance to scratching, abrasion and indentation Compressive strength = 20 (Askeland, 1994). Hardness testing can be done with Rockwell, Vickers, durometer and rebound and barcol hardness tester.

3.1.2 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.

3.1.2.1 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 under formed cross sectional area because the cross sectional area of a material may change if the material deforms on stretching.

$$\text{Tensile stress } (\delta) = \frac{Load\ or\ force}{Original\ cross\ sectional\ area} = \frac{P}{A} \qquad (2.1)$$

3.1.2.2 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,

$$\text{Strain} = \frac{\textit{Change in length}}{\textit{Original length}} = \frac{\textit{Change in length}}{\textit{Guage length}}$$

$$= \frac{Lf - Lo}{Lo} = \frac{\Delta L}{Lo} \qquad (2.2)$$

Where Lo = Original length

ΔL = Axial deformation or change in length, Lf = final length

3.1.2.3 Ultimate 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.

$$\text{Ultimate tensile strength} = \frac{\textit{Maximum Tensile Force Applied}}{\textit{Original cross sectional area}} \qquad (2.3)$$

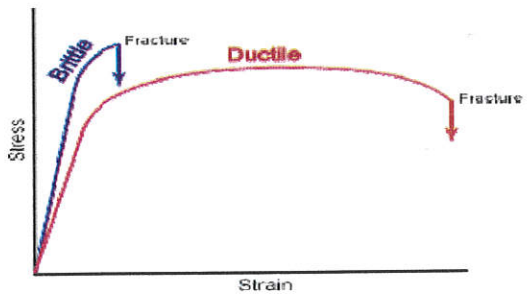

Figure 3.1: Stress – Strain curve

3.1.2.4 Young's Modulus (MPa)

Young's modulus or modulus of elasticity (E) is the slope of the stress - strain curve in the elastic region (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.

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

3.1.2.5 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.

$$\% \text{ Elongation at fracture} = \frac{Lf - Lo}{Lo} \times 100 \text{ or } \frac{Extension}{Original\ length} \times 100 \qquad (2.5)$$

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

3.2 Materials Used

3.2.1 Matrix Material

An Unsaturated Polyester resin (matrix), Methyl Ethyl Ketone Peroxide (catalyst) and Cobalt Octate (accelerator) were used as the matrix material. The resin, catalyst and accelerator were bought from chemical market in Ojota, Lagos.

3.2.2 Filler Material

Groundnut Shell

The filler material used in this study is groundnut shell which was produced from groundnut waste sourced through agricultural means from Otun-Ekiti, Ekiti State, Nigeria. The groundnut shell fillers were collected in large quantity and washed with water to remove impurities like dust, small groundnut particles and fine sand particles. After washing, drying followed by spreading in an open atmosphere under sunlight for 2 weeks in order to remove moisture from it. The groundnut shell was characterized with XRF test and the chemical compositions of the major compounds were determined.

3.3 Methods

3.3.1 Preparation of Composite Mould

Mild steel sheets were cut and formed into different sizes that served as molds for the test samples. The testing techniques for the composite required that five sets of pattern (tensile, compressive, hardness, wear and flexural) should be produced. The patterns were made according to the required dimensions of the test samples. The molds were constructed to within ± Imm to give allowance for machining, and the surfaces were rubbed with wax releaser to ensure easy removal of the composite. Figure 3.I below shows the mold used for composite production.

3.3.2 Composite Fabrication

The composites were produced using facilities at the Federal University of technology Akure, Ondo State. Manual mixing method and hand lay-up technique were used for the composite production. The composites were prepared using 2%, 4%, 6%, 8% and 10% volume fraction of groundnut shell.

An Unsaturated Polyester resin (matrix), Methyl Ethyl Ketone Peroxide (catalyst) and Cobalt Octate (accelerator)were prepared in the ratio of 35.3 parts of polyester resin to 1 part of catalyst and 1 part of accelerator (35.3:1:1). The measured volume of resin, catalyst and accelerator were mixed in a container and stirred at low speed for 2 minutes until the mixture became uniform, tacky and exothermic reaction occurred. The mixture of the matrix material was poured into the mold. Groundnut shell fillers were uniformly and evenly spread in the mold and adequately impregnated into the matrix and allowed to cure. For the cast neat polyester samples, the polyester resin and hardener were poured into the mold and allowed to cure. The curing time was 12 minutes at room temperature. Finally the composite plates were demolded into different mould dimensions for mechanical property tests.

Tables 3.1: Composition of the composites.

Specimen	Composition (wt%)			
	Particulate groundnut shell	**Unsaturated polyester**	**Methyl ethyl ketone peroxide (catalyst)**	**Cobalt Octate(accelerator)**
Control	0	100	1.5	1.5
2wt%	2	98	1.5	1.5
4wt%	4	96	1.5	1.5
6wt%	6	94	1.5	1.5
8wt%	8	92	1.5	1.5
10%	10	90	1.5	1.5

3.4 Mechanical Property Tests

3.4.1 Tensile Testing of Composite Samples

Tensile testing of the samples was done at Centre for Energy Research & Development, Obafemi Awolowo University, Ile-Ife, State of Osun. Double cell Universal Instron machine (Instron 3369) was used in performing the tensile test at a cross head speed of 5mm/min and a load cell capacity of 50KN. The universal machine was automatically operated. The cut tensile test samples were of dimension 60mm long by 13.5mm width and by 6.25mm thickness. Prior to the test the cross sectional area of the samples was calculated. The tensile test specimen preparation and testing procedures were conducted in accordance with ASTM, using dumbbell test piece. Each tensile specimen was positioned in the Instron Universal Tester and then subjected to tensile load, as the specimen stretches the computer generates graph as well as all the desired parameters until the specimen fractures.

3.4.2 Hardness Test

The hardness test was done at Federal University of Technology, Akure, Ondo State. An electric powered Rockwell hardness testing machine (HRA 60) was used to determine the hardness value of the cast neat polyester and the composites. The procedure followed in performing the test were: switching on the machine, selecting the desired load, 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 Rockwell tester. At the end of the indentation a red light showed and instantly reading was directly done from the digital indicator. The value was reported as Rockwell hardness value in HRA.

3.4.3 Flexural Strength Test

The flexural strength test was done at Centre for Energy Research & Development, Obafemi Awolowo University, Ile-Ife, State of Osun. The three point flexing and loading arrangement was used in which fracture occured at the middle. The flexural test was carried according to ASTM standards at a cross-head speed of 5mm/min. This test was conducted at room temperature. The flexural test specimens were of 65 X 49.05 X 6.95 mm.

The Double cell Universal Instron machine (Instron 3369) was used to carry out the three points bending flexural test on the polymeric material composite at different filler content at 0, 2, 4, 6, 8 and 10 wt% of filler content.

3.4.4 Water Absorption Test

The samples were cut in dimension; their initial weights were taken with the aid of an electronic weighing scale. Each of the samples was immersed in a beaker containing water for 168 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.

$$\text{Water Absorption} = \frac{initial\ weight - final\ weight}{initial\ weight} \times 100$$

3.4.5 Friction and Wear Behaviour test

The friction and wear behavior test was done at Federal University of Technology Akure, Ondo State. Taber abrasive machine was used at 150 Rpm. Polyester composite test specimen mounted as a pin in the test holder, composite pins subjected to rotating counter face -steel disk-

under contact pressure. Friction force displayed by means of load cell then used to calculate the wear index for each sample under each test condition.

Rate of wear measured on the principle of weight loss for sliding distance for each sample under each test condition.

$$\text{Wear index} = \frac{initial\ weight - final\ weight}{RPM} \times 1000$$

CHAPTER FOUR

4. RESULTS AND DISCUSSION

4.1 Results

The result of the study shows the mechanical properties and shows the behaviour of the composites and cast neat polyester to different kinds of forces.

Results for Mechanical Properties are shown as Follows:

4.1.1 Tensile Strength

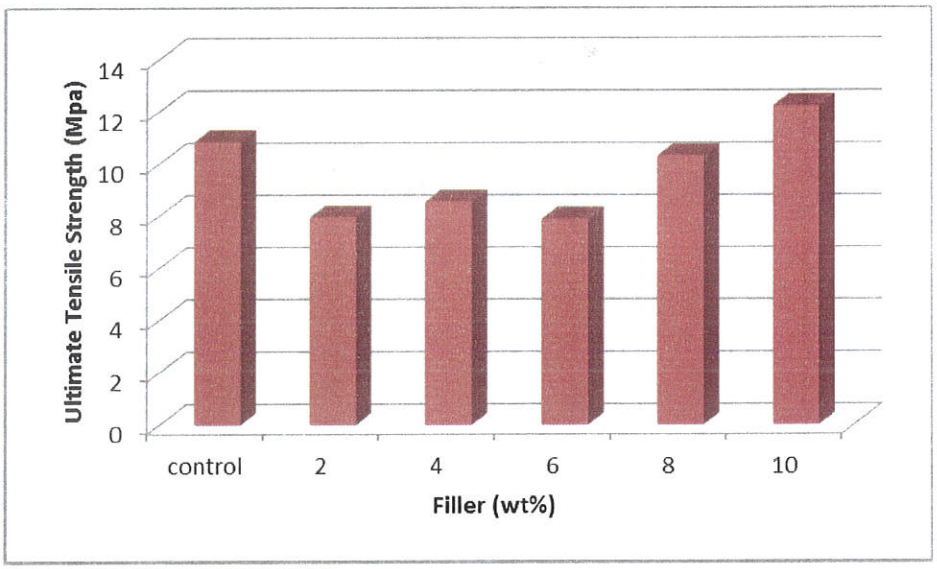

Figure 4.1: Variation of Ultimate Tensile Strength with Reinforcement

Table 4.1: Ultimate Tensile Strength Data

Filler Wt%	Tensile Strength (MPa)
Control	10.84155
2	7.95167
4	8.54971
6	7.8881
8	10.29971
10	12.20553

Figure 4.1 and Table 4.1 shows the relationship between ultimate tensile strength and groundnut shell particulate. It was observed from the graph that the developed composite with 10 wt % reinforcement possessed the best tensile strength than the control with a value of 12.20553 MPa to 10.84155 MPa, however the other reinforcements from 2 – 8 wt% had a lower tensile performance compared to the control sample. With regard to this ultimate tensile strength result, it can be denoted that groundnut shell is a good reinforcement material.

4.1.2 Flexural Strength

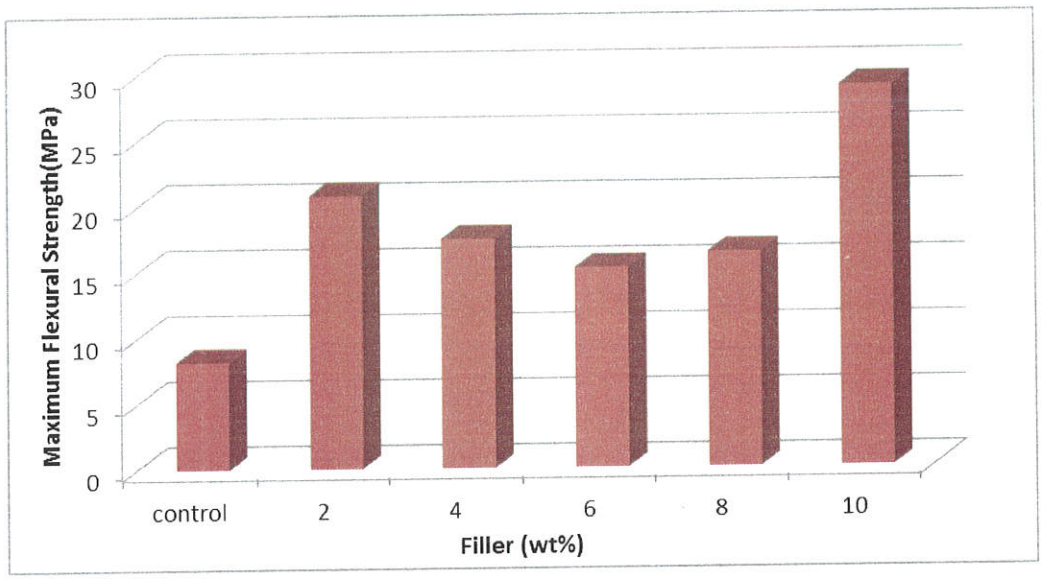

Figure 4.2: Variation of maximum flexural strength to Reinforcement

Table 4.2: Maximum Flexural Strength Data

Filler Wt%	Maximum Flexural Strength (MPa)
Control	8.22583
2	20.8578
4	17.52853
6	15.26563
8	16.40172
10	29.03878

The results from Figure 4.2 and table 4.2 show that the 10 wt % groundnut shell reinforcement gave the best flexural property compared to other samples with a value of 29.03878 MPa.

Although the values of the performance undulated from 2 wt % reinforcement with value 20.8578 MPa reducing to 17.52853 MPa of 4 wt% which performed better compared to the control and other reinforcement. This may have been as a result of an inhomogeneous dispersion of the groundnut shell particulate in the matrix.

4.1.3 Tensile Modulus

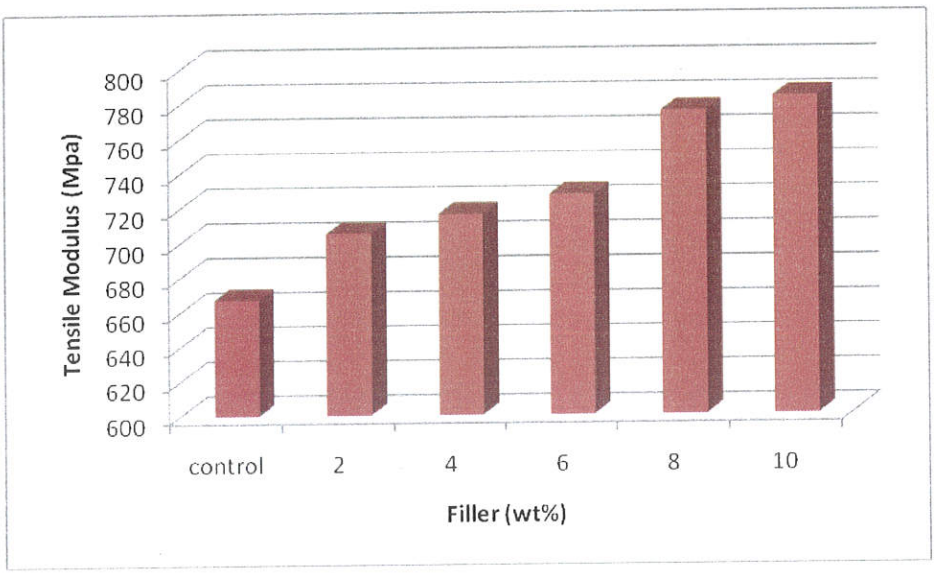

Figure 4.3: Variation of Tensile modulus to the groundnut shell particulate

Table 4.3: Tensile Modulus Data

Filler Wt(%)	Tensile Modulus
Control	667
2	705
4	716
6	727
8	775
10	783

Figure 4.3 and table 4.3 above show the effect of the groundnut shell particle on the composite. It indicates that the material property was enhanced and performed better at each increase in filler content compared to the control, having a value of 667 MPa. From the graph above the 10 wt% filler had the best tensile modulus property.

4.1.4 Hardness Test Result (Rockwell)

Table 4.4 shows the Hardness results on the composites.

S/N	Materials	Hardness value
1	100% Polyester 0% GSF	74.966
2	98% Polyester 2% GSF	79.666
3	96% Polyester 4% GSF	77.433
4	94% Polyester 6% GSF	81.533
5	92% Polyester 8% GSF	79.166
6	90% Polyester 10% GSF	75.566

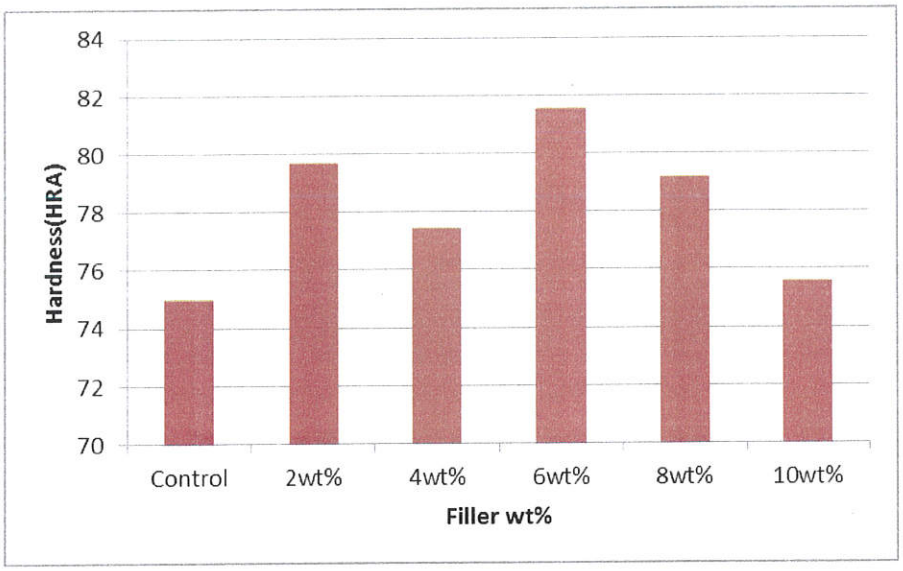

Figure 4.4: Variation of Hardness to filler concentration

Figure 4.4 and Table 4.4 above indicates the relationship between the hardness of the composite and the variation in ground shell particulate. The graph shows an irregular increase and decrease in hardness as the groundnut shell wt % increased. However there is a noticeable increase in the 6 wt% with value 81.533 HRA which is high compared to the control having a value of 74.966 HRA. This connotes that the groundnut shell particulate is a good reinforcing material and will suffice in areas of application where hardness is required.

4.1.5 WEAR TEST RESULT

Table 4.5 shows the wear index of the composites.

Specimen	Wear index
100% Polyester 0% GSF	0.251
98% Polyester 2% GSF	0.007
96% Polyester 4% GSF	1.04
94% Polyester 6% GSF	0.183
92% Polyester 8% GSF	0.896
90% Polyester 10% GSF	0.073

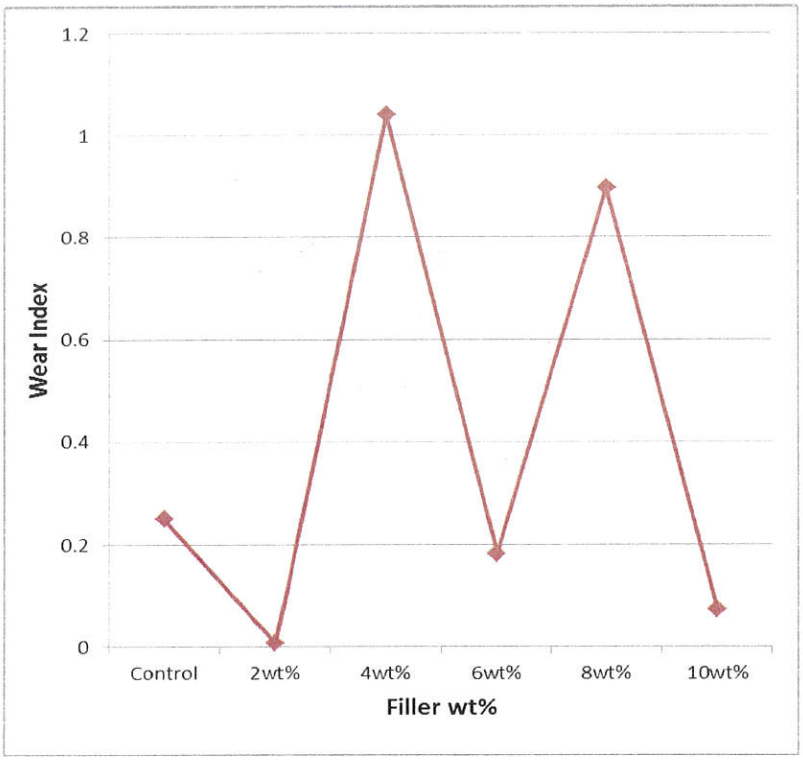

Figure 4.5: Chart of wear index of composites.

The results shown in figure 4.5 above show the behavior of the groundnut shell reinforced composite to wear property. During the course of friction occurring between the surface of the composites and the disc of the wear machine (Taber Abrasive Machine). It clearly shows that the composites wear property was enhanced at 4wt% reinforcement and had the best wear index of 1.04 compared to the control of 0.251.

4.1.6 WATER ABSORPTION

Table 4.6: Variation of Weights of Composites Immersed in Water

S/N	Specimen	Initial Weight (g)	Final Weight (g)
1	100% Polyester 0% GSF	4.40	4.40
2	98% Polyester 2% GSF	8.79	8.66
3	96% Polyester 4% GSF	28.82	28.82
4	94% Polyester 6% GSF	27.90	27.98
5	92% Polyester 8% GSF	32.30	32.34
6	90% Polyester 10% GSF	6.43	6.35

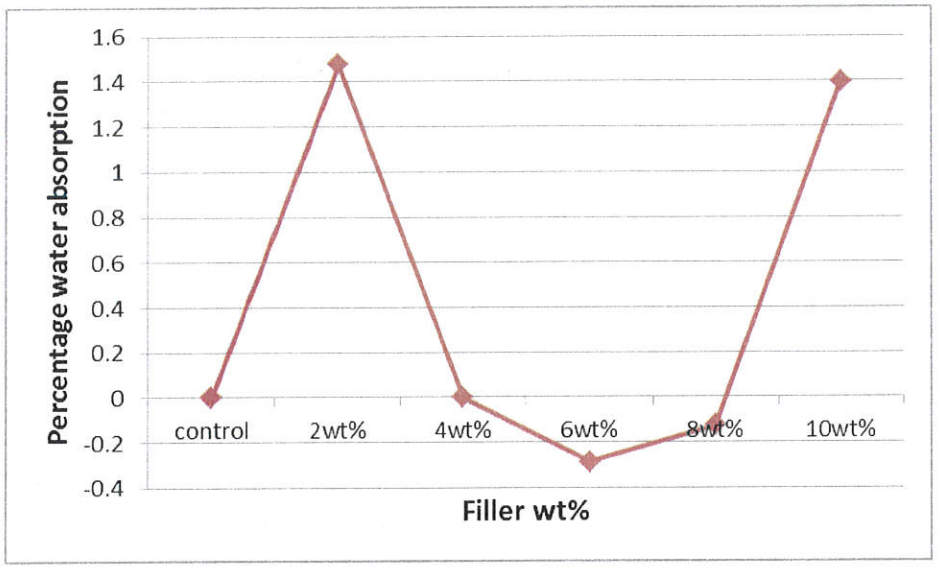

Figure 4.6: Chart of water absorption against filler concentration

The relationship between water absorption and groundnut shell particulate reinforced composites at different variation is as shown in figure 4.6. The composites showed an irregular pattern in its wear property as the control sample did not absorb water and the 2 wt% filler content had the highest water absorption property. However, the 4 wt% reinforced composite had the water resistance as it did not absorb water. This was similar to the water resistance property of the control sample. Further addition of groundnut shell particulate for 6 wt % and 8 wt % indicated that the composite deteriorated as it loosed weight. This could be as a result of the loose particle bonding of the groundnut shell.

CHAPTER FIVE

CONCLUSION

5.1 Groundnut shell reinforced polyester composite was successfully investigated and the following conclusions were drawn:

1. Groundnut shell particles consist of Potassium, calcium, phosphorus which makes the groundnut shell a good reinforcement.

2. The addition of groundnut shell as a reinforcement into the polyester resin enhanced the mechanical properties of the composite.

3. The tensile properties of the composite were enhanced as the 10 wt% reinforcement had the best tensile value and modulus of 12.20553 MPa and 783 MPa compared to the control of 10.84155 MPa.

4. It was also observed that the flexural property had a better strength of 29.0378 MPa at 10 wt% reinforcement compared to the control of 8.2253 MPa.

5. The composite produced an immense hardness in 6 wt% composite as it had a value 81.533 HRA which exceeds that of the control of 74.996 HRA.

6. The wear property of the composite was enhanced at 4wt% reinforcement and had the best wear index of 1.04 compared to the control of 0.251.

With regards to these results, the groundnut shell reinforced polyester composite will perform in areas of application that require mild strength such as in window frames of automobiles, casing of electrical appliances, and floors of airplanes.

5.2 RECOMMENDATIONS

1. The use of low volume fractions of Groundnut shell particle filler for composite production should be investigated.

2. The particle size of Groundnut shell should be reduced to a nano particle to improve the mechanical properties.

3. 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 Groundnut shell particle.

4. The rheological properties of polyester should be studied.

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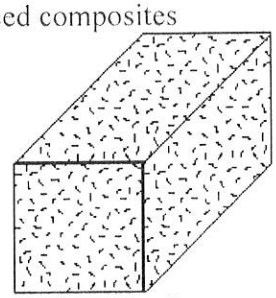
APPENDIX I
XRF Analysis of Groundnut Shell

Elements	Conc Value	Conc Error	Unit
P	502.35	±5.89	ppm
S	565.03	±4.35	ppm
K	8006.89	±1526.12	ppm
Ca	2987.75	±590.02	ppm
Ti	274.72	±51.13	ppm
Mn	62.44	±12.40	ppm
Fe	516.74	±87.08	ppm
Ni	52.33	±9.47	ppm
Cu	30.05	±5.25	ppm
Zn	11.50	±2.09	ppm

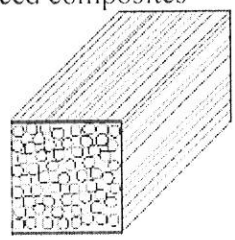
APPENDIX II

Structures of different types of composites

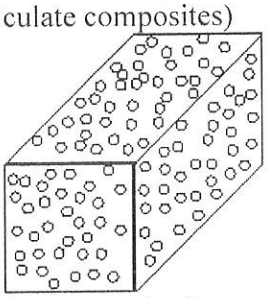
a. Random fiber (short fiber) reinforced composites



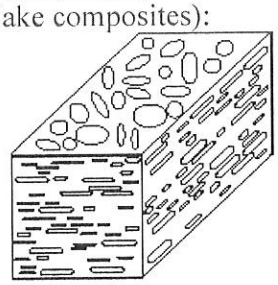
b. Continuous fiber (long fiber) reinforced composites



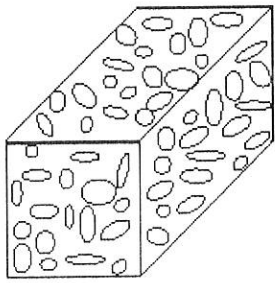
c. Particles as the reinforcement (Particulate composites)



d. Flat flakes as the reinforcement (Flake composites):



e. Fillers as the reinforcement (Filler composites):



APPENDIX III

Groundnut shell particle Production Process

Plate 1: Washed and Dried Groundnut Shell

Plate 2: Crushing and Grinding of Groundnut shell

Plate 3: Seive Analysis of Groundnut Shell Particle

Plate 4: Groundnut Shell Particles

APPENDIX IV
Composite Fabrication Procedures

Plate 5: Composite Fabrication

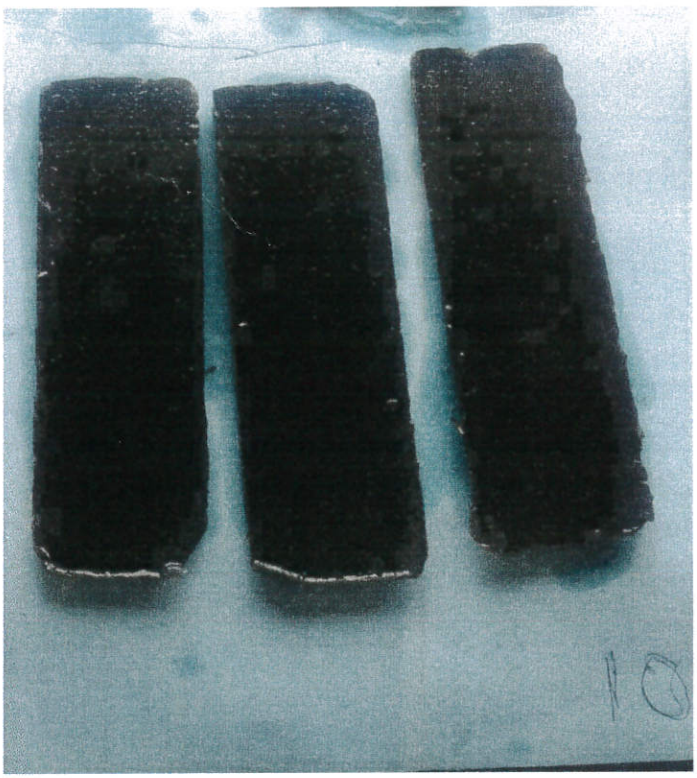


Plate 6: Flexural Test Specimen

Plate 7: Tensile Test Specimen

Plate 8: Wear Test Specimen

Plate 9: Cast Composites

APPENDIX V

Testing of Composites

Plate 10: Double cell Universal instron machine (Instron 3369)

Plate 11: An Electric powered Rockwell hardness testing machine (HRA 60)

APPENDIX VI

Water Absorption Test

Plate 12: Water Absorption Test