# THE USE OF NON-CONVENTIONAL NATURALCELLULOSIC FIBRES AS REINFORCEMENT FOR UNSATURATED POLYESTER COMPOSITES

**BY**

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**DEPARTMENT OF TEXTILE SCIENCE AND TECHNOLOGY**

**AHMADU BELLO UNIVERSITY, ZARIA**

**NIGERIA**

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## DECLARATION

I declare that the work in the dissertation entitledThe use of Non-Conventional Cellulosic Natural Fibres as Reinforcement for Unsaturated Polyester Composites has been performed by me in the Department of Textile Science and Technology under the supervision of Professor O.K.Sunmonu, Professor E.G. Kolawole and Professor K.A. Bello.

The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation was previously presented for another degree or diploma at any University.

SABO, Aminu MusaOctober, 2014

| _____ | _____ | _____ |
|---|---|---|
| Name of student | Signature | Date |

## CERTIFICATION

This dissertation entitled "THE USE OFNON – CONVENTIONAL CELLULOSIC NATURAL FIBRES AS REINFORCEMENT FOR UNSATURATED POLYESTER COMPOSITES" by Sabo, Aminu Musa meets the regulations governing the award of the degree of Doctor of Philosophy of Ahmadu Bello University, Zaria, and is approved for its contributions to knowledge and literary presentation.

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## ABSTRACT

This research investigates the use of three natural cellulosic plant fibres in the production of fibre reinforced composite materials. These fibresincluded 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 $80^0$C. 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 Hardnesswere 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.

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# CHAPTER ONE

## 1.0 INTRODUCTION

The word polymer is derived from the classical Greek word poly meaning "many" and meres meaning "parts" (Bilmeyer, 1980).Simply stated,a polymer is a long chain molecule that is composed of a large number of repeating units of identical structures.Certain polymers,such as protein and cellulose are found in nature, while others, including polystyrene,polyethene and polyamide,are produced by synthetic routes.In some cases some naturally occuring polymers can be synthesized.An important example is natural rubber which is known as polyisoprene in its synthetic form. (Natinee *et al*, 2006).

The birth of polymer science may be traced back to the mid nineteenth centuary.In the 1830s,Charles Goodyear developed the vulcanization process that transformed the sticky latex of natural rubber to a useful elastomer for fire use in nitrate.This was used in the 1860s as the first man-made thermoplastic fibre.In 1907, Leo Baekland produce Bakelite(phenolformaldehyderesin),and glyptal(unsaturated-polyester resin)was developed as a protective coating by General Electric in 1912. (Ashby and Jones, 1994).

### 1.1 Composites

A composite derives from the essentially simple and practical idea of blending together two or more homogenous materials which have very different properties (Smallman and Bishop, 1999).

These are materials that combine with one or more separate componentsin a design to exhibit the best properties of each component such as mechanical, optical, electrical and magnetic. Carbon fibres and bundles of multiwalled carbon nanotubes are used in polymers to control or enhance conductivity in application such as antistatic packaging. In the ancient Babylon, one of the lesser

wonders of the ancient world was made of bitumen reinforced with plaited straw. Straw and horse hair hadbeen used to reinforce mud bricks (for atleast 5000years) to improve their fracture toughness (Azura, 2007). Paper is a composite, so also concrete which is made up of cement, gravels and sands and often reinforced with steel rods. All natural materials which must bear load such as wood, bone and muscle are composites (Morton and Jones, 1989).

Generally, composites are applied to fibre reinforced engineering structural materials, in which the fibres are continuous or long enough that they can be oriented to produce enhanced strength properties in one direction (unidirectional) (Agawal *et al*, 1980).

### 1.1.1 **Fibre Polymer Composites**

Polymer composites are materials made from polymers or from polymer along with other materials such as fibres as reinforcing materials. Polymer composites werefirst developed during the 1940's for military and aerospace applications. Since then a considerable advancement has been made in the use of these materials and application in construction and automobile engineering. Nowadays designers and engineers readily turn to polymer composites, because of their synergistic combination of properties that are not readily available in any other material like metals and ceramics (Bilmeyer, 1980). The modern composites are usually made up of two components, a fibrous component and the matrix. The fibre is most often glass, but sometimes carbon, kevlar, boron, silicon, polyethylene, polyethyleneterephthalate (PET) fibre etc are used. The matrix is usually a thermosett like epoxy resin, Polydicylopentadiene or polyamides but recent advancement shows a shift toward thermoplastic polymer because of their lightness, resistance to corrosion and ease of processing (Kenny and Maffezolli, 1991). Composites, though generally more expensive than isotropic materials such as steel, aluminium and plastics, are used because they deliver benefits that cannot otherwise be obtained. Such benefits are the

result of features such as light weight, high modulus, high strength, low dielectric constant and good chemical resistance. Often,the end use application demands a combination of properties that eliminates the use of other materials.

In the last few decades, composites have become the preferred materials mainly used in high technology industries. The reasons composite materials are favoured over conventional materials is mostly due to their superior mechanical and chemical properties as well as their ability to be tailored specifically for the object at hand. That is, properties of composite structures being a function of its component materials, their distribution, the interaction between them etc. enables the designer to select individual materials; combine them together with a specific alignment and therefore manufacture a product with definite properties. Polymer composites are highly favoured materials in a wide variety of ways because they are relatively easy to manufacture and are lower in weight. In addition, mostly fibre reinforced composites are preferred for high technology applications due to their superior mechanical properties. A fibrous reinforcement is determined by their high aspect ratio. Fibre reinforced polymer (FRP) composites have a wide application range due to their orthotropic nature that is, their mechanical properties are different in different axes. This property results in much flexible designs that cannot be obtained with conventional isotropic materials or particle reinforced composites.

However these features come with a price; these materials are difficult to design and characterize while high performance composites are also expensive to manufacture. In recent years, studies have led to the development of several new manufacturing processes; some are expensive and exhibit high performance while some are cheap and display low performance and some in between. In the quest for cheaper, yet high performance processes, resin impregnation methods

were developed. The dozens of different impregnation techniques mostly differ in small details. These different processes have been patented by various companies.

## 1.2 STATEMENT OF RESEARCH PROBLEMS

The quality of composite materials is derived from inherent properties of its parent materials. These qualities that are derived from the physical properties include density, water absorption, and tensile strength, bending strength, impact strength and hardness. Other properties include resistance to chemicals and thermal degradation. These are some important properties of the composite materials. The presence and or magnitude of these properties vary from one material to another. However, the challenge is that no single monolithic material on its own could possess all these properties to our satisfaction in the production of composite materials. There is thusonly a slight emphasis for monolithic materials to satisfy the increasing need of our modern society.

On the other hand, different composite materials provide variety of properties to address the challenges of composites production. This is not possible without matching the inherent desired properties of composites materials. Increasing needs for the composite materials comes also with various challenges which include the need to define in both quantitative and qualitative terms, the properties of composite which include environmental, eco-friendliness, light weight as well as economic advantages. Hence the need to conduct research covering locally available resources that could be used for composite materials to satisfy the ever - increasing need of modernday life. The research problems also include;

i Improvement in reinforcement matrix composite cohesion

ii Use of non-conventional natural cellulosic fibre

iii Chemical treatment on the fibres surface

## 1.3 SCOPE AND LIMITATION

This research is limited to the use of non-conventional natural cellulosic fibres in reinforcing unsaturated polyester resin.The fibres involved are Baobab tree fibres, Dum palm fibres, and Luffa gourd fibres. These composites were produced and their properties were tested.

## 1.4 AIM AND OBJECTIVES OF THE RESEARCH

The aim is to fabricate composites with the following three different fibres (Dum palm, Luffa gourd and Baobab) using the handlay-up technique and to characterize the fabricated composite.

The specific objectives of this research are to:

1. Extract the fibres from the plants;

2. Determine some properties of the fibres such as Tensile and Morphology using Scanning Electron Microscopy;

3. Modify the fibre surface using sodium hydroxide;

4. Fabricate fibre-reinforced composites using hand lay-up technique;

5. Determine the physical, mechanical and chemical properties of the composites;

6. Determine the thermal properties of the composites;

7. Determine morphological properties of the composites using SEM and AFM;

8. Determine areas of utilization of the composites.

## 1.5 JUSTIFICATION FOR THE RESEARCH

Natural cellulosic fibres have high potential for use as composite materials to satisfy production needs. However, research is needed in order to scientifically explore and define the inherent physico-mechanical properties of materials as well as solve some environmental problems associated with the use of some of these materials. This research will concentrate on the potentials of some natural fibres that can be used in composite production that are suitable in light weight engineering applications thereby expanding the use of locally available and eco-friendly natural fibres.

# CHAPTER TWO

# 2.0 LITERATURE REVIEW

This chapter reviews research activities in the study of cellulosic fibres; material properties including reinforced materials and polyester matrix. The mechanical, physical and thermal properties of natural fibre reinforced composites are also reviewed.

## 2.1 Material properties

The major constituents of fibre-reinforced composites are the fibres as the reinforcement and polymers as matrix.

### 2.1.1 Properties of natural fibres

Natural fibres, especially bast (bark) fibres, such as flax, hemp, jute, henequen, and many others have been investigated by many researchers as fibre reinforcement for composites in recent years (Singleton *et al*, 2003; Keller, 2003; Valadez-Gonzalez *et al*, 1999; Oksman *et al*, 2003). Advantages of natural fibres over man-made fibres include low density, low cost, recyclability and biodegradability. These advantages make natural fibres potential replacement for glass fibres in composite materials for the use of its fibre and primarily grown in Europe and Asia (Berglund and Zollinge, 2002). The major natural fibre components are cellulose, Hemicellulose, lignin, pectin, waxes, and water soluble substances. Lignin and pectin act mainly as bonding agents. Cellulose is a hydrophilic glucan polymer of D-glucopyranose units, which are linked together by β-(1-4)-glycosidic bonds (Rowell *et al*, 2012). The large amount of hydroxyl group in cellulose gives natural fibre hydrophilic properties when used to reinforce hydrophobic plastic matrices; the result is a poor interface and resistance to moisture absorption (Alvarez *et al*, 2003). The hemicellulosic and pectin materials play important roles in fibre bundle integration and fibre bundle strength and individual fibre strength. Hemicellulosic polymers are branched, fully amorphous, and have a significantly lower molecular weight than cellulose. Because of its open structure containing many hydroxyl and acetyl groups, hemicellulose is partly soluble in

water and hygroscopic (Wallenberger and Weston, 2004). “Lignin's are amorphous, highly complex, mainly aromatic polymers of phenyl propane units” (Rowell *et al*, 2012). But lignin has the least water absorption among the natural fibre components (Wallenberger and Weston, 2004). Flax fibres are low in lignin content, but flax shives approaches close to that of the wood species (Sain and Fortierb, 2002).

Although natural fibre cannot achieve as high mechanical properties as glass fibres, it was reported that flax fibres exhibited very good mechanical properties compared to other natural plant fibres. This, together with good availability and competitive price, makes flax fibre promising for future use as fibre reinforcement in bio composite. In natural fibres, strength usually increases with moisture content and decreases with temperature, whereas, the Young’s modulus decreases as water is absorbed (Baley, 2002). Defects (‘nodes’ or ‘dislocations’) in fibres (weak points) are often the break points during a tensile test and may influence the mechanical properties (Davies and Bruce, 1998).

The mechanical properties of a natural fibre-reinforced composite depend on many parameters, such as fibre strength, modulus, fibre length, their orientation, the fibre-matrix interfacial bond strength etc. A strong fibre-matrix interface bond is critical for high mechanical properties of composites. A good interfacial bond is required for effective stress transfer from the matrix to the fibre whereby maximum utilization of the fibre strength in the composite is achieved. Modification to the fibre also improves resistance to moisture-induced degradation of the interface and the composite properties. In addition, factors like processing conditions/techniques have significant influence on the mechanical properties of fibre reinforced composites. Mechanical properties of natural fibres like flax, coir, hemp, jute and sisal, are very good and may compete with glass fibre in specific strength and modulus. Numbers of investigations have been conducted on several types of natural fibres such as kenaf, hemp, sisal, flax, bamboo, and

jute to study the effect of these fibres on the mechanical properties of composite materials. (Mansur and Aziz,1983) studied bamboo-mesh reinforced cement composites, and found that these reinforcing materials could enhance the strength and toughness of the cement matrix, and increase its tensile, flexural, and impact strengths significantly. On the other hand, jute fabric-reinforced polyester composites were tested for the evaluation of mechanical properties and compared with wood composite, and it was found that the jute fibre composite has better strengths than wood composites. A pulp fibre reinforced thermoplastic composite was investigated and found to have a combination of stiffness increased by a factor of 5.2 and its strength increased by a factor of 2.3 relative to the virgin polymer. Information on the usage of banana fibres in reinforcing polymers is limited in the literature. In dynamic mechanical analysis, (Laly*et al*,2003) have investigated banana fibre reinforced polyester composites and found that the optimum content of banana fibre is 40%. Mechanical properties of banana–fibre–cement composites were investigated physically and mechanically by (Corbiere-Nicollier *et al, 2001).* It was reported that kraft pulped banana fibre composite has good flexural strength. In addition, short banana fibre reinforced polyester composite was studied by( Pothan*et al,1997)*; the study concentrated on the effect of fibre length and fibre content. The maximum tensile strength was observed at 30 mm fibre length while maximum impact strength was observed at 40 mm fibre length. Incorporation of 40% untreated fibres provides a 20% increase in the tensile strength and a 34% increase in impact strength. Joseph *et al*. tested banana fibre and glass fibre with varying fibre length and fibre content as well.( Luo and Netravali,1999) studied the tensile and flexural properties of the green composites with different pineapple fibre content and compared with the virgin resin. Sisal fibre is fairly coarse and inflexible. It has good strength, durability, ability to stretch, affinity for certain dyestuffs and resistance to deterioration in seawater. Sisal ropes and twines are widely used for marine, agricultural, shipping, and general industrial use. It was found

that sisal, henequen, and palm fibre have very similar physical, chemical, and tensile properties. (Cazaurang *et al*, 1991). Carried out a systematic study on the properties of henequen fibre and pointed out that these fibres have mechanical properties suitable for reinforcing thermoplastic resins. Ahmed et al. carried out research work on filament wound cotton fibre reinforced for reinforcing high-density polyethylene (HDPE) resin. (Khalid *et al*, 1998) also studied the use of cotton fibre reinforced epoxy composites along with glass fibre reinforced polymers. ( Fuad *et al*,1998) investigated the new type woodbased filler derived from oil palm wood flour (OPWF) for bio-based thermoplastics composites by thermo gravimetric analysis and the results are very promising. (Schneider and Karmaker, 1996) developed composites using jute and kenaffibre and polypropylene resins and they reported that jute fibre provides better mechanical properties than kenaffibre. (Sreekala *et al, 1997)* performed one of the pioneering studies on the mechanical performance of treated oil palm fibre-reinforced composites. They studied the tensile stress-stain behaviour of composites having 40% by weightfibre loading. Isocyanine-, silane-, acrylated, latex coated and peroxide-treated composite withstood tensile stress to higher strain level.Isocyanate treated, silane treated, acrylated, acetylated and latex coated composites showed yielding and high extensibility. Tensile modulus of the composites at 2% elongation showed slight enhancement upon mercerization and permanganate treatment. The elongation at break of the composites with chemically modified fibre was attributed to the changes in the chemical structure and bond ability of the fibre. Alkali treated (5%) sisal-polyester bio composite showed about 22% increase in tensile strength. ( Ichazo *et al*,2001) found that adding silane treated wood flour to PP produced a sustained increase in the tensile modulus and tensile strength of the composite. Joseph and Thomas studied the effect of chemical treatment on the tensile and dynamic mechanical properties of short sisal fibre reinforced low density polyethylene composites. It was observed that the CTDIC (cardanol derivative of toluene diisocyanate)

treatment reduced the hydrophilic nature of the sisal fibre and enhanced the tensile properties of the sisal-LDPE composites. They found that peroxide and permanganate treated fibre-reinforced composites showed an enhancement in tensile properties. They concluded that with a suitable fibre surface treatment, the mechanical properties and dimensional stability of sisal-LDPE composites could be improved.( Mohanty *et al*, 2001) studied the influence of different surface modifications of jute on the performance of the biocomposites. More than a 40% improvement in the tensile strength occurred as a result of reinforcement with alkali treated jute. Jute fibre content also affected the biocomposite performance and about 30% by weight of jute showed optimum properties of the biocomposites.

Generally, natural fibres are processed below 200 ºC. Compared to glass and carbon fibres, natural fibres have low thermal degradation temperatures. The thermal degradation of natural fibres involves two main steps, one in the temperature range 200–290 ºC and the other, in the range of 300 ºC or higher (Saheb and Jog, 1999; Manfredi *et al*, 2006). The first one is the thermal depolymerisation of the hemicellulose and the cleavage of glycosidic linkages of cellulose. The second one is related to the decomposition of the α-cellulose (Manfredi *et al*, 2006). Lignin degrades at a temperature around 200 ºC (Sharifah *et al*, 2004), but the range is as broad as between 200 and 500 °C (Manfredi *et al*. 2006). Sometimes lignin degradation cannot be observed from thermal analysis curves because it overlaps with that of hemicelluloses and α-cellulose decomposition (Manfredi *et al*. 2006).

## 2.2 Classification of composite materials

Composites can be classified by the form of the components or by their nature.

### 2.2.1 Classification by the form

As a function of the form of constituents, composites are classified into two large classes:composite materials with fibres and composite with particles.

#### 2.2.1.1 Fibre composites

A composite material is a fibre composite if the reinforcement is in the form of fibres.The fibres used are either continuous or discontinuous in form,chopped fibres,short fibres etc.The arrangement of fibres and their orientation allow us to tailor the mechanical properties of composites to obtain materials ranging from strongly anisotropic in one plane.Designers thus have in composites a "tailored"material in which they can modify and tailor the physical and mechanical behavior by adjusting;

-the nature of the constituents;
-the proportion of the constituents;
-the orientation of the fibres;

According to the performances required.

#### 2.2.1.2 Particle composites

A composite material is a particle composite when the reinforcement is made of particles,in contrast to fibres,does not have a direction.

Particles are generally used to improve certain properties of materials or matrices,such as stiffness behavior with temperature,resistance to abrasion,decrease of shrinkage etc.In numerous cases particles are simply used as fillers to reduce the cost of the material without degrading the characteristics.

The choice of the particle-matrix association depends upon the properties wanted.For example lead inclusion in copper alloy make them easier to machine.Particles of brittle metals such as

tungsten,chromium and molybdenum incorporated in ductile metals improves their properties at higher temperatures while preserving their ductility at room temperatures.

Cermets are also examples of particle ceramic-metal composites adapted to high temperature applications.For example oxide based cermets are used for high speed cutting tools and for protectors at high temperatures.

Also,elastomer particles can be incorporated in brittle polymer matrices to improve their fracture and slack properties by decreasing their sensitivity to cracking.Thus particle composites cover a large domain that is constantly expanding.

#### 2.2.1.3 Laminated composites

The most common form in which reinforced composites are used in structural applications is called a laminate (e.g plywood,papers, etc.).It is obtained by stacking a number of this layers of fibres and matrix and consolidating them into the desired thickness.If the layers are of different constituent materials or of the same material with reinforcing pattern,the laminates is said to be a hybrid laminate.

#### 2.2.1.4 Hybrid composites

The incorporation of two or more types of fibres within a single matrix,is known as hybridization and the resulting material is reffered to as hybrid composite.The behavior of hybrid composite appears to be simply a weighted sum of the individual components in which there is a more favourable balance between the inherent advantages and disadvantages.

### 2.2.2 Classifcation by the nature of constituents

According to the nature of the matrix,composite materials are classified as organic,metallic,or mineral matrix composites.Various reinforcements are associated with these matrices.Only certain pairs of associations have an actual industrial uses;others are subject of development in research laboratories.Among these composites we can cited;

Organic matrix composites(polymer resins with fillers)with

-Mineral fibres; glass,carbon,kevlar etc

-Organic fibres;kevlar polyamide, etc.

-Metallic fibres;boron,alluminium etc.

Metallic matrix composites(light and ultra light alloys of alluminium, magnessium,titanium)with

-mineral fibres;carbon,siliconcarbide

-metallic particles;boron

-metallo-mineral particle;boron fibres coated with siliconcarbide

Mineral matrix compsites(ceramics)with

-metalic fibres;boron

-metallic particle;cermets

-mineral particle;carbides,nitrides etc.

Composites minerals with an organic matrix can be used only in a temperature range not exceeding 200-300 $^{0}$C,although composite materials with metallic or mineral matrix are used beyond that up to 600 $^{0}$C for a metallic matrix up to 1000$^{o}$C for a ceramic matrix.

**2.3 FIBRE - REINFORCED PLASTICS**

The reinforcement is usually in the term of long fibres, flakes, whiskers, discontinuous fibres or fabrics. However, in high performance composites, the choice is directed toward long fibres or fabrics. The desirable functional requirement of a fibre in fibre-reinforced plastics are; high modulus of elasticity in order to give efficient reinforcement, high ultimate strength, low variation in strength between the individual fibres, stable and retain their strength during handling and fabrication and the surface of the fibres should be uniform (Hallaway, 1978). Glass fibres are the most common of all reinforcing fibres for polymeric composites matrix, because of its cheapness, strength and flexibility, other fibres are carbon, use for high performance composites, Boron use in aerospace and military, Aramid (Kevlar) used in sporting goods and aircraft industries, silicon, polyethylene, polypropylene and many others (Agawal *et al*, 1980).

## 2.4 STRUCTURAL COMPOSITES

**2.4.1 Structural composites:** This is composed of two dimensional sheets or panels that have preferred high tensile strength direction such as is found in wood, continuous and aligned fibre reinforced plastics. The layer in laminar composites are stacked and lamented together such that the orientation of the high strength direction varies with successive layer. One example of relatively complex structure is modern ski and plywood.

### 2.4.2 Sandwich panel composites:

This consists of two strong outer sheets, which are called tace sheet and may be made of Aluminium alloys fibre-reinforced plastics, titanium alIoys and steel, the tace sheet carry most of the loading and stresses. The cone may be a honey comb structure that has less density than the

tace sheet, resist perpendicular stresses, and provide shear rigidity. Sandwich panel materials can be used in a variety of application that includes roots, floors and walls of building.

## 2.5 Nanotechnology:

In the simplest form, nanotechnology is defined as the control of matter on the atomic and molecular scale, generally 100 nanometer or smaller and fabrication of devices or materials that lie within that size range (Mas□ ud, 2008).

### 2.5.1 Concept:

The name "Nanotechnology" derives from nanometer, which is $10^{-9}$ of a metre. Nanometre is least imaginable unit of length and is better understood when compared human hair which is said to have the diameter of above 8 x $10^4$ nm. This implies that nanotechnology deals with matter at the nanometer scale, taking advantage of the minute particle size.

As particles decrease in size, the result is an increase in their surface area to volume ratio, making the proportion of atoms at the surface to be higher than the proportion of atoms in internal volume. A particle of 30 mm size for instance, has 5%' of its atoms at the surface, a particle of size 10 nm having 20% of its atom at the surface and particle of 3nm size having 50% of its atoms at surface (nanowerk), thus small particles have much greater surface advantage than large particle growth and catalytic chemical reactions take place at the surface. Therefore a given mass of material in nanometer size range is reactive and more catalytic than the same mass of material of large particle size (nanowerk). Additionally, increase in surface of quantum effect have the secondary benefit of enhanced reactivity, strength, electrical, optical and magnetic characteristics of the material and this phenomenon forms the basic ideas of nanotechnology.

#### 2.5.1.1 Nano materials

Nano materials are those materials with at least one dimension when 1-100 nm. There are three classes of nano materials

### 2.5.1a Layers

These are materials with one dimension (thickness) in the nano scale (1-100 nm) while the other two dimensions (length and breadth) are extended. Examples in this class are this film and surface coating.

### 2.5.1b Nano wires and nanotubes

These form the class of nano materials having two dimensions (cross-section) in the nano scale while the other (length) is extended. Nanotubes (popularly known as carbon nanotubes) are molecular scale tubes of graphite carbon with outstanding properties. They are among the stiffest and strongest fibre known and have remarkable electronic properties and many other unique characteristics.

### 2.5.1c Particles

These are materials with the entire three dimensions in the nano scale. Examples are precipitate, colloid and quantum dots (tiny particles of semi-conductor materials).

## 2.6 Atomic Force Microscopy

Atomic force microscopy (AFM) is another imaging technique with a resolving power well beyond that of optical microscopes, and is readily capable of resolving features on the order of 1-10 nm and in ideal circumstances less. A wide variety of AFM hardware and modes of operation

are available, but all work by rastering a nanoscopically sharp solid probe or "tip," typically 5-50 nm in diameter at its point, over the surface and measuring a response as the sample interacts with it. This response can be mechanical, electrical, thermal, and magnetic depending on the experiment performed, but mechanical forces normal to the surface with an oscillating tip is most commonly used to study organic materials.

In this type of AFM, the tip is mounted on the end of a flexible piezoelectric cantilever and so forces acting on the tip are transferred into deformation of the cantilever. The flexing of the cantilever is easily detected by the reflection of a laser from the cantilever to a position sensitive detector. The cantilever is mounted so that it can be scanned in X, Y and Z axes with respect to the sample surface, and in the very common "tapping mode" the tip is oscillated by applying a current to the cantilever piezo. Tapping mode is preferred for organic materials, as the tip only momentarily contacts the sample as it is rastered across the surface, thus minimizing the application of significant lateral forces which can damage the surface being studied. Tapping mode generates three-dimensional height maps by oscillating the "tip" vertically near its resonant frequency and the change in amplitude ($A$) is measured as the tip traverses the surface. The cantilever height (Z-axis) is adjusted rapidly during scanning to maintain constant amplitude in a continuous feedback loop, and the distance of this displacement serves as a proxy for the height of the sample features. Changes in the frequency or shifts in its phase as a function of the oscillatory force placed on the cantilever is also used to map changes in attractive or repulsive forces between the tip and sample. This phase mapping, which can be collected simultaneously with the height mapping, can offer contrast between materials with differing mechanical properties.

When feature size approaches the diameter of the tip (~15 nm), the aspect ratio of the tip fineness becomes a factor and influences the path the tip travels. This is not a trivial source of error, since

the width of the tip prevents the point from reaching the edge of small features, making them appear wider than they are.

## 2.7 Differential Scanning Calorimetry

Differential Scanning Calorimetry (DSC) is a thermal analysis technique that measures heat flow, the amount of energy needed to raise the temperature of a sample as a function of temperature. DSC works by applying heat energy to a sample and an empty cell, and measuring the differential in heat flow needed to raise both to a target temperature. The difference in heat flow is therefore due to the difference in heat capacities of the sample. DSC experiments performed in this work are primarily concerned with the temperature at which changes in heat flow occur. DSC is useful in polymer blends since it can detect changes in temperature-dependent kinetic transitions. DSC was used in this work to monitor the *glass transition temperature* ($T_g$), where a polymers changes from a glassy to a rubbery state, and is the temperature at which the polymer have sufficient thermal energy to enable chain segment motion. This transition causes an increase in free volume and a decrease in the heat capacity of polymers, as chain segments are free to move, thus requiring less energy to increase their vibrational motion. $T_g$ is derived from the midpoint of onset and end set temperatures of the discontinuity in the heat flow response curve. These onset and end set temperatures are defined by the intercept between the tangent of the heat flow midpoint and respective baselines. Uncertainty in the glass transition temperature calculation of materials as a function of temperature or time in order to quantify reactions arises from defining the baseline above and below the discontinuity and the resolution of the heat flow.

The $T_g$ of polymer blends can be used as a relative measure of polymer solubility. According to the Fox equation, when polymers are completely miscible, then a blend will exhibit a single glass transition temperature at a weight fraction averaged intermediate between the respective

homo polymer transition temperatures. As solubility increases, the two respective $T_g$ will shift until coalescing into a single transition, and so the degree of which the observed $T_g$ of blend components deviate from the temperature predicted in the Fox equation can be used to measure the relative solubility of partially miscible polymer blends.

## 2.8 THERMOGRAVIMETRIC ANALYSIS (TGA)

Thermal analysis is an important and very useful method to characterize any materials such as thermoplastic or thermosetting polymer matrix and also to determine the influence of natural fibres addition into the polymers (George *et al*, 1998). One of the accepted methods for studying the thermal properties of polymeric materials is the thermogravimetric analysis (TGA). TGA provides some information about the thermal stability and decomposition rate of materials and measures weight changes in involving gaseous emissions (Reis*et al*. 2008). Thermal analysis is also used as an analytical method in understanding the structural property relationships and thermal stability of composite materials, such as the incompatibility between fibres and polymer matrices.

The incompatibility of components affected deteriorating performance for thermal and mechanical properties. Stress transfer from the matrix to the fibre depends on fibre-fibre and fibre-matrix interactions, cellulose fibres and lignin were important terms on the properties of the composites. The majority of natural fibres as a function of cellulose fibres and lignin, have low degradation temperatures (~200°C), which make them inadequate for processing temperatures above 200°C (Pracell *et al*, 2006)

## 2. 9 Unsaturated polyester resins.

Unsaturated polyester resin is by far, the most important materials for general purpose composite structures and parts. They are the materials used for the familiar fibre glass boat. Corvette

automobile reactional vehicle, and all sorts of storage tanks, pultrudded piping, portable toilets, and countless commercial and military application. Unsaturated polyester is styrene polyester or polymer resin and usually contains inhibitors that allow their storage as liquid for a year or more. Once catalyses, the resin becomes rigid in time as short as one minute or as long as several hours. These changes occur during fabrication of polymer composite product, it is known as curing. The advantages of unsaturated polyester are its dimensional stability, affordable cost, ease of handling, processing and fabrication. Commercial polyester is heterogeneous products consisting of some phthalic anhydride, making anhydride and ethylene glycol, to form the resin. This resin is characterized with carbon-carbon double bonds which are capable to copolymerize with styrene monomers to produce hardened and cross-linked product (Lindsay, 1995).

**2.10 Epoxy resin**

Epoxy resins are probably the most important matrix for high performance structural composites.Epoxy is the matrix material that produces the highest strength and stiffness with the stronger reinforcements such as boron and graphite.The epoxies used in composites are mainly the glycidyl-ethers and amines.Epoxy resins can be synthesised from reaction of bisphenol a and epichlorohydrin and cured by the addition of polyamines. Cured epoxy resins are resistant to non oxidizing acids,alkalis and salts(Scott,1999).

**2.11 Phenol-formaldehyde resin**

Cured phenolic resins have out standing heat resistance,and resistance to cold flow,good electrical(insulation)properties and good dimentional stability.Based on these properties they are widely used in decorative laminates.Phenolic resins are produce from phenols and formaldehyde,and are divided into resole and novolac resins.The resole are prepared under

alkaline condition with Formaldehydel/Phenol(f/p) ratio less than one.While novolacs are prepared under acidic condition with (f/p) ratio less than one.Resoles are cured by applying heat/by-adding acids while novolacs are cured when reacting chemically with methylene groups into the hardenerd state (Goodman,1986).

### 2.12 Vinyl Esther Resin

The vinyl esther resins were developed to take advantage of both the workability of the epoxy resin and the fast curing of the unsaturated polyester resin.The vinyl resin has high physical properties than polyester resin,but costs less than epoxy resins.The synthesis starts with acrylic esther dissolved in a styrene monomer to produce vinyl esther resin which when cured with organic peroxide produced a tough and hard material.A composite product containing a vinyl esther can withstand high toughness demand and offer excellent corrosion resistance(Smith,1990).

### 2.13 Interface

The interface can be defined as the two-dimensional boundary between the fibre and matrix.It is critical in contributingto the control in composite properties because fibre matrix interaction occurs at the interface.This interaction can occur through three mechanisms:Mechanical coupling or micromechanical interlocking of the two materials,physical coupling such as vander waals or electrostatic interaction, orcovalent bonding(by way of coupling agent)between the fibre on the matrix.These interactions create an interphase region,which is a three-dimensional region near the fibre with properties different from either the fibre or the matrix.Fibre-matrix interface is the critical factor that determines to what extent the potential properties of the composite will be achieved and maintained during usage.Locallized stresses are usually highest at near the

interface,which may be the locus of premature failure of the composite.The interface must have appropriate chemical and physical features in order to provide the necessary load transfer from the matrix to the reinforcement(Gorbatkina,1992).

### 2.14 Coupling Agent

The high strength of polymer composites is dependent on a transfer of external stress from the matrix to the fibre.The bonding between the matrix and fibre could be improved by treating the fibre surface with a reagent,which could form an adhesion bridge between the matrix and fibre.For this reason,the use of surface active or coupling agent is used.Such coupling agents as alkyl silanes,organotitanates,high-molecular-weight carboxylic acids and esters,whichare attracted by both the matrix and fibre surfaces,it is believed that the hydroxyl groups which bond with the fibre surface and alkyl group to the matrix (Birley and Scott,1982).

### 2.15 POLYMER MATRIX

Polymer matrix is divided into two major categories thermoplastic and thermosets

Thermoplastic matrices consist of linear molecules which are held together by a weak secondary force, upon heating they become softened, melted and reshaped as many times desired. Examples are polyester, polyethylene, and polyvinylchloride.

Thermosetsmatrices are chemically joined together by crosslinking forming a rigid structure, and once formed or cross-linked; they cannot be melted or reshaped any more. Examples are epoxy resins, amino resin, alkyl resins, phenolic resins etc. The major function of a matrices in a fibre reinforced composites are, to transfer stresses between the fibre, provide barrier to adverse environment, protect the surface of the fibre from mechanical and chemical aggression and also to be responsible for off-axis properties and interlaminar shear as well as in phase shear

properties. Also the processibility on the eventual defect of a composite rely heavily on the physical characteristics of the matrix, such as viscosity, glass transition temperature and processing condition.

### 2.15a Constituents of Polymer Composites

The principal constituents that influence the strength and stiffness of the modern advanced polymer composites are the reinforcing fibres,the matrix and the interphases has to perform certain esssential functional requirements based on their mechanical properties so that a system containing them may perform satisfactorily as a composite(Hollaway,1978).

### 2.16.1 Fibre phase

Fibre is an important constituent in composites.A great deal of research and development has been carried out with the fibres,on their effects,types,volume fraction, architecture and orientation.Fibres generally occupy 30%-70% of the matrix volume in the composites.The fibre contributes the high strength and modulus to the composites.The element provides the resistance to breaking and bending under the applied stress or load.The fibres used in structural composites can be in form of chopped,woven,stitched or braided and the most common types of fibres used are glass,aramid,carbon,cellulose,metal and foil fibres(Podolyn,1996).

### 2.16.1a Glass fibres

Glass **f**ibre is one of the strongest reinforcement available in large quantities.It is relatively low in density,inexpensive,stiff,chemically resistance,fairly submissive to textile manufacturing techniquesand capable of some modification in composition.Glass fibres are divided into three main classes:E-glass,S-glass and C-glass. The E-glass is designated for electrical uses,S-glass for high strength,and the C-glass for high corrosion resistance.The E-glass is the most common

reinforcement material used in civil structures.It is produced from lime alumina-borosilicate that can be easily obtained from sand.The fibres are drawn into very fine filaments with diameters ranging from 2-13times$10^{-6}$m(Zweben,1989).

### 2.16.1b Aramid fibres

Aramid fibres are relatively recent industrial products and can be considered as an evolutionary products following nylon.The stiffness of aramid fibre range between glass and steel,they are lighter than glass,comparable energy before breaking,and much tougher and absorb considerable energy before breaking,even under impact conditions.Aramid fibres are two to five times the cost of glass fibres,and they are used in sport and transportation equipments and in protective systems where theballistic stopping exploits their superior impact resistance(Katz and Milweski,1978).Aramid fibres are synthetic organic fibres consisting of aromatic polyamides.The aramid fibres have excellent fatique and creep resistance.There are several commercial grades of aramid fibres available,the common ones used in structural applications are Kevlar 29 and Kevlar 49.As an anisotropic material,its transverse and shear modulus are in order of magnitude less than those in the longitudinal direction (Zweben,1989).

### 2.16.1c Carbon fibres

Carbon fibres represent the ultimate high temperature-end member of polymer matrix materials.It has one of the highest temperature capabilities under non-oxidizing conditions among known materials.The carbon or graphite fibre is made from three types of polymer precursors-polyacrylonitrile(PAN),rayon fibre and pitch.Although there are many carbon fibres available in the market,they can be divided into three grades:High strength,high modulus and

ultra-high modulus.They have lower thermal expansion coefficients than both the glass and aramid fibres.Carbon fibre is an anisotropic materialand its transverse modulus is in order of magnitude less than its longitudinal modulus.The material has a very high fatique and creep resistance(Zweben,1989).

### 2.16.1d Cellulose fibres

Cellulose is a natural substance that makes up a significant portion of plant life.The cellulose molecule(monomer) is quite complexand bonding is obtained by oxygen linkages to form polymer.Most polymers are made from petroleum or natural gas feed stocks.The raw material for the manufacture attractive properties of stiffness,strength,low specific gravity and resistance to handling and damage(Oleesky and Mohr,1964).

### 2.16.1e Metal fibres

An important area in the use of metal fibres in polymer composites is that of metal skeletal structures in the form of honeycomb panels.Beehive pattern honeycombs are often made from aluminium foil.These honeycombs are often made from aluminium foil.These honeycombs form the core of laminates with metal fabric-reinforced polymers on the fabrication of laminate.These types of structures have long been useful in the aircraft industry,sometimes for structural members of automobile interior panels such as doors,seats,and headlines(Kenneth and Michael,2004).

## 2.17Matrix phase

Matrix (resin) is another important constituent in polymer composites.The matrix transmits and distributes stresses onto the individual fibres and maintains the fibre separated in a desired orientation.It also provides protection against fibre abrasion as well as fibre exposure to moisture

or adverse environmental aggressionresponsible for off axis properties.The matrix causes the fibres to act as well as a team in resisting failure or deformation under load.There are two common classes of matrices(resins):Thermoplastic and Thermosetts resins(Steenkamer ,1992).

### 2.17.1 Thermoplastic matrix

Thermoplastic matrices have been used widely as a base for reinforced plastics.Thermoplastic materials remain solid at room temperature,but melt when heated and solidifies when cooled,since the long chain polymers do not chemically cross-link because they do not cure permanently.In this line,the thermoplasticmatrices are undesirable for structural applications.The most common thermoplastics matrices are polysulfone,polyetherimide,polyether ether ketone,polyphenylene etc.Composites made from these materials(resin) have strength and stiffness approaching that of a thermosett compositesand offer much better toughness(Titowa and Lanham,1975).

### 2.17.2 Thermosetting matrix

Thermosetting polymer matrices are usually formed from low viscosity liquids that becomes cross-linked by combination with a catalyst or by the application of someexternal form of energy,such as heat or radiation (UV and other types).Once thermosetting resins cure,they give a permanent shape that cannot be reshaped by means of heat or pressure.This characteristic makes thermosetts matrix composites very desirable for structural applications.The earliest composites were made with phenolic thermosetting matrix.The epoxides followed, then the urea,unsaturated polyesters and the siliconand there are now considerable more different types(Yang and Lee, 1994).

## 2.18 Surface modification of natural fibres

Because the hydrophilic nature of natural fibres often causes low interfacial properties between fibre and hydrophobic plastic matrix, chemical modifications are considered to optimize the interface of fibres. Chemicals may activate hydroxyl groups or introduce new moieties that can effectively interlock with the matrix. The development of a definitive theory for the mechanism of bonding by chemicals in composites is a complex problem. Generally, chemical coupling agents are molecules possessing two functions. The first function is to react with hydroxyl groups of cellulose and the second is to react with functional groups of the matrix.(Bledzki and Gassan, 1999) outlined several mechanisms of coupling in materials, namely: a) elimination of weak boundary layers; b) production of a tough and flexible layer; c) development of a highly cross linked interphase region with a modulus intermediate between that of substrate and of the polymer; d) improvement of the wetting between polymer and substrate; e) formation of covalent bonds with both materials; and f) alteration of acidity of substrate surface. The different chemical modifications of natural fibres aimed at improving the adhesion with a polymer matrix was performed by a number of researchers. Some examples of chemical treatments are listed as follows:

### 2.18.1 Alkaline treatment

Alkaline treatment is also known as mercerization which is one of the most used chemical treatments for natural fibres when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. Addition of aqueous sodium hydroxide (NaOH) to natural fibre promotes the ionization of the hydroxyl group to the alkoxide (Agrawal *et al*, 2000):

Fibre-OH + NaOH Fibre-O-Na + $H_2O$

Thus, alkaline processing directly influences the cellulosic fibril, the degree of Polymerization and the extraction of lignin and hemicellulosic compounds (Jähn, 2002). In alkaline treatment, fibres are immersed in NaOH solution for a given period of time. A solution of 5% aqueous NaOH had been used to treat jute and sisal fibres for 2 h up to 72 h at room temperature (Ray *et al.* 2001; Mishra *et al*. 2001). Jacob and co researchers (2004) examined the effect of NaOH concentration (0.5, 1, 2, 4 and 10%) in treating sisal fibre-reinforced composites and concluded that maximum tensile strength resulted from the 4% NaOH treatment at room temperature. Mishra and co-researchers (2002) reported that NaOH treated (5%) sisal fibre-reinforced polyester composite had better tensile strength than 10% NaOH treated composites. Alkaline treatment also significantly improved the mechanical, impact fatigue and dynamic mechanical behaviours of fibre-reinforced composites (Sarkar *et al*, 2004; Joseph and Thomas, 1996).

**2.18.2 Silane treatment**

Silane is a chemical compound with chemical formula $SiH_4$. Silane coupling agents may reduce the number of cellulose hydroxyl groups in the fibre-matrix interface. In the presence of moisture, hydrolysable alkoxy group leads to the formation of Silanols. The silanol then reacts with the hydroxyl group of the fibre, forming stable covalent bonds to the cell wall that are chemisorbed onto the fibre surface (Agrawal *et al*. 2000). The reaction schemes are given as follows:

$$CH_2CHSi(OC_2H_5)_3 - H - {}_2O \rightarrow CH_2CHSi(OH)_3 + 3C_2H_5OH$$

$$CH_2CHSi(OH)_3 + \text{Fibre-OH} \longrightarrow CH_2CHSi(OH)_2O\text{-Fibre} + H_2O$$

Silane coupling agents were found to be effective in modifying natural fibre polymer matrix interface and increasing the interfacial strength. Three-aminopropyl trimethoxy silane with

concentration of 1% in a solution of acetone and water (50/50 by volume) was reported to be used to modify the flax surface at the interval of 2 h (Joseph and Thomas, 1996). Rong and co-researchers (2001). Soaked sisal fibre in a solution of 2% aminosilane in 95% alcohol for 5 min at a pH value of 4.5 to 5.5 followed by 30 min air drying for hydrolyzing the coupling agent. Silane solutions in a water and ethanol mixture with concentration of 0.033% and 1% were also carried by other researchers (Agrawal *et al*, 2000; Valadez-Gonzalez *et al*. 1999) to treat henequen fibres and oil palm fibres. It was verified that the interaction between the silane coupling agent modified fibre and the matrix was much stronger than that of alkaline treatment, which led to composites with higher tensile strength from silane-treated than alkaline-treated fibre (Valadez-Gonzalez *et al*, 1999). Thermal stability of the composites was also improved after Silane treatment (Agrawal *et al.* 2000).

**2.18.3 Acrylation treatment**

Acrylic acid ($CH_2$=CHCOOH) is also used in graft polymerization to modify fibre surface (Xu, *et al*, 2002; Karlsson and Gatenholm, 1999). This reaction is initiated by free radicals of the cellulose molecule. The cellulose is treated with an aqueous solution with selected ions and exposed to a high energy radiation. Then, the cellulose molecule cracks and radicals are formed (Bledzki and Gassan, 1999). Acrylation reaction is expected to occur at the hydroxyl groups of the fibre as shown below (Sreekala *et al,* 2000):

Fibre-OH + $CH_2$=CH-COOH $\xrightarrow{-NaOH}$ Fibre-O-$CH_2$-$CH_2$-COOH

Sreekala and co-researchers (2002) reported that fibres were mixed with 10% NaOH for about 30 min and then treated with solution containing different concentrations of acrylic acid at 50ºC for 1h. The fibres were washed with water/alcohol mixture and dried.

**2.18.4 Permanganate treatment**

Permanganate is a compound that contains permanganate group, $MnO_4^-$. Permanganate treatment leads to the formation of cellulose radical through $MnO_3^-$ ion formation. Then, highly reactive $Mn^3+$ ions are responsible for initiating graft copolymerization. (Wallenberger and Weston, 2004):

Most permanganate treatments are conducted by using potassium permanganate ($KMnO_4$) solution (in acetone) in different concentrations with soaking duration from 1 to 3 min after alkaline pre-treatment (Sreekala *et al*. 2002; Joseph *et al*. 1999; Paul *et al*. 1997; Joseph and Thomas, 1996). Paul and co-workers (1997) dipped alkaline-treated sisal fibres in permanganate solution at concentrations of 0.033, 0.0625 and 0.125% in acetone for 1 min. As a result of permanganate treatment, the hydrophilic tendency of the fibres was reduced, and thus, the water absorption of fibre-reinforced composite decreased. The hydrophilic tendency of fibre decreased as the $KMnO_4$ concentrations increased. But at higher $KMnO_4$ concentrations of 1%, degradation of cellulosic fibre occurred which resulted in the formation of polar groups between fibre and matrix.

**2.18.5 Other chemical treatment**

Sodium chlorite ($NaClO_2$) is usually used in bleaching fibres; however, it can delignify lignocelluloses. Studies have been conducted wherein it was used in fibre surface treatment for composites. Mishra and co-researchers (2002) dipped untreated sisal fibre, for use in sisal-polystyrene biocomposite, in sodium chlorite solution with a liquor ratio of 25:1 at 75 ºC for 2 h. It was reported that flexural strength was increased for bleached fibre composite because of lower stiffness and more flexible character of fibres after delignification. After delignification, the polymer replaces the role of lignin in fibres and makes composites more hydrophobic and tougher (Mishra *et al.* 2002). Other coupling agents like benzoyl peroxide (Sreekala *et al*. 2000; Joseph and Thomas, 1996), acetic anhydride (Sreekala and Thomas, 2003; Nair et al. 2001; Hill

*et al.*1998), maleic acid anhydride (Joseph and Thomas, 2003; Oever and Peijs 1998; Gassan and Bledzki, 1997), isocyanates (George *et al*, 1996; Maldas *et al*, 1989), and stearic acid (Zafeiropoulos *et al*, 2002; Paul *et al*, 1997) were also studied and used to modify the surface between fibre and matrix. Most researchers found these treatments were effective and showed better interfacial bonding.

**2.19 Fabrication Techniques**

The process of fabrication involves the conversion of a material from a less useful shape to a more useful one.The choices of manufacturing technique for the fabrication of fibre reinforced plastics or composite material is intimately related to the performance,economics and application of the materials.It also depend on a number of moulded surfaces and type of reinforcement.Important moulding methods for fabrication of polymer matrix composites.Structural parts may be classified under matched die mould,contact mould(also open mould),filament winding and pultrusion.These processes either use separate fibres and filament winding and pultrusion.These processes either use separate fibres and resins which are pre-imprignated fibres(indirect processes).There are two important stages in all moulding processes laying and curing.The laying is the process in which moulding materials are laid on a mould in the mould cavity or on the mould surface that conforms to the shape of the part to be fabricated. The process of curing helps the resin to set, there by providing the fabricated part a stable structural form.

An ideal processing technique is characterised by the following aspects;

-High productivity

-Maximum material cost

-Maximum geometrical flexibility

-Maximum property flexibility

-Minimum finishing requirement

-Reliability and quality manufacture.

## 2.20 Moulding Processes and Architecture of Composite Materials

The majority of molding processes consist in moulding structure of compositematerials with successive layers made up of matrix and reinforcement.This general technique,usually lamination,leading to forming laminates,will next lead us to being interested in the architecture of composite materials.

### 2.20 .1 Moulding processes

#### 2.20.1.1 Contact moulding

Contact moulding is the simplest method used for the manufacture of composite structures.This method requires a minimum of equipment and consquently a minimum of investment.These advantages were the origin of the success of glass fibre composite in both industry and crafts.

Contact moulding can be used to manufacture both small and large composite structures such as boat hulls,vehicles bodies,building panels,tanks etc,where small production runs are required.Although the proportion of fibres may vary, it nevertherless stay low,usually between 30% and 35% by weight of reinforcements.Contact moulding lead generally only to one smooth surface,whose quality finish is dependent upon the mould's surface.Lastly,the quality of the laminate obtained depend upon to a large measure upon the skill of the moulder.

**2.20.1.2 Hand lay-up**

Before moulding,the mould surface must be coated and thoroghly polished to obtain smooth glossy finish.A mould release agent is then generally applied using a spray.When the release agent is completely dry,a gelcoat is applied by spray or brush.In some cases a surfacing tissue can be used to reinforced the gelcoat.

The moulding is then performed according to the following operations;

1-The mould is coated with the catalysed and accelerated resin with a paint brush or roller

2-The reinforcement mat,cloth,e.t.c. is arranged inside the mould.Various types of reinforcement can be used for different parts of the structure.The reinforcement must be butted together and the joint compacted.

3-The reinforcement is next impregnated with the matrix,then the trapped air must be forced out by rolling with a split washer or similar roller.

4-Next,layers are built up using the same technique until the desired thickness of laminate has been obtained.In some cases it can be advantageous to allow theprevious layer to gel

before laminating is continued.Inserts can be put between these layers,tubes,bolts,nuts,frame works etc.

5-Removal from the mould is then carried out after a time that depend upon the resin and the temperature(of the order of 10hours).This time allows the moulding to mature.

6-Post cure is next carried out in the atmosphere over several weeks.This postcure can be accelerated by heating(for eg 5to 10hours at about 80 $^{0}$C).

7-After postcure,one then proceeds to the finishing of the moulding.

**2.20.1.3 Spray lay-up**

In this process the mould surface and the gelcoat are prepared in the same way as in the hand lay-up process.Next,the laminate is built up from chopped fibres and the resin system(polyester resin) sprayed simultaneously onto the mould.Continuous rovings are fed to an air chopper attached to a spray gun and chopped onto suitable lengths.Then the chopped fibres are shot onto surface by the stream of resin from the spray gun.Next,each layer must be carefully compacted by rolling to ensure that no air bubbles are trapped in the layer.Cure,demoulding and postcure are the same as hand lay-up.Spray moulding allows us to obtain short production cycle with a low cost price.The reinforcement is however restricted to chopped fibres and the mechanical properties of the laminate low.

It is possible to obtained two smooth faces using a mould and die coated separately and then brought together.This process also offers the possibility of inter posing a layer between both of them and then allows us to obtain laminate with better mchanical proprties.

**2.20.1.4 Vaccum moulding**

Vaccum moulding consist in the simultaneous use of vaccum and almospheric pressure. The mould is coated with release agent and the wet lay-up applied either or spray process. The pressure is exerted by means of a porous die covered by flexible membrane(generally made from neoprene) and a vaccum pump which sucks air out of the membrane.Heat is frequently applied to speed up the cure.If only one smooth surface is necessary the die can be suppressed and the pressure is exerted by the flexible membrane alone, this process is known as vaccum moulding.

The vaccum moulding process is suitable for limited production runs and forth manfacture of complex structures. It provides good mechanical properties as a result of the uniform distribution of finish is obtained on both surfaces. The speed of production is always quite low, however.

### 2.20.1.5 Compression Moulding

Resin injection moulding or resins transfer moulding (RTM) consists in resin injection under pressure to impregnate a reinforcement placed inside a very rigid and closed mould and die set. The automatic injection of the resin system reduces manipulation and materials processed.In this way moulding compounds including the resin system and the reinforcement (microsphere,chopped strands etc.) or pre-impregnated mats. These materials are extruded by an archimede screw, and the matrix is fluidized by heating and injected under high pressure into a heated mould where polymarization takes place.The types of material obtained is most frequently called 'reinforced plastics' rather than composite materials. In fact owing to the nature of reinforcements (short fibres, microspheres) the strength and the young modulus of the resins are multiplied by a factor of the order of two or four. Because ofthe high cost of moulds, this technique is generally suitable for large scale production of components of small and medium size.

### 2.20.1.6 Continuous Moulding

Continuous moulding is used for production of plates, sandwich panels, corrugated sheets for roofing, ribbed plates etc

This process can be divided into several stages

1. A stage of impregnating the renforcement fibre mats or cloths. Catalysed resin and reinforcing material are put onto a sheet of release film(cellophane,nylon,polypropylene etc.)

2. A stage in which the laminate is consolidated and shaped.

3. A polymerization stage carried out in an oven(at 60-150 $^{0}$C) in the form of a tunnel,the length of which(15-50 M) is a function of the temperature and the resin.

4. A cooling and cutting stage.

The stage of consolidating and shaping is done simply by acting on the thickness of the plate by passing between callendering rollers.In the case of corrugated sheets,the shaping occurs during polymerization.

A continuous moulding process can be completely automated,which then permits plates or panel to be made continuously.However,this process requires a very substantial investment in plant.

#### 2.20.1.7 Pultrusion moulding

Pultrusion moulding is a process used for producing profiles,straight or curved with constant cross section,strongly reinforced in the principal direction.In this technique,the reinforcement(roving,yarn,woven rovings,ribbon etc.) pass through a bath of catalysed resin where they are impregnated.The impregnated reinforcement is then drawn through a heated

die,which removes the excess of resin,determines the cross-sectional shape and cures the resin system.

This process is applicable to thermoplastic and thermosettingresins. The profile obtained have high mechanical properties taking into account the possibility of obtaining high proportions of reinforcement,upto 80% by volume.Production rates of 60m/hr can be achieved by this process.It requires a substantial investment in plant.Example of fabrication include fishing rods,various profiles and stiffeners.

#### 2.20.1.8 Centrifugal moulding

This technique is used for the moulding of items of revolution,in particular tubes,pipes,tanks,etc.It is an extrapolation of techniques for making pipes out of cast iron or centrifuged concrete

The mould revolution,coated with a release agentis set in in rotation(at least 2000rpm).After a possible gelcoating there is a simultaneous and continuous introduction of

-the reinforcement; chopped strands or rovings

-catalysed and accelerated resin(epoxide resin,polyester,etc.)

Impregnation of the reinforcement by the resin is accomplished by centrifugal motion.The resin system and chopped fibres are applied to the mould surface using a travelling feeder arm fitted with a chopped and spray gun which passes backward and forward while the mould is rotated.Polymerization is carried out at room temperature,or possibly accelerated in an oven.A reinforcement in the form of a roll(eg.mat,cloth) can possibly be introduced by interupting the rotation of the mould.The resin is next introduce at the same time of rotation

After polymerization the cylindrical pipe is extracted from the mould,the shrinkage of resin system allowing release from the mould.This technique allow us to obtain a good surface finish on the out side with awell-defined diameter and thickness of pipes.This fabrication process requires a large plant and great precision in balancing the mould.

# CHAPTER THREE

## 3.0 MATERIALS AND METHODS

### 3.1 MATERIALS

| Materials | Manufacturers /Suppliers | Remark |
|---|---|---|
| Unsaturated Polyester Resin | Nycil Nig. Ltd Ikeja | |
| Methylethylketoneperoxide | Nycil Nig. Ltd Ikeja | |

| | | |
|---|---|---|
| Cobalt | Nycil Nig. Ltd Ikeja | |
| Dimethylformamide | | |
| H2SO4 25% v/v | | |
| NaOH 25% w/v | | |
| Acetone | | |
| Tetrahydrofuran | | |
| Vaseline blue seal | | |
| Dumpalm fibres | From the plant | |
| Luffa Gourd Fibres | From the plant | |
| Baobab Fibres | From the plant | |
| Electronic Balance | | |
| Instron universal Machine | | Standard Organization of Nig. Kaduna Textile and Leather Lab |
| Izod Impacter | | Nilest, Zaria. |
| Hardness tester | | Nilest, Zaria. |
| Scanning Electron Microscope | ZEISS | Australia |

| | | |
|---|---|---|
| Atomic Force Microscope | | UPM, Malaysia. |
| Differential Scanning Calorimeter | PERKIN ELMER | South Africa. North-West University. |
| Thermogravimetric Analyzer | PERKIN ELMER | South Africa. North-West University. |
| X-Ray Diffraction Machine | | South Africa. North-West University. |
| FTIR Machine | SHIMADZU | NARICT Zaria. |
| Density bottle | | |

**3.2METHODS**

The three fibres for the study were extracted from the different plants, Dum palm fibres from Dum palm plant leaves. Luffa gourd fibres from Luffa gourd plant and Baobab fibres from Baobab plant.The fibres were extracted by retting in water for 7 days each. The extraction was done by mechanical processusing a special metal device designed which has a pointed edge. Fibres were all dried after extraction and thereafter treated with different concentrations of NaOH between 5 and 20 %. The sources of the fibre are shown in Figure 3.1-3.3



Figure 3.1 Baobab tree



Figure 3.2 Dum palm plant



Figure 3.3 Luffa gourd plant

### 3.2.1 MOULD FABRICATION

Mould was fabricated to produce composite in dumbbell shape. The mould was fabricated in Engineering Department of NARICT, Zaria

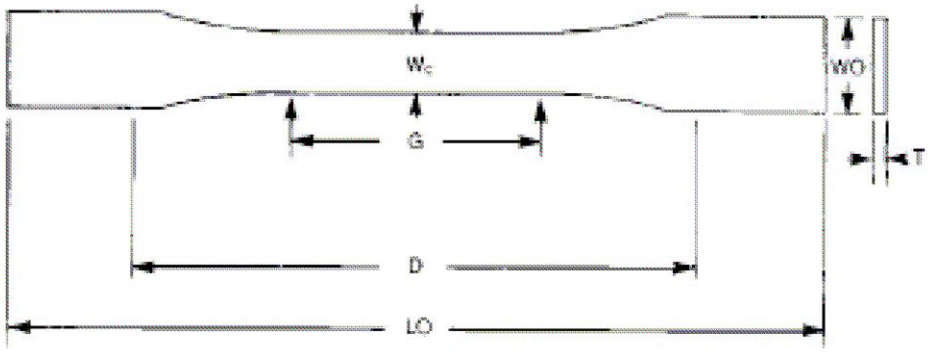


Figure3. 4 Dumb Bell Shape

$W_c$- Width of narrow section = 15mm
$W_o$- Width overall = 25mm
T- Thickness =3mm
G- Gauge length = 70mm
$L_o$- Length overall = 150mm
D- Distance between grips = 110mm



Figure 3. 5 Mould.

### 3.2.2 COMPOSITE FABRICATION

The composites were fabricated in dumbbell shape. Hand lay-up method was used. The mould surface was coated thoroughly with mould releasing agent, the release agent used was blue seal Vaseline petroleumjelly. It was with cotton lint. The resin mix was prepared with cobalt (1.25 %) as catalyst Methylethylketone Peroxide (.025 %) as accelerator, a layer of resin was first laid into the mould and then the fibres were arranged over a layer of the resin and resin layer over the layer of fibres, it was then allowed to cure at room temperature. After curing the composite was then removed. This was how each composite was fabricated. Fabrication of the composite was done using hand lay-up technique. The fibre surfaces were studied using Scanning Electron Microscope. Breaking load and extension percent of the three different fibres was determined using Instron testing machine, the composites were produced from untreated fibres and 5% treated fibres




Figure 3.6 Dumpalm Fibre Composites




Figure 3.7: Baobab Fibre Composites




Figure 3.8: Luffa Gourd Fibre Composites

## 3.3 TESTING PROCEDURE

The testing of the fabricated composites was carried out in accordance with the American Society for Testing and Materials (ASTM) Standard.

### 3.3.1 TENSILE PROPERTIES

The test procedure was carried out in accordance with ASTM Standard D638 of 2003. The tested composites were attached and gripped to the clamp at the upper and lower part of the universal testing machine. The test length of the composite was 85 mm and load cell of 5000 N force was applied on the specimen at a speed of 60 mm/min until a failure occurred. Elongation and breaking load and tensile strength were recorded for the three different composites i.e. Baobab fibre composites, Luffa gourd fibre composites and Dum palm fibre composites. The tests were carried out 3times for each composite.

### 3.3.2 IMPACT STRENGTH

The Izod impact strength tests were carried out in accordance with ASTM Standard D256 2003, to measure the impact strength. The specimen dimension of 100mm x 40mm x 3mm were tested using the machine Resil Impactor model P/N6957.000MN1. A hammer size of one joule (1 J) was used at an angle of $150^0$ to the sample axis to obtain a consistent starter crack. The samples were fractured in Resil Impactor Machine and the impact energies were recorded. The process was repeated three times for each sample.

### 3.3.3 BENDING MODULUS

The test was performed in accordance with the standard ASTM Standard D790 M. Three point bending test was performed to measure the flexural properties at a speed of 50 mm/min using the universal testing machine. Bending stress and elongation were recorded.

### 3.3.4 HARDNESS TEST

The hardness test of the composite was performed using Muver Durometer model 53505 EN ISO 88. The test was carried out according to the standard ASTM Standard D2240.

### 3.3.5 CHEMICAL RESISTANCE TEST

The chemical resistances of the composite were measured in accordance with ASTM Standard D543.The specimens were made into dumbbell shape. The chemicals used for the study are Hydrogentetraoxosulphate (VI) acid, $H_2SO_4$ (25%V/V). Hydrochloric acid, HCl (25%v/v). Sodium hydroxide NaOH (25%w/v). Ethanol, Acetone, Tetrahydrofuran (THF), Dimethylformamide (DMF)

The test was performed by dipping each of the composite samples in 100 ml of the above named reagents for 7 days at room temperature. The sample weight and thickness were measured and

recorded. After 7 days the specimens were taking out from the reagents and after drying were re-examined for their thickness and weight.

### 3.3.6 WATER ABSORPTION

The water absorption determination was carried in accordance with ASTM Standard D570 of 2003. Percentage water uptake (%Mt) was calculated according to the following equation

$\%Mt = (W_w _ W_d/W_d)\ x100$

Where $W_d$ and $W_w$ are weight of sample before and after immersion in water respectively.

The composite samples were weighed and recorded. They are then immersed in water for 48 hours after which they were removed and weighed again. The change in weight was recorded the percentage water uptake was calculated.

### 3.3.7 DENSITY

Density determination was carried out in accordance with the standard ASTM Standard D1895

Standard, (Floatation method using a density bottle).The densities of the samples were calculated using the formula

$$\text{Density} = \text{Mass (g)}/\text{Volume (cm}^3\text{)}$$

V is the volume of water displaced by the composite

M is the mass of the composite. The mass was measured using analytical balance mettle. Each sample was measured 3 times and the averages were calculated

### 3.3.8 SCANNING ELECTRON MICROSCOPY

The SEM of fibres was first studied after extraction, then the composites were fabricated and the interaction between the fibres and polyester matrix was investigated by SEM. The SEM micrograph of the untreated and treated fibres at different percentage concentration of alkali treatment and that of the untreated composite and 5% caustic soda treated fibre composites were investigated.

### 3.3.9 FTIR spectroscopy

. The chemical components of untreated and treated fibres were studied by using FTIR Spectrophotometer for ranges of wave numbers 4000–400cm$^{-1}$. The FTIR spectra were recorded by using KBr pellet technique. The fibres (1.15 mg) were crushed into fine particles and mixed with KBr powder (3.45 mg). The mixtures (0.77 mg) were then pressed into thin discs

### 3.3.10 Atomic Force Microscopy (AFM).

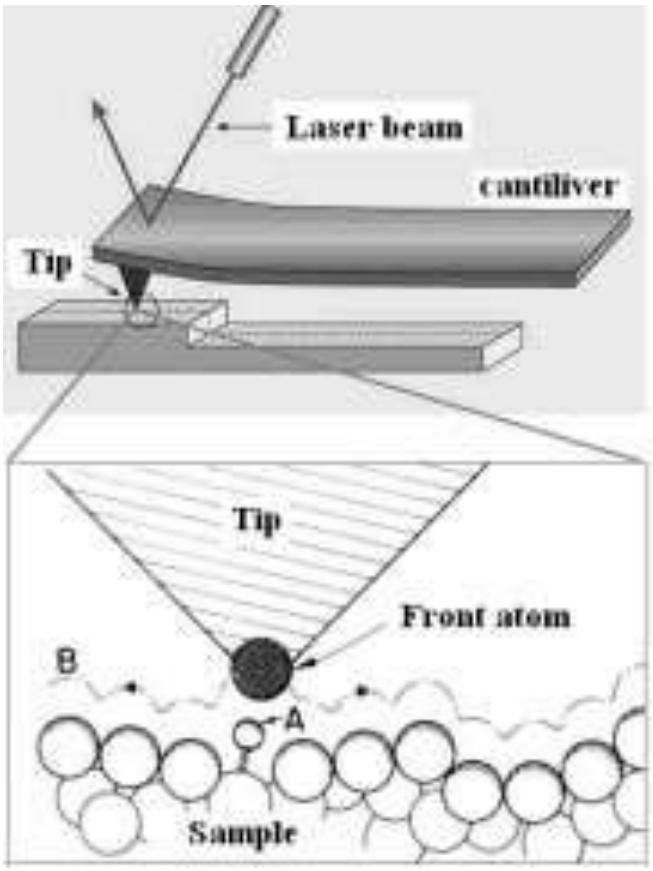



Figure 3.9: Schematic working principle of Atomic Force Microscopy (Hennady *et al*, 2004)

### 3.3.11 Measurement of thermal characteristic temperatures by DSC

Differential Scanning Calorimetry (DSC) is a technique that measures the amount of energy required to establish a nearly zero temperature difference between the sample and a reference material (Walshaw *et al*, 1998). DSC measurements provide quantitative and qualitative information about endothermic and exothermic reactions, as well as changes in heat capacity of a material, therefore it can be used to measure a number of characteristic properties of a sample. Polymer absorbs heat when being melted and releases heat during degradation. Therefore, the melting point and degradation temperatures of polymers or composites could be observed from the DSC curve. (Wang, 2004).

### 3.3.12: TGA MEASUREMENT

**Thermogravimetric Analysis**

The thermal stability of the samples (about 5 mg) was investigated with a Thermogravimetric Analyser Model Perkin Elmer TGA Pyris 1 under nitrogen gas from about 0 $^0$C to 600 $^0$C at a heating rate of 10 $^0$C/min.

### 3.3.13 X-ray diffraction Principle:

The strains developed in the crystallites due to treatment manifest as change in lattice planes, causing line shifting. These changes in the lattice planes are measured by X-ray diffraction method. Micro stress causes diffraction line broadening, while macro stress causes line shifting. The relation between the broadening produced and the non uniformity of the strain can be verified by differentiating the Bragg law. The spacing, d, of a set of planes in a crystal is related to the Bragg angle, $\theta$, and the wavelength, $\lambda$, by the equation

$D = \lambda/2\sin\theta$

# CHAPTER FOUR

## 4.0 RESULTS

### 4.1 Breaking Load and Extension of Bundle of Fibres

The breaking load and extension at break (%) of the three different fibres tested are shown in Figure 4.1a and 4.1b

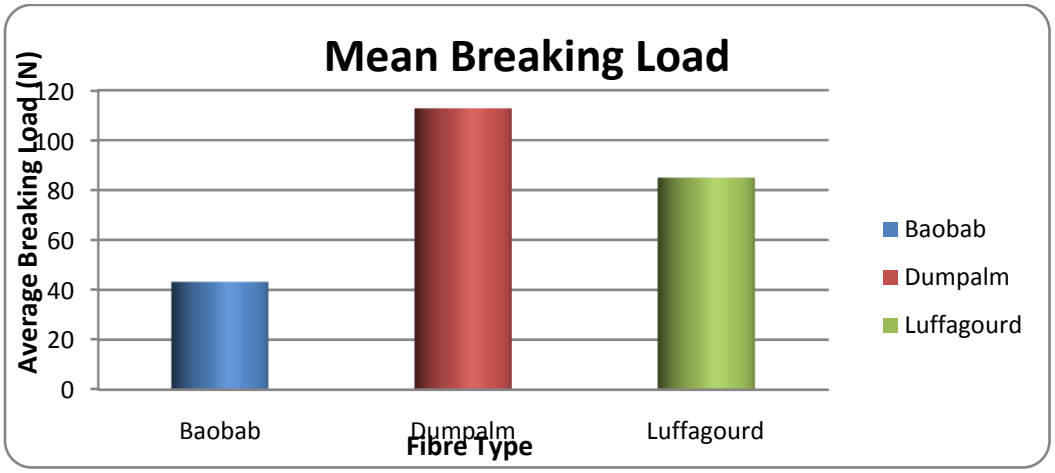



Figure 4.1a: Mean breaking load of fibres

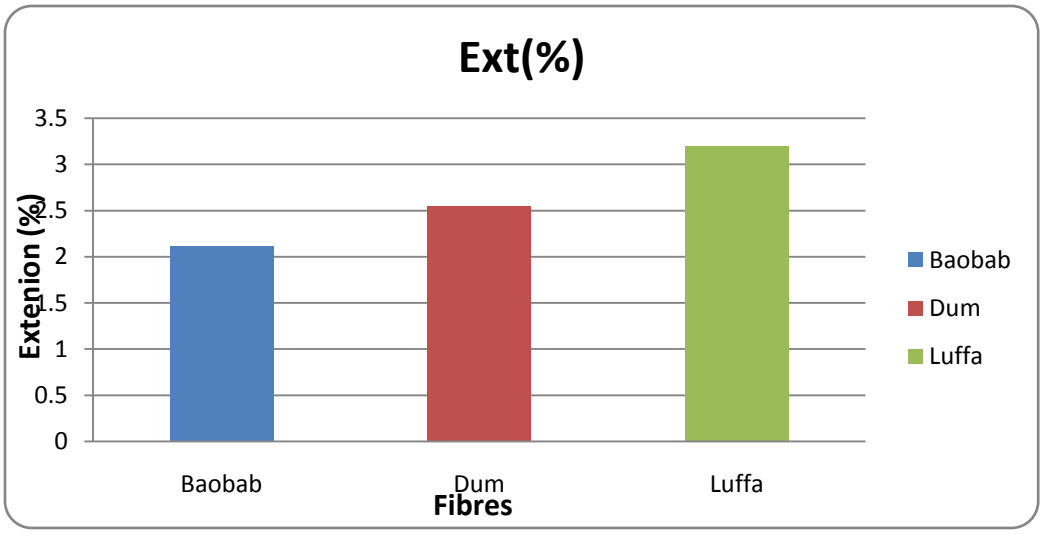



Figure 4.1b: Percentage Extension of Fibres

## 4.2 Scanning Electron Microscopy of the Fibres

Figure 4.2a-4.2i is showing the SEM micrographs of fibres for the three different fibres, that is Baobab fibre, Dum palm fibre and Luffa gourd fibre. Both untreated fibres, 5% sodium hydroxide treated fibres and 20% sodium hydroxide treated fibres were studied as shown in the micrographs.

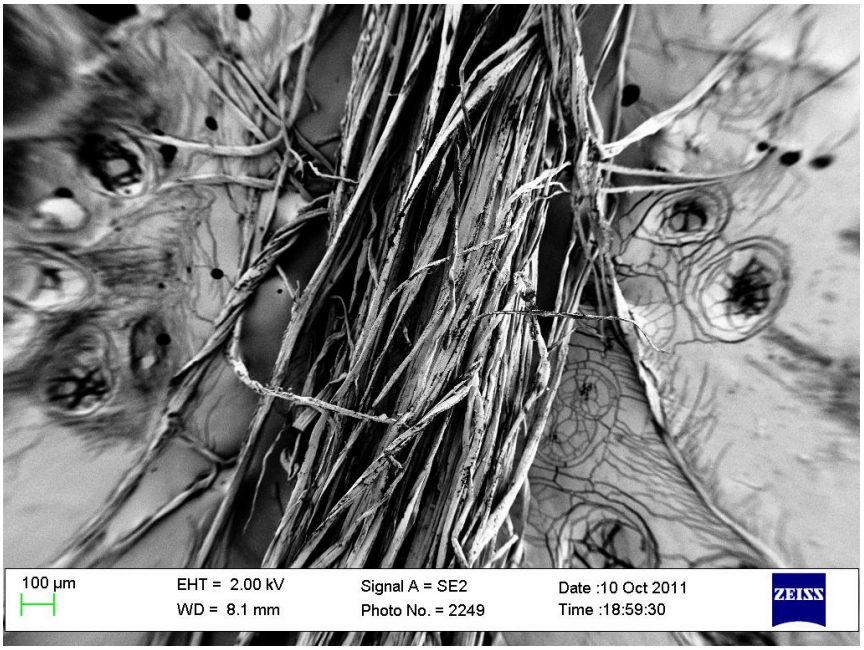



Figure 4.2a: Untreated, unwashed Baobab Fibres

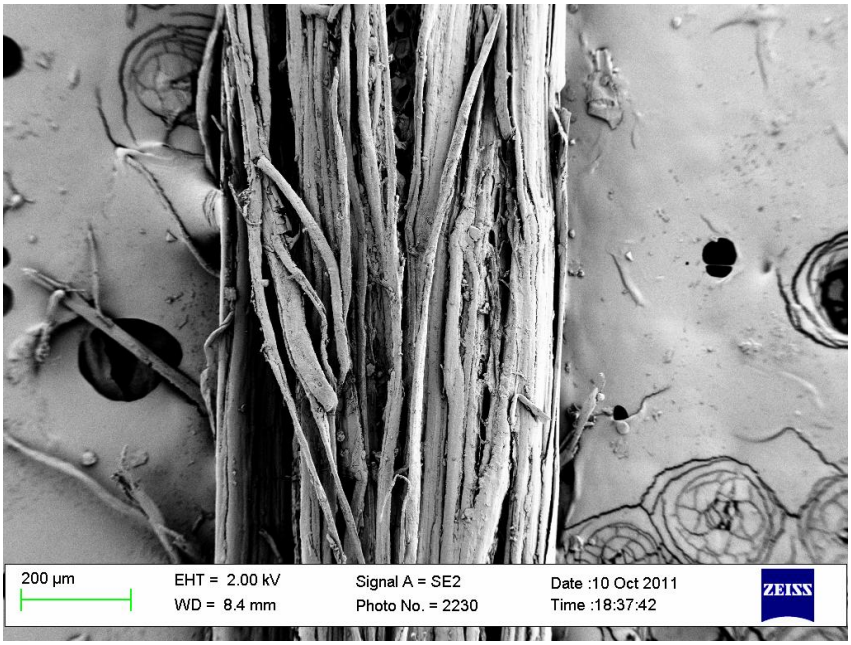



Figure 4.2b: 5% NaOH Treated Baobab Fibres

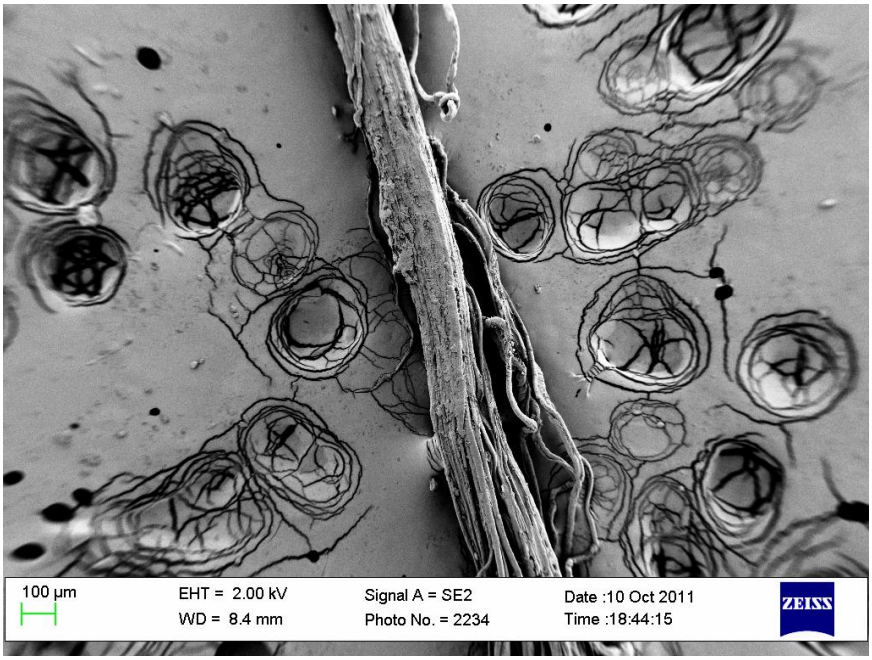



Figure 4.2c: 20% NaOH Treated Baobab Fibres

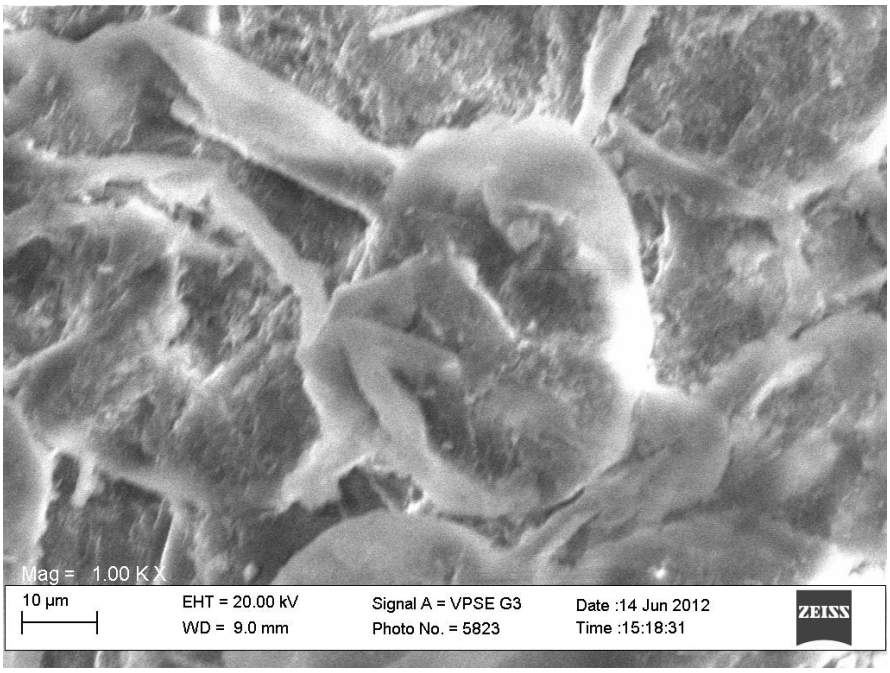



Figure 4.2d: Unwashed, untreated Luffa Gourd Fibres

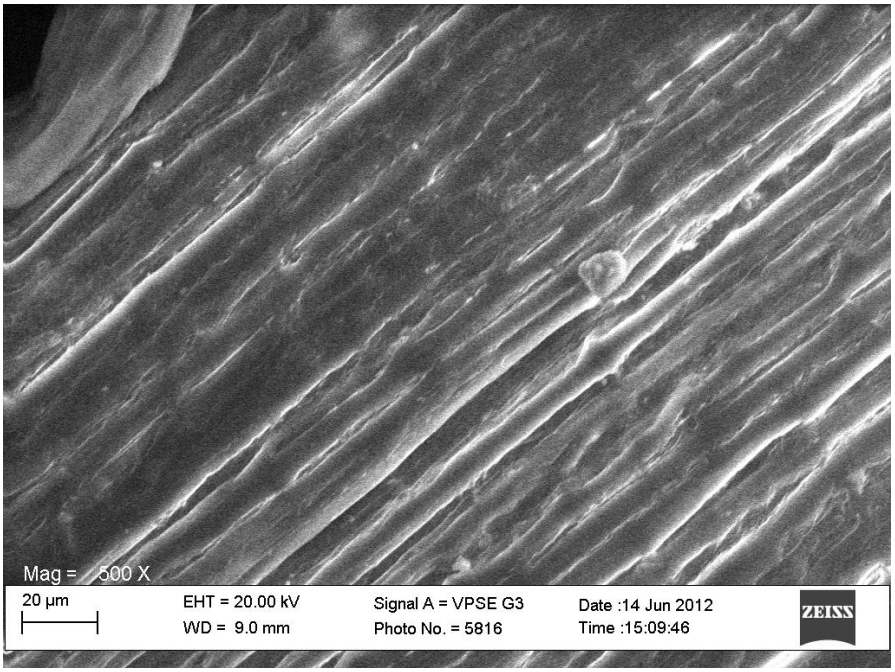



Figure 4.2e: 5% Treated Luffa Gourd Fibres

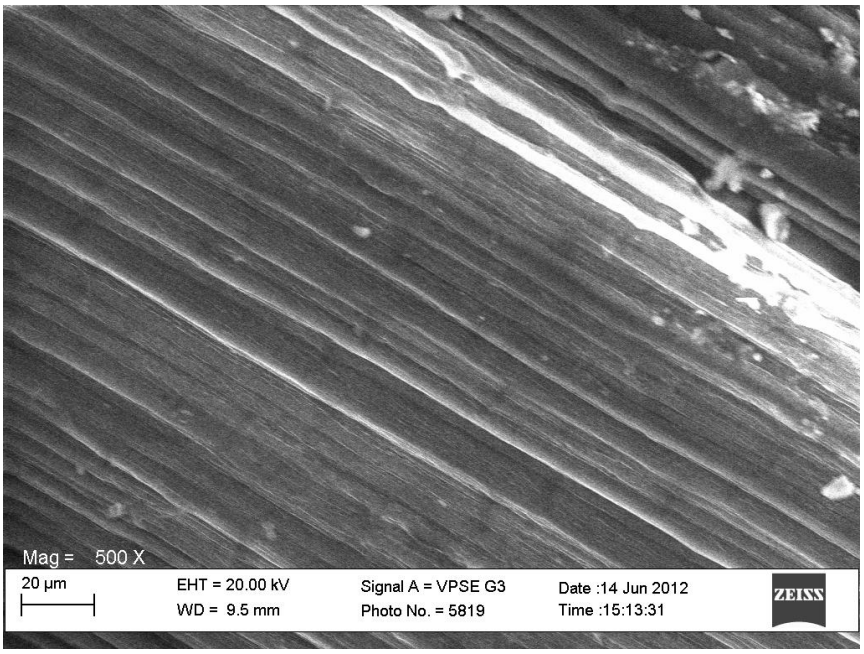



Figure 4. 2f: 20% Treated Luffa Gourd Fibres

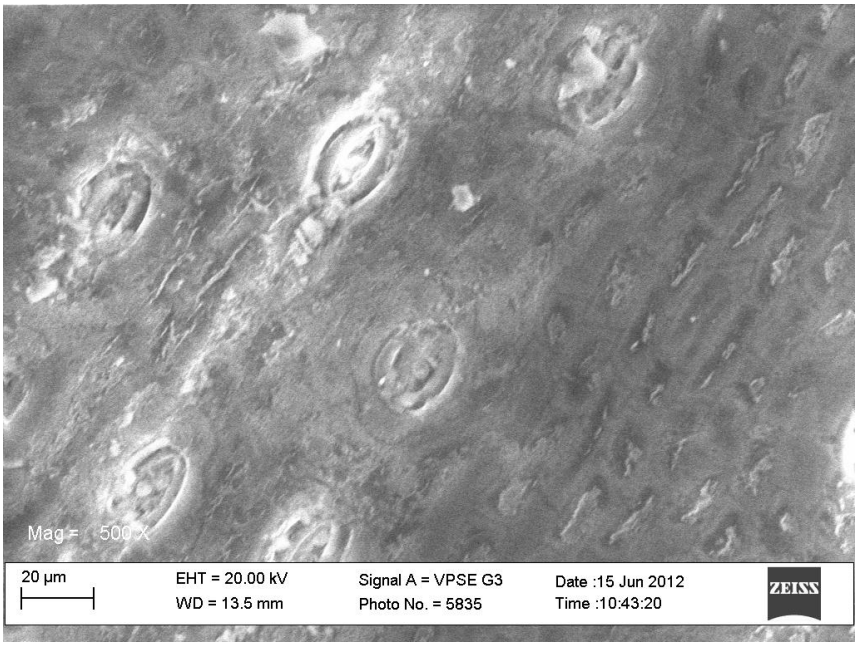



Figure 4.2g: Untreated unwashed Dumpalm Fibres

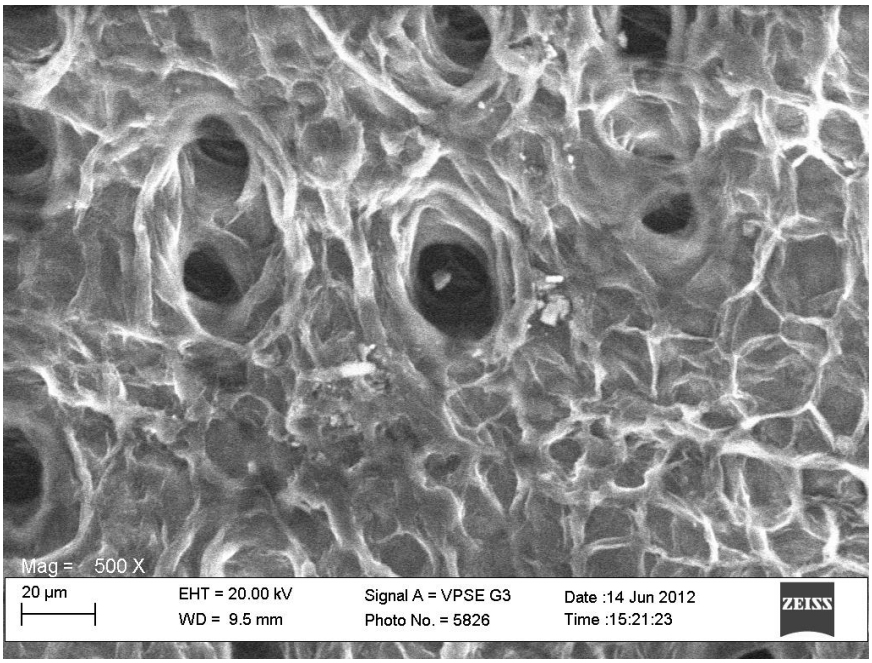



Figure 4.2h: 5% Treated Dum palm Fibres

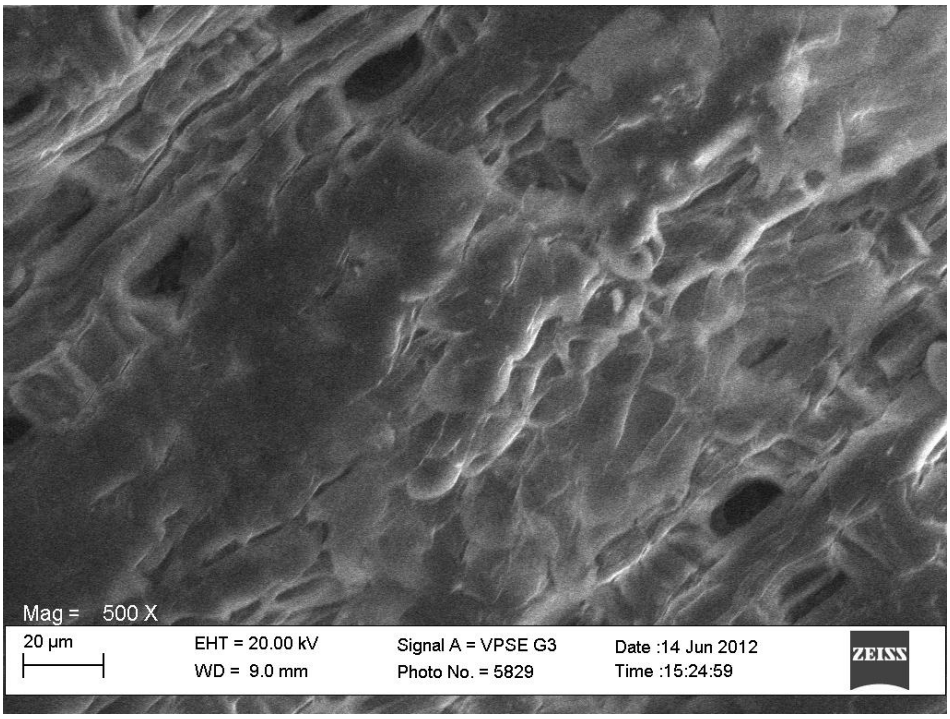



Figure 4.2i: 20% Treated Dumpalm Fibres

**Tensile Strength of Composites**

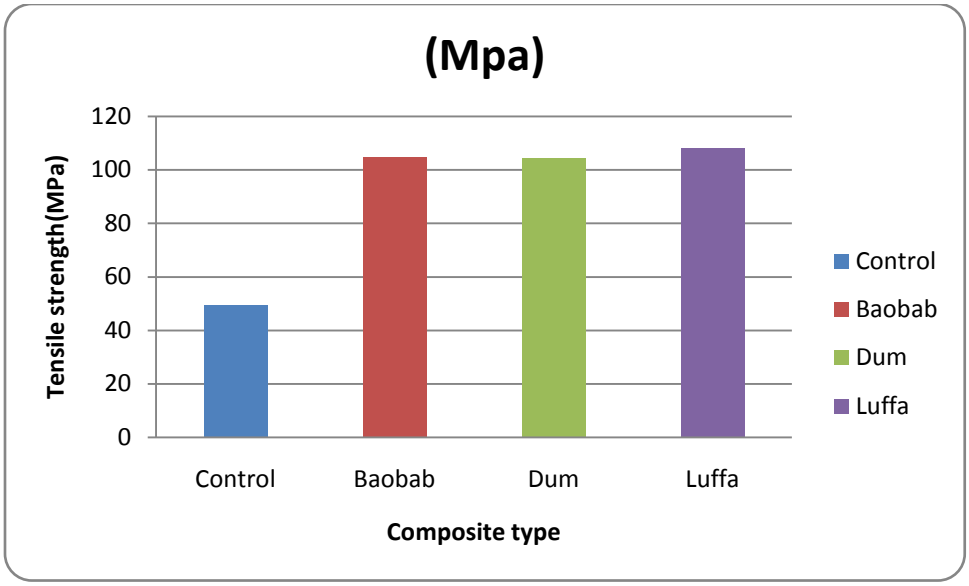



Figure4.3a: Tensile strength of untreared fibre composites

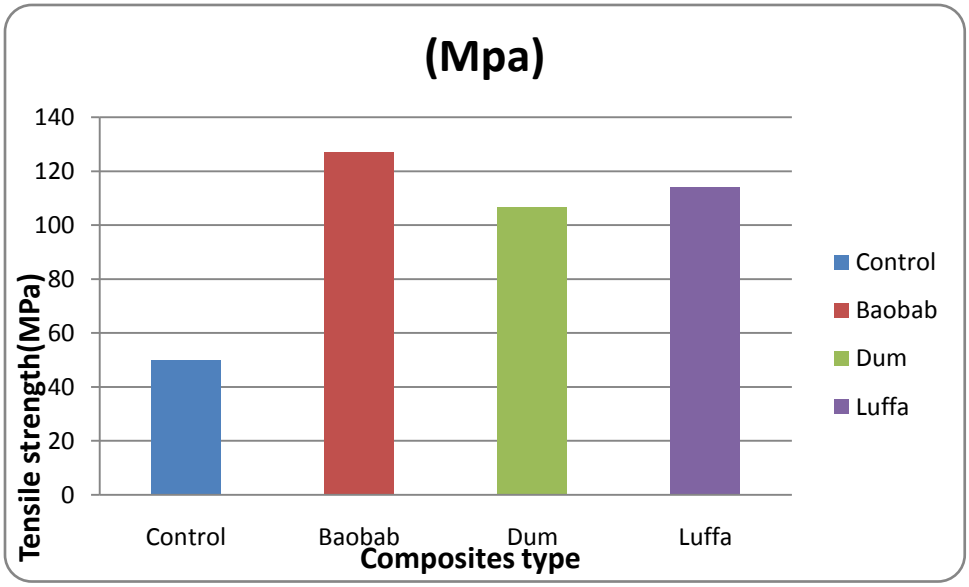



Figure4.3b: Tensile strength of untreared fibre composites

## 4.4 IMPACT STRENGTH OF COMPOSITE

The impact strength of the composites is as shown in Tables' 4.4a-4.4b. Impact strength of three fibres reinforced composites both untreated and 5% sodium hydroxide treated composites were studied and results are shown in the Tables.

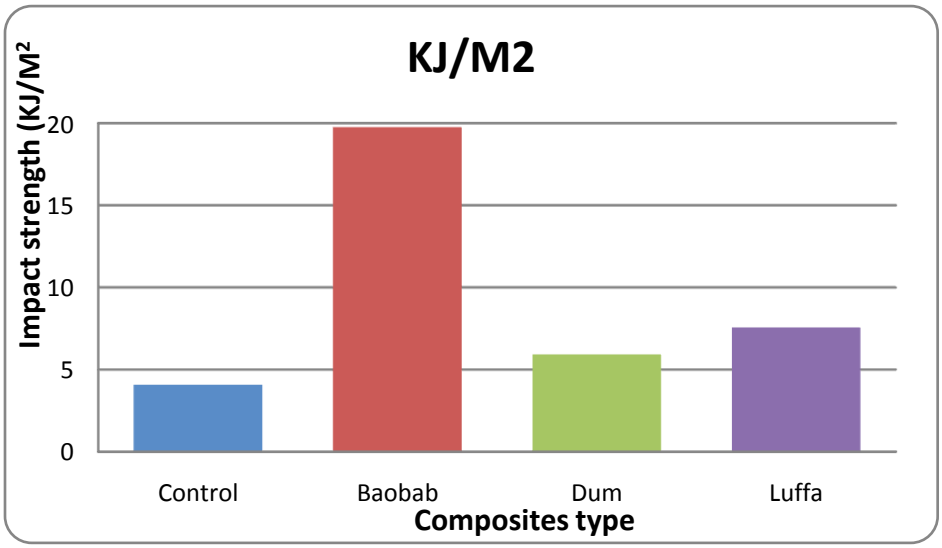



Figure 4.4a: Impact energy of untreated composites

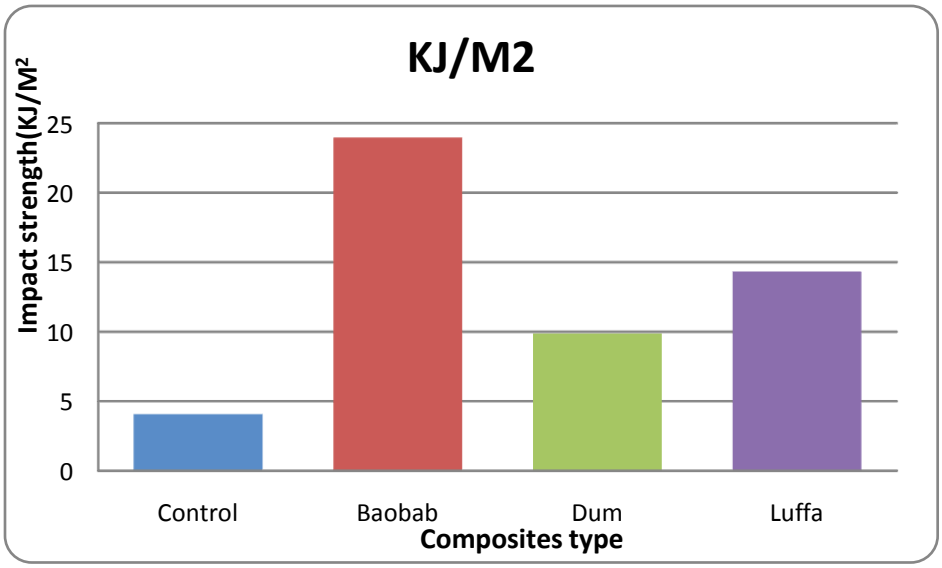



Figure 4.4b: Impact energy of treated composites

## 4.5 Bending Modulus of Composite

Bending modulus and elongation (%) of the composites is as shown in Figure 4.5a-4.5b Bending modulus and elongation percent of three different composites were determined, the determination was for untreated and treated fibre composite.

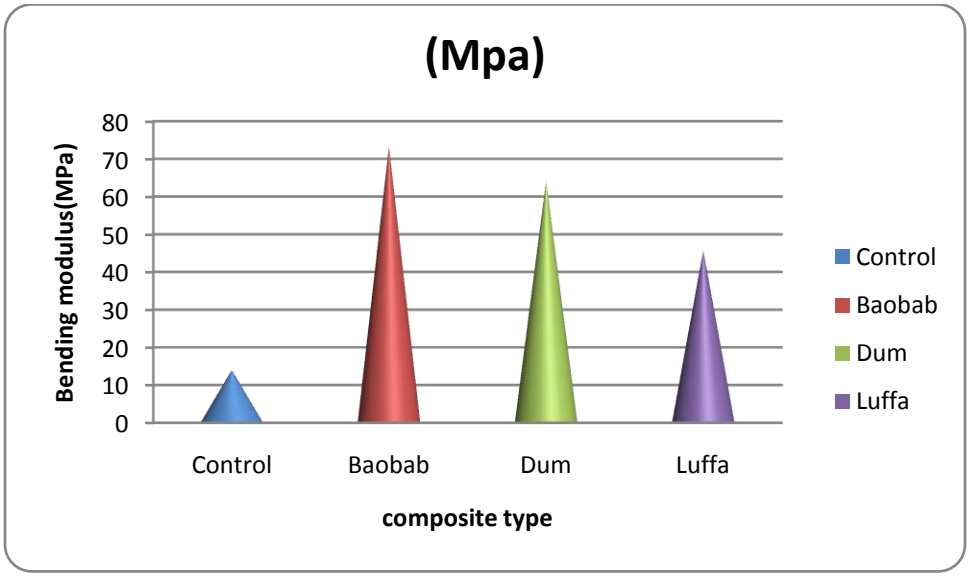



Figure 4.5a: Bending Modulus of untreated Fibre Composites

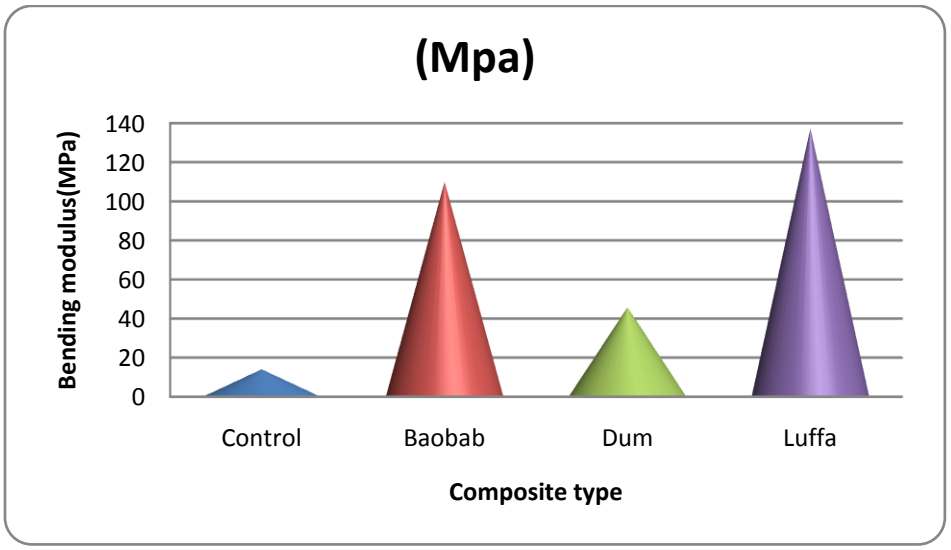



Figure 4.5b: Bending Modulus of Treated Fibre Composites

Hardness properties of the composites as shown in Table 4.6 Hardness properties of the three different fibres reinforced composites were studied, both untreated and treated fibre composites were studied.

## 4.6 Hardness Property of Composite

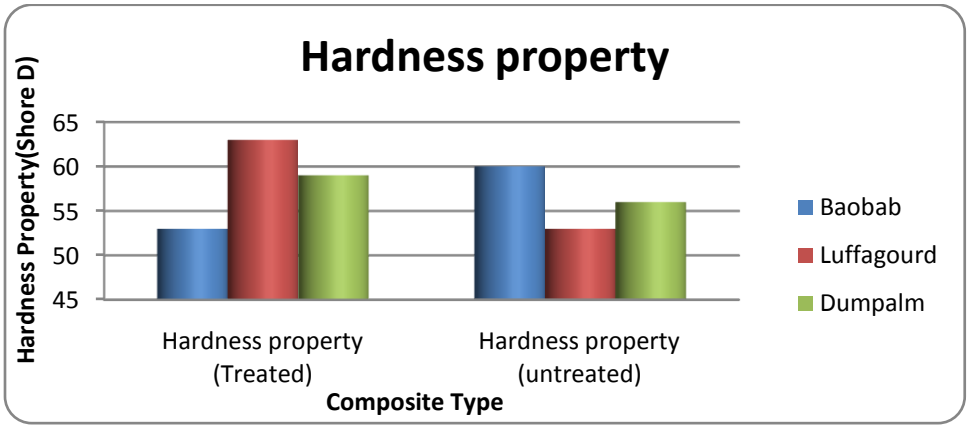



Figure 4.6: Hardness Property of Untreated and Treated Composites

## 4.7 Chemical Resistance of the Composites

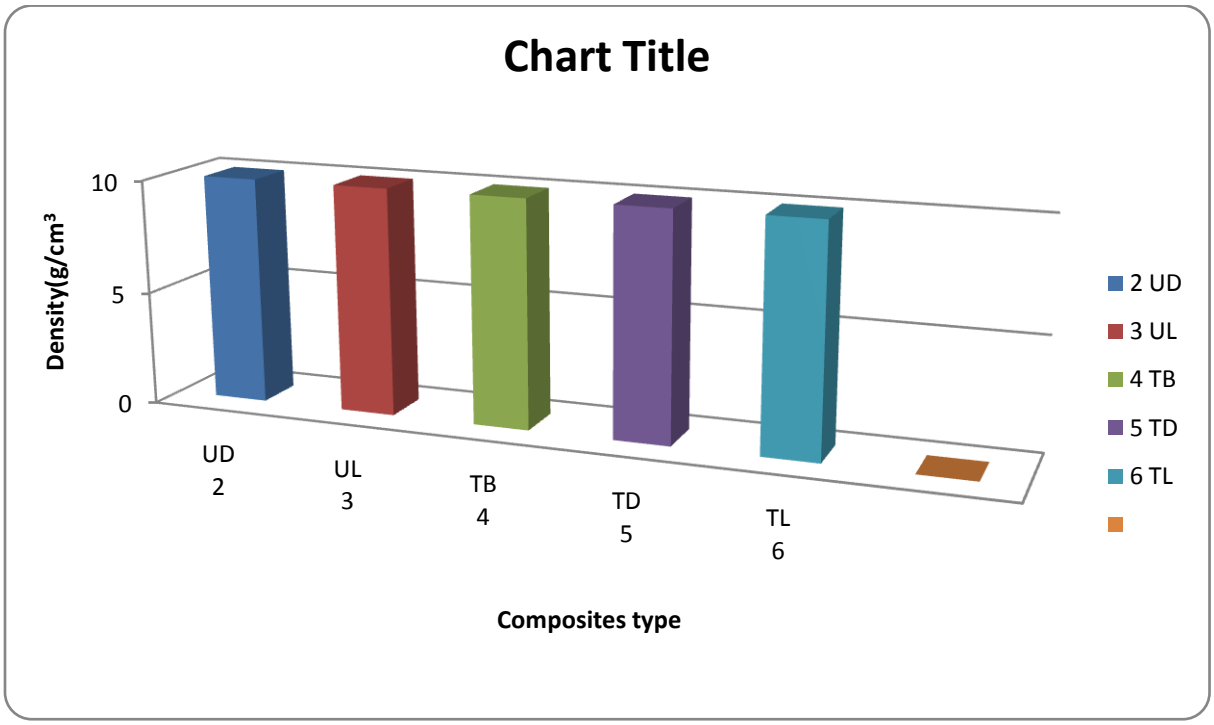
The chemical resistance property of composites is as shown in Table 4.6 Chemical resistance properties of composites were studied, in these study acid, alkali and solvents were used as seen in the Table.

**Table 4.6 Chemical resistances of Unsaturated polyester reinforced composites**

| Chemicals | Initial thickness (mm) | Final thickness (mm) | % Difference in thickness | Initial weight (g) | Final weight (g) | Difference In weight (g) | % Change in weight |
|---|---|---|---|---|---|---|---|
| Acetone | 3.32 | 3.30 | 0. 6 | 11.40 | 11.35 | 0.05 | 0.44 |
| DMF | 3.32 | Completely Dissolved | | 10.70 | Completely Dissolved | - | - |
| THF | 3.32 | 3.30 | 0. 6 | 10.70 | 10.40 | 0.30 | 2.8 |
| Ethanol | 3.32 | Completely Dissolved | | 13.90 | Completely Dissolved | - | - |
| 25%NaOH | 3.32 | 3.29 | 0.9 | 13.50 | 12.86 | 0.64 | 4.7 |
| 25%HCl | 3.32 | 3.28 | 1.2 | 12.20 | 11.50 | 0.70 | 5.7 |
| 25%$H_2SO_4$ | 3.32 | Completely Dissolved | | 12.20 | Completely Dissolved | - | - |

## 4.8 Density of Composite

The densities of the composites is as shown in Figure




Key:UD –Untreated Dum Palm ,UL – Untreated Luffa Gourd, TB –Treated Baobab,TD –Treated Dum palm TL- Treated Luffa Gourd

Figure 4.7: Densities of Composites

**Water Absorption of Composites**

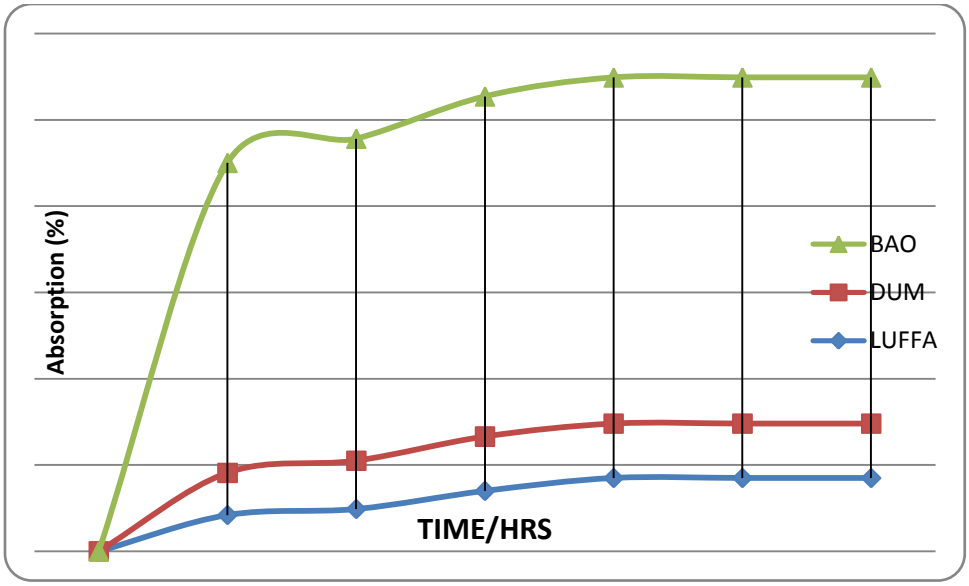



Figure 4.8a: % Water Absorption of the Untreated Composites

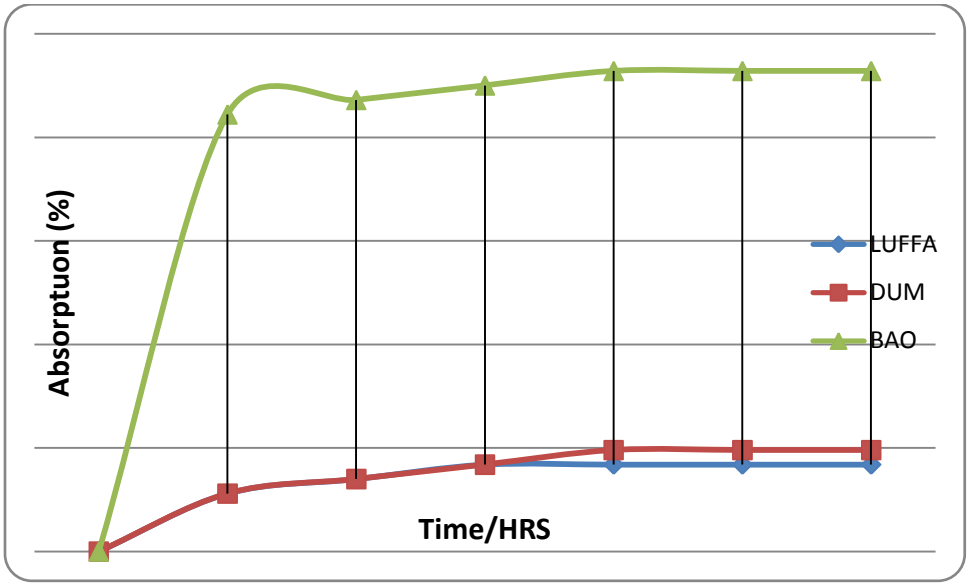
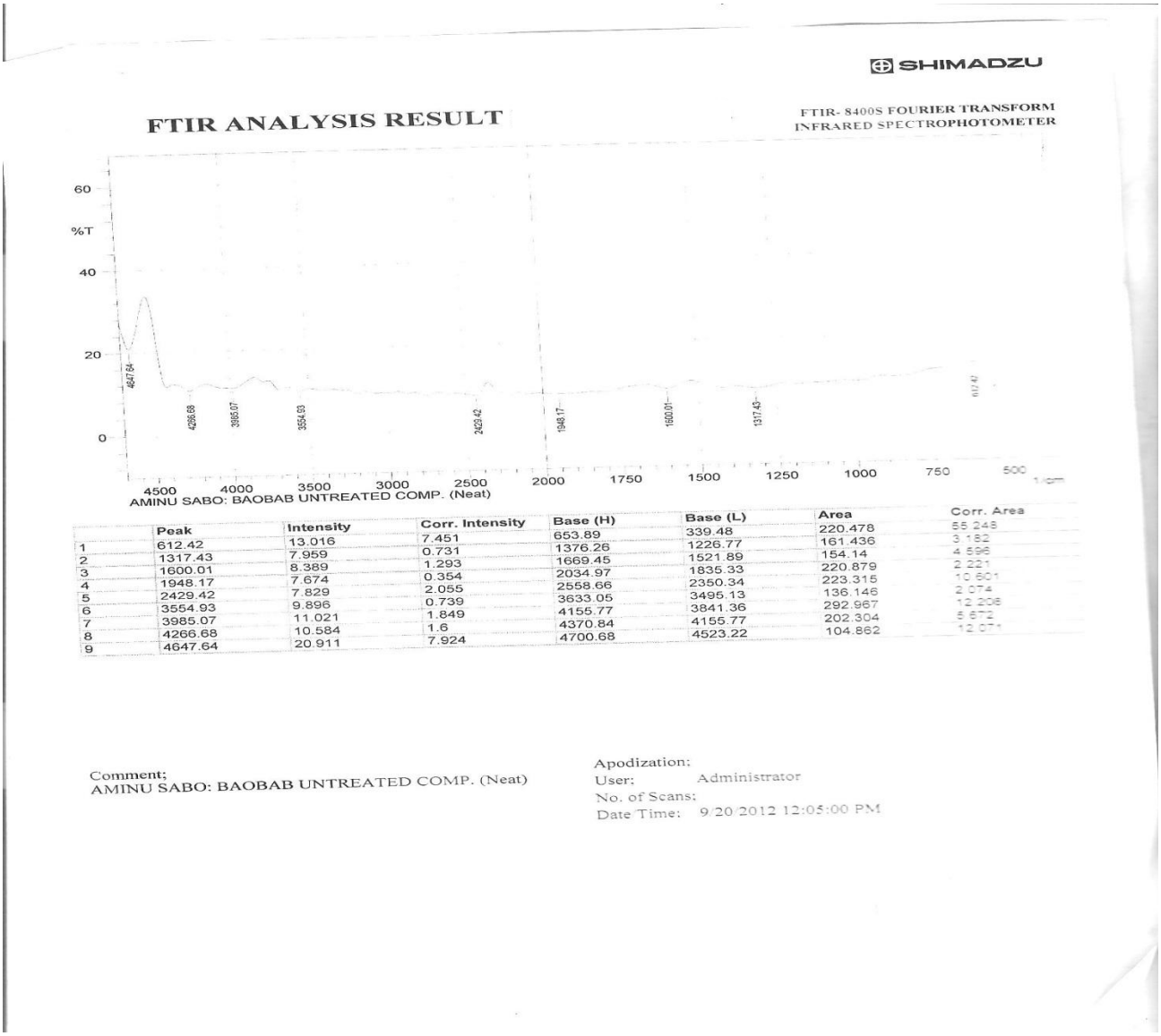



Figure 4.8b: % Water Absorption of the treated Composites

## 4.10 Fourier Transform Infrared Spectroscopy of the Composite




| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 612.42 | 13.016 | 7.451 | 653.89 | 339.48 | 220.478 | 55.248 |
| 2 | 1317.43 | 7.959 | 0.731 | 1376.26 | 1226.77 | 161.436 | 3.182 |
| 3 | 1600.01 | 8.389 | 1.293 | 1669.45 | 1521.89 | 154.14 | 4.596 |
| 4 | 1948.17 | 7.674 | 0.354 | 2034.97 | 1835.33 | 220.879 | 2.221 |
| 5 | 2429.42 | 7.829 | 2.055 | 2558.66 | 2350.34 | 223.315 | 10.601 |
| 6 | 3554.93 | 9.896 | 0.739 | 3633.05 | 3495.13 | 136.146 | 2.074 |
| 7 | 3985.07 | 11.021 | 1.849 | 4155.77 | 3841.36 | 292.967 | 12.206 |
| 8 | 4266.68 | 10.584 | 1.6 | 4370.84 | 4155.77 | 202.304 | 5.872 |
| 9 | 4647.64 | 20.911 | 7.924 | 4700.68 | 4523.22 | 104.862 | 12.071 |

Comment;
AMINU SABO: BAOBAB UNTREATED COMP. (Neat)

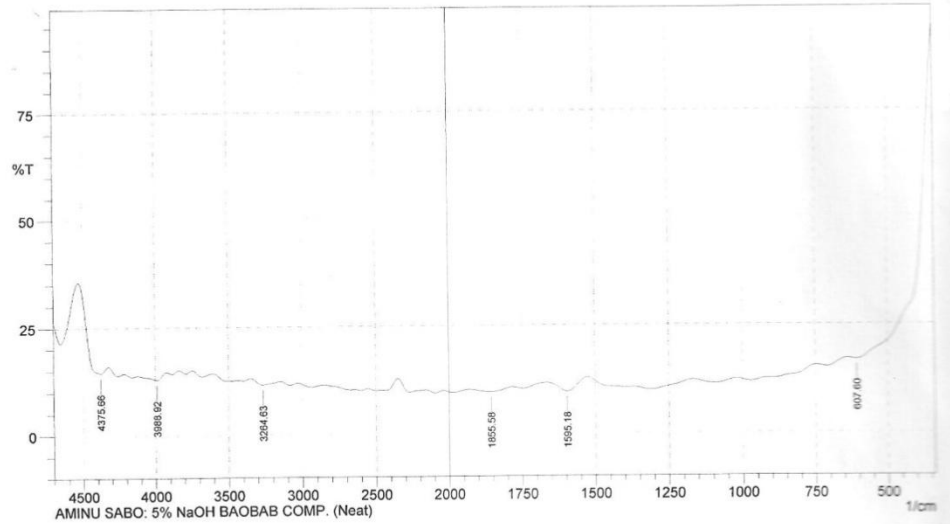
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Figure 4.9a: FTIR of Untreated Baobab Fibre Composite

SHIMADZU

## FTIR ANALYSIS RESULT

FTIR- 8400S FOURIER TRANSFORM
INFRARED SPECTROPHOTOMETER




| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 607.6 | 16.703 | 8.549 | 639.42 | 339.48 | 175.492 | 56.647 |
| 2 | 1595.18 | 9.541 | 2.609 | 1666.55 | 1525.74 | 135.813 | 6.806 |
| 3 | 1855.58 | 9.581 | 0.779 | 1930.81 | 1782.29 | 149.167 | 2.891 |
| 4 | 3264.63 | 11.555 | 1.139 | 3349.5 | 3146.97 | 186.274 | 4.264 |
| 5 | 3988.92 | 12.834 | 1.516 | 4122.98 | 3922.38 | 174.673 | 4.58 |
| 6 | 4375.66 | 14.419 | 6.7 | 4527.08 | 4321.66 | 147.402 | 19.441 |

Comment;
AMINU SABO: 5% NaOH BAOBAB COMP. (Neat)

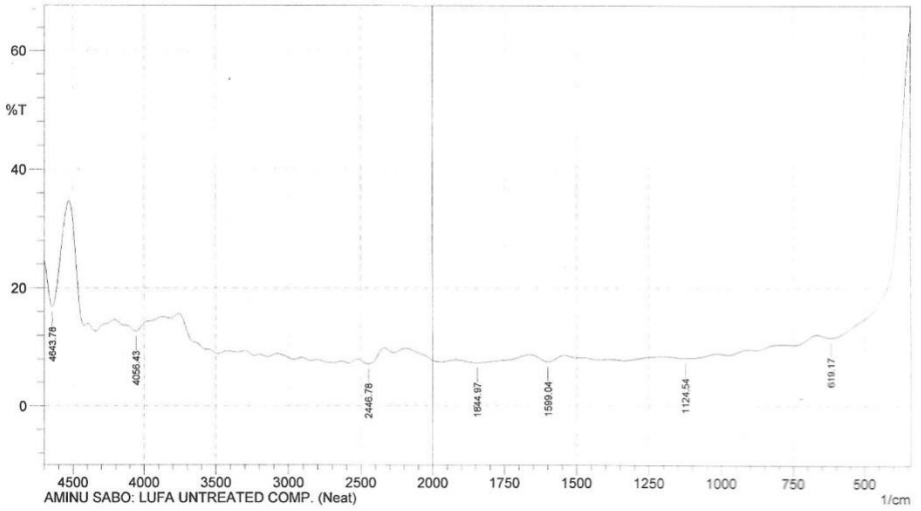
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Figure 4.9b: FTIR of 5% NaOH Treated Baobab Composites

SHIMADZU

## FTIR ANALYSIS RESULT

FTIR- 8400S FOURIER TRANSFORM
INFRARED SPECTROPHOTOMETER




AMINU SABO: LUFA UNTREATED COMP. (Neat)

| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 619.17 | 11.642 | 8.365 | 668.36 | 339.48 | 248.352 | 66.724 |
| 2 | 1124.54 | 8.069 | 0.505 | 1205.55 | 1014.59 | 206.336 | 2.945 |
| 3 | 1599.04 | 7.476 | 1.118 | 1662.69 | 1543.1 | 130.842 | 3.409 |
| 4 | 1844.97 | 7.319 | 0.723 | 1919.24 | 1662.69 | 285.206 | 6.779 |
| 5 | 2446.78 | 7.165 | 1.576 | 2523.94 | 2337.8 | 204.754 | 8.76 |
| 6 | 4056.43 | 12.74 | 1.432 | 4126.84 | 3870.3 | 219.154 | 3.812 |
| 7 | 4643.78 | 16.851 | 11.187 | 4700.68 | 4527.08 | 110.085 | 17.589 |

Comment;
AMINU SABO: LUFA UNTREATED COMP. (Neat)

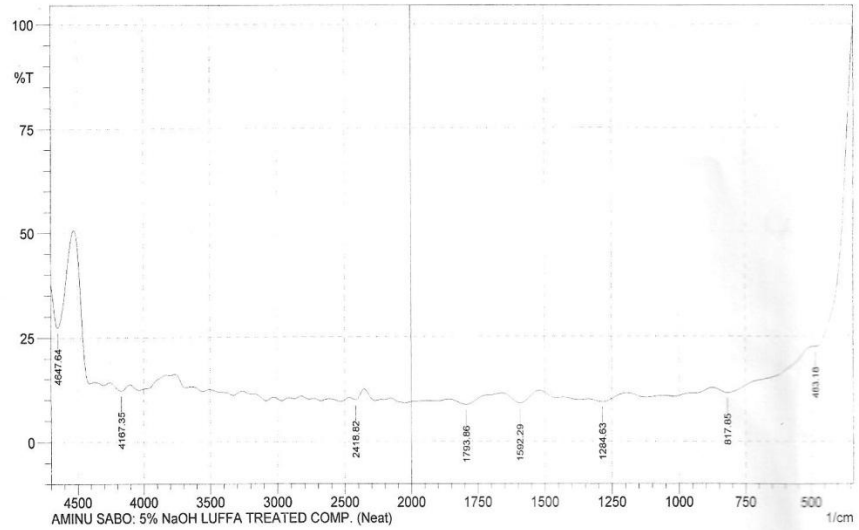
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Figure 4.9c: FTIR of untreated Luffa Gourd Fibre Composites

SHIMADZU

## FTIR ANALYSIS RESULT

FTIR- 8400S FOURIER TRANSFORM
INFRARED SPECTROPHOTOMETER




AMINU SABO: 5% NaOH LUFFA TREATED COMP. (Neat)

| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 483.18 | 22.452 | 4.431 | 491.86 | 339.48 | 65.362 | 15.954 |
| 2 | 817.85 | 11.453 | 2.664 | 874.75 | 491.86 | 315.517 | 19.536 |
| 3 | 1284.63 | 9.257 | 1.167 | 1336.71 | 1193.98 | 142.434 | 3.31 |
| 4 | 1592.29 | 8.981 | 2.657 | 1659.8 | 1518.99 | 138.968 | 7.484 |
| 5 | 1793.86 | 8.652 | 1.734 | 1858.48 | 1659.8 | 199.091 | 5.522 |
| 6 | 2418.82 | 9.851 | 1.359 | 2465.11 | 2353.23 | 108.883 | 3.122 |
| 7 | 4167.35 | 12.128 | 1.707 | 4256.07 | 4102.73 | 136.105 | 4.575 |
| 8 | 4647.64 | 27.358 | 14.153 | 4700.68 | 4526.12 | 78.193 | 15.238 |

Comment;
AMINU SABO: 5% NaOH LUFFA TREATED COMP. (Neat)

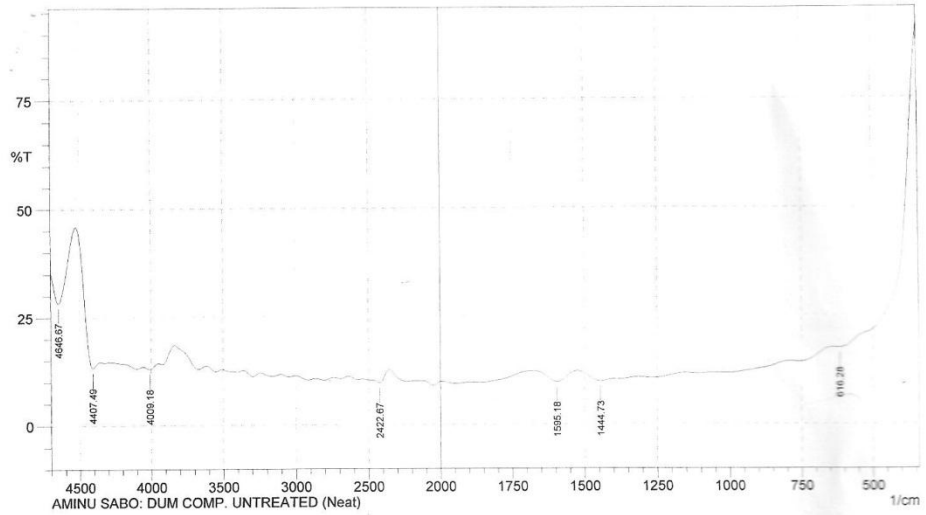
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Figure 4.9d: FTIR of 5% NaOH Treated Luffa Gourd Fibre Composites

SHIMADZU

## FTIR ANALYSIS RESULT

FTIR- 8400S FOURIER TRANSFORM
INFRARED SPECTROPHOTOMETER




| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 616.28 | 17.354 | 5.596 | 638.46 | 339.48 | 170.136 | 51.202 |
| 2 | 1444.73 | 9.822 | 1.254 | 1524.78 | 1387.83 | 133.696 | 3.45 |
| 3 | 1595.18 | 9.689 | 2.453 | 1680.05 | 1524.78 | 148.84 | 6.659 |
| 4 | 2422.67 | 9.613 | 1.665 | 2473.79 | 2353.23 | 117.627 | 3.925 |
| 5 | 4009.18 | 12.944 | 0.882 | 4054.51 | 3947.45 | 93.268 | 1.46 |
| 6 | 4407.49 | 13.252 | 11.061 | 4523.22 | 4355.41 | 110.705 | 12.201 |
| 7 | 4646.67 | 28.384 | 10.186 | 4700.68 | 4523.22 | 81.36 | 11.303 |

Comment;
AMINU SABO: DUM COMP. UNTREATED (Neat)

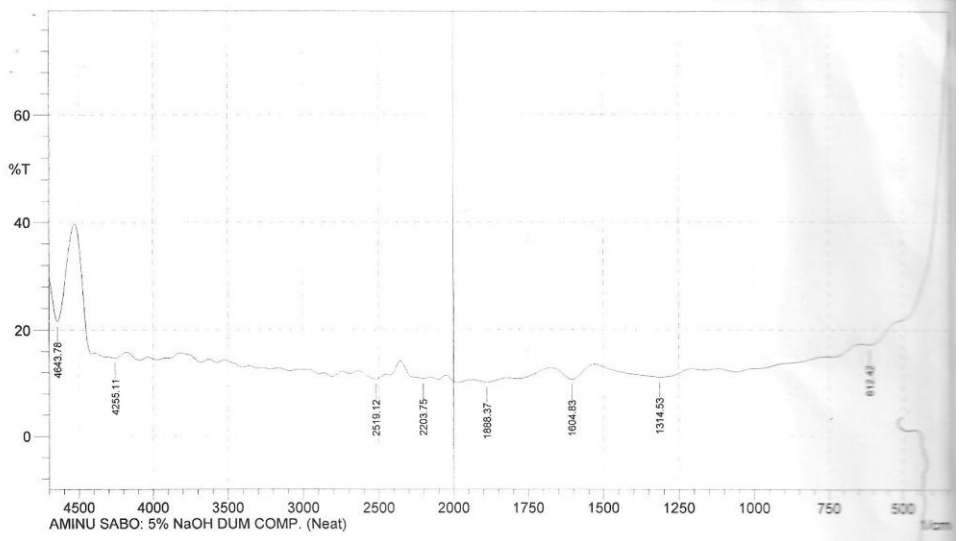
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Figure 4.9e FTIR of untreated Dumpalm fibre composites

SHIMADZU

**FTIR ANALYSIS RESULT**

FTIR- 8400S FOURIER TRANSFORM
INFRARED SPECTROPHOTOMETER




| | Peak | Intensity | Corr. Intensity | Base (H) | Base (L) | Area | Corr. Area |
|---|---|---|---|---|---|---|---|
| 1 | 612.42 | 17.293 | 5.797 | 641.35 | 339.48 | 175.513 | 43.32 |
| 2 | 1314.53 | 11.063 | 1.824 | 1528.64 | 1206.51 | 298.538 | 13.416 |
| 3 | 1604.83 | 10.613 | 2.513 | 1673.3 | 1528.64 | 133.635 | 6.125 |
| 4 | 1888.37 | 10.113 | 0.6 | 1939.49 | 1820.86 | 116.416 | 1.428 |
| 5 | 2203.75 | 10.798 | 1.029 | 2355.16 | 2152.63 | 190.171 | 7.271 |
| 6 | 2519.12 | 10.721 | 1.093 | 2635.81 | 2449.68 | 176.319 | 4.143 |
| 7 | 4255.11 | 14.697 | 0.636 | 4312.01 | 4178.92 | 109.442 | 1.272 |
| 8 | 4643.78 | 21.654 | 11.458 | 4700.68 | 4526.12 | 95.486 | 14.735 |

Comment;
AMINU SABO: 5% NaOH DUM COMP. (Neat)

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Figure 4.9f: FTIR of 5% NaOH treated Dumpalm Fibre Composites

## 4.11 SCANNING ELECTRON MICROSCOPY OF COMPOSITES

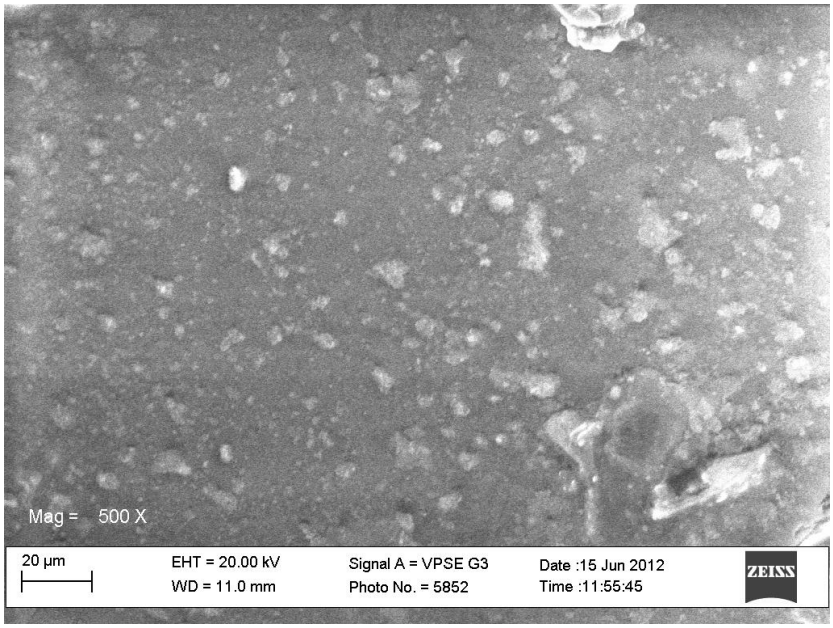



**Figure 4.10a: Untreated Baobab Composite**

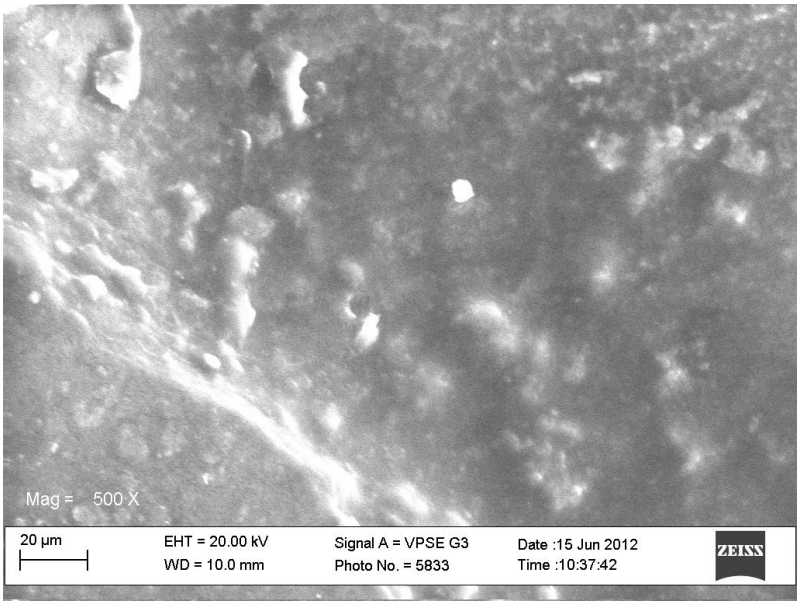



**Figure 4.10b 5% NaOH Treated Baobab composite**

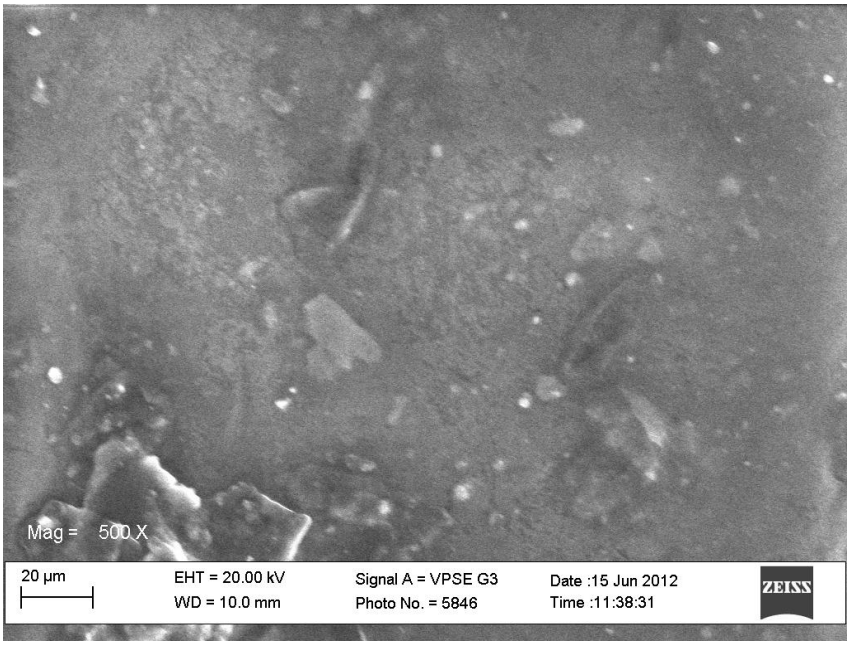



**Figure 4.10c: Untreated Luffa Gourd Fibre Composite**

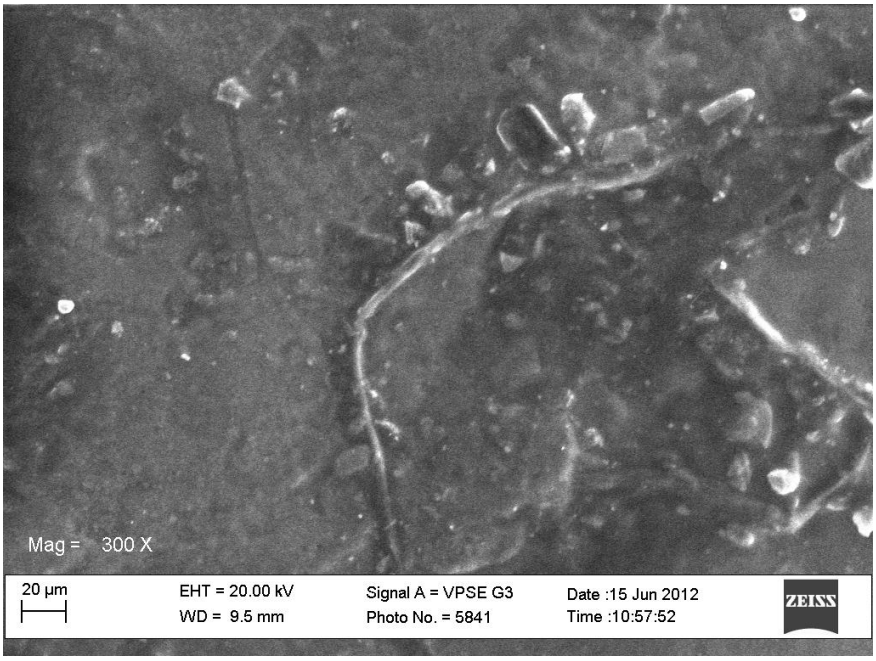



**Figure 4.10d: 5% NaOH Treated Luffa Gourd Fibre Composite**

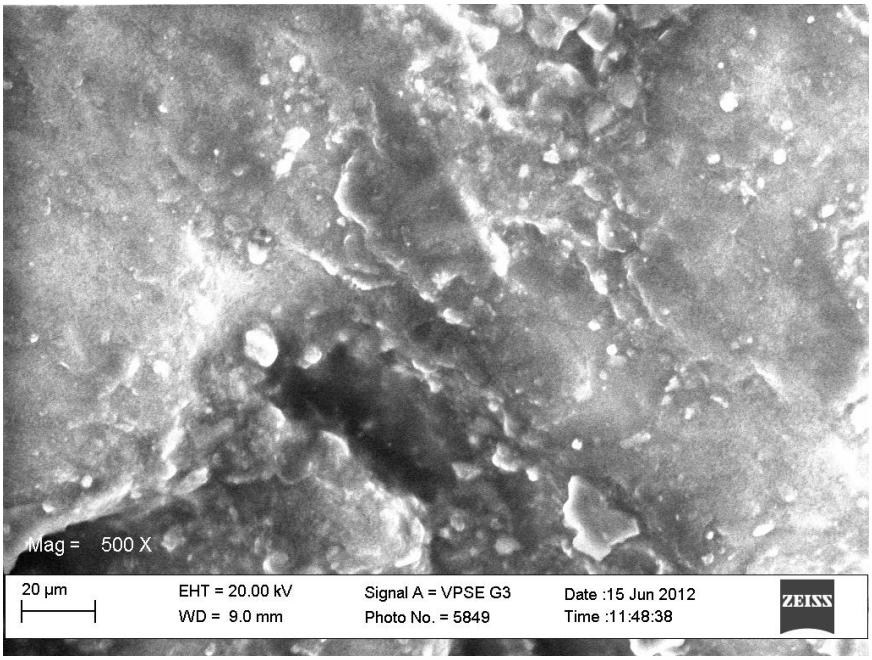



**Figure 4.10e: Untreated Dum palm Fibre Composite**

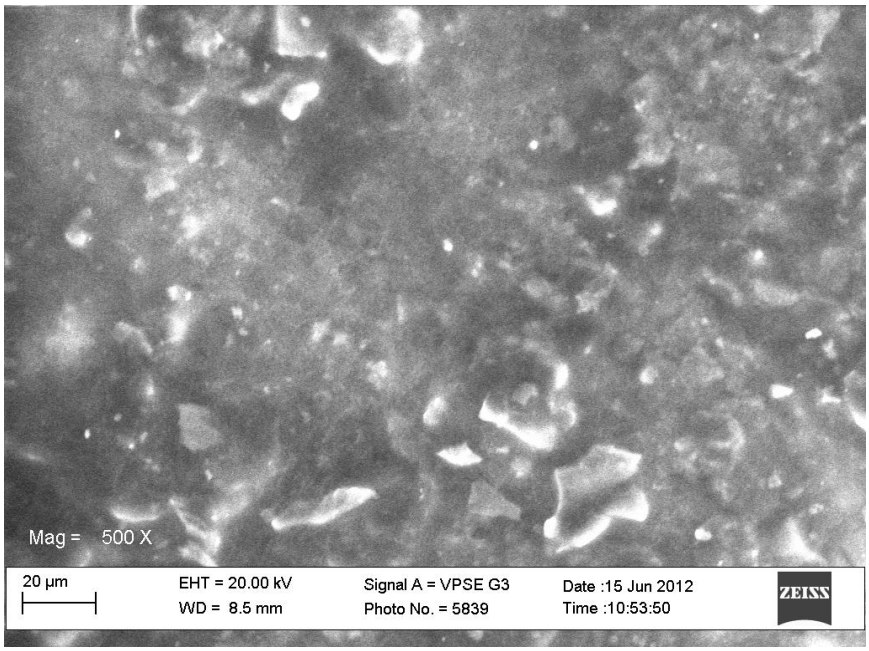



**Figure 4.10f; 5%NaOH Treated Dum palm Fibre Composite**

## 4.12 ATOMIC FORCE MICROSCOPY OF COMPOSITES

The composites were subjected to study using Atomic Force Microscopy

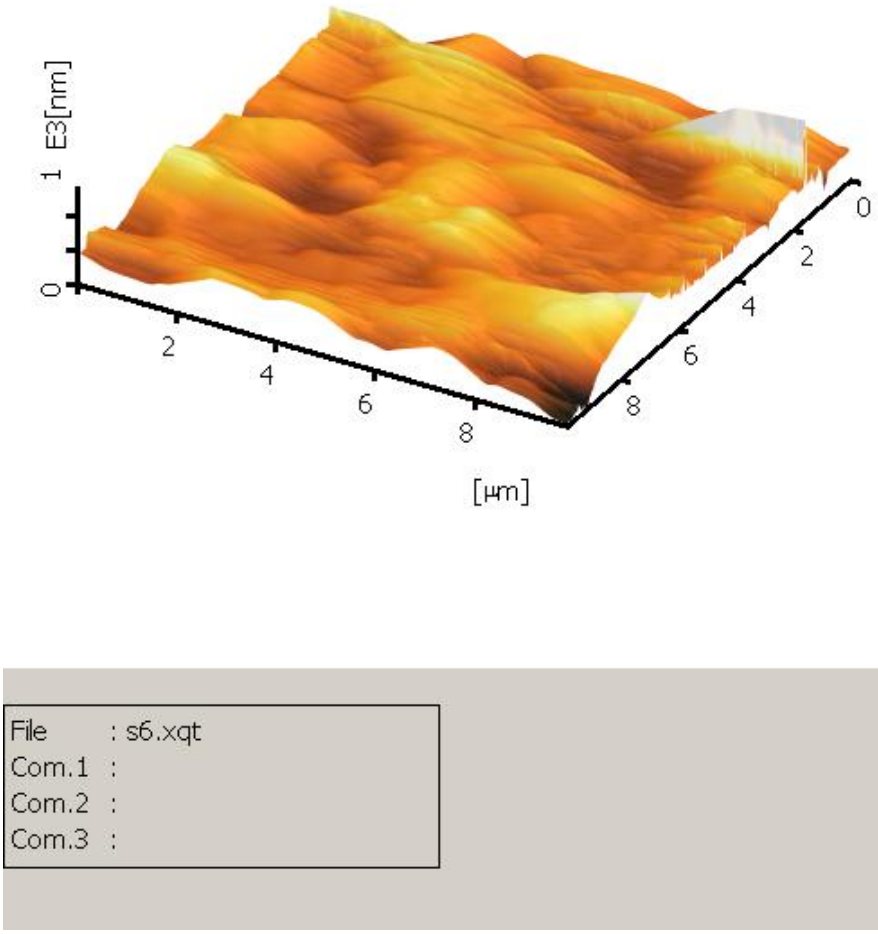
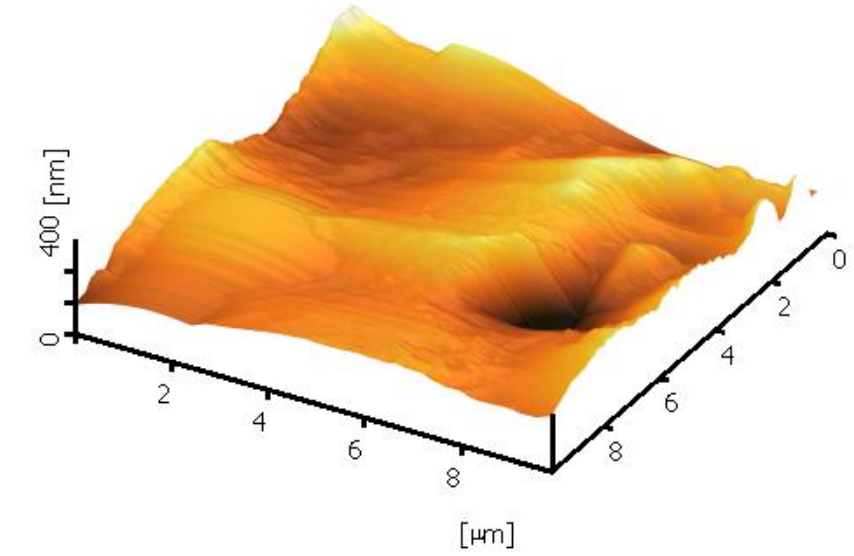
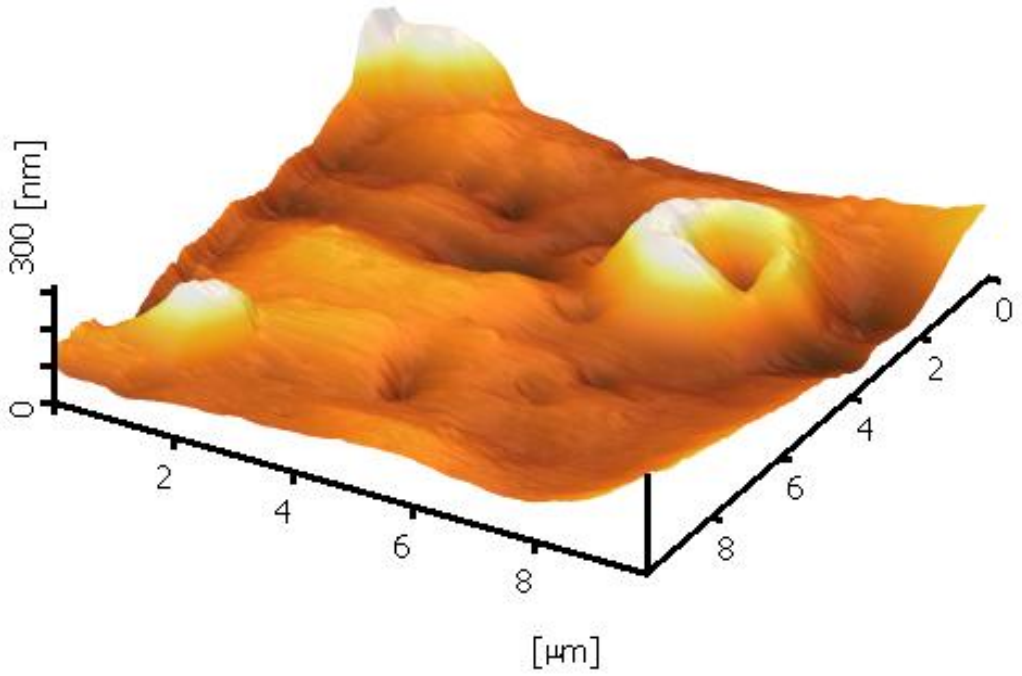



**Figure 4.11a: AFM Scan of Untreated Baobab Fibre Composite**



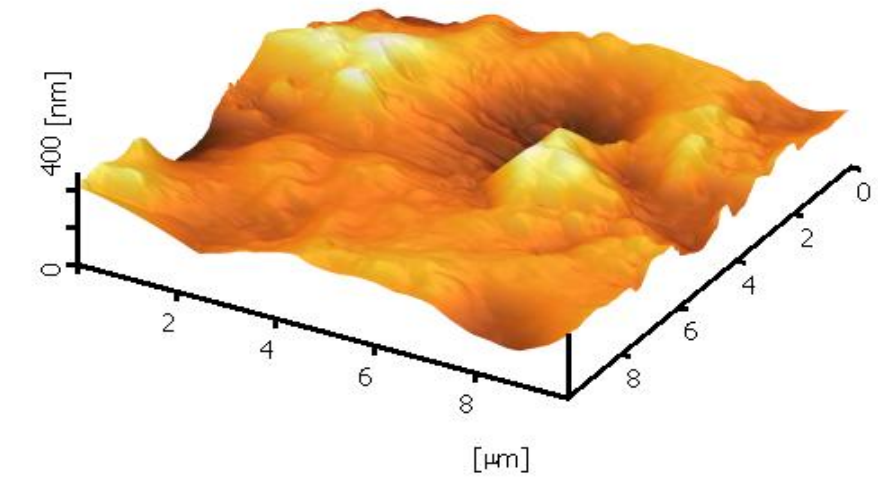

File : s2.xqt
Com.1 :
Com.2 :
Com.3 :

**Figure 4.11b AFM Scan of Treated Baobab Fibre Composite**




File : s4.xqt
Com.1 :
Com.2 :
Com.3 :

**Figure 4.11c: AFM Scan of Untreated Luffa Gourd Fibre Composite**




File : s5.xqt
Com.1 :
Com.2 :
Com.3 :

**Figure 4.11d: AFM Scan of 5% Treated Luffa Gourd Fibre Composite**

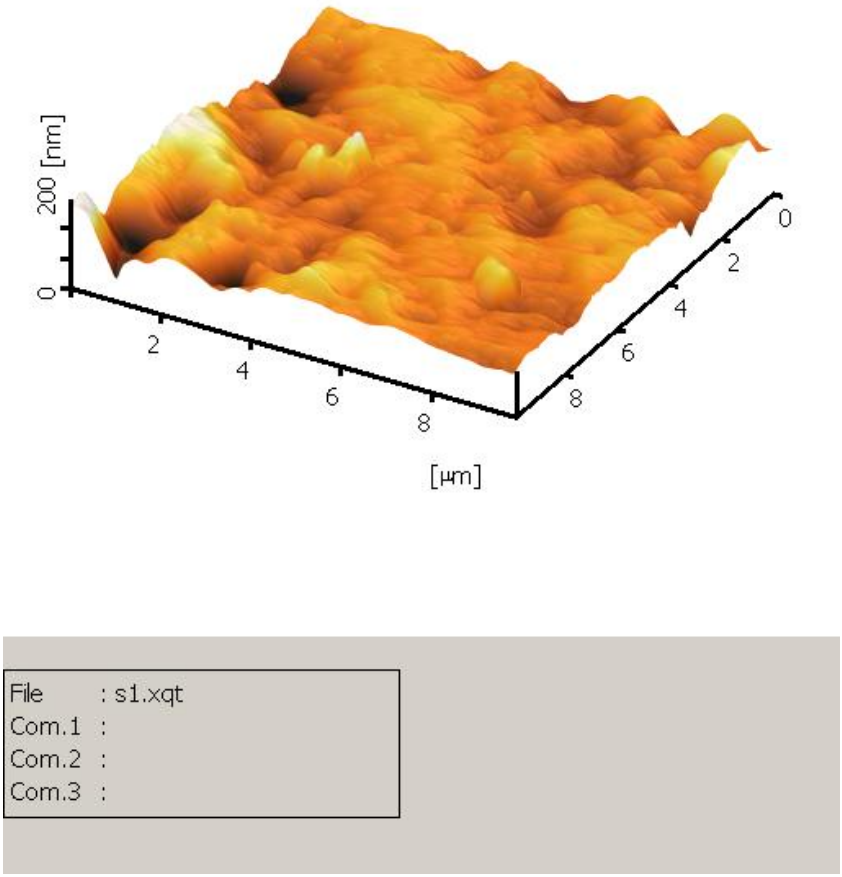
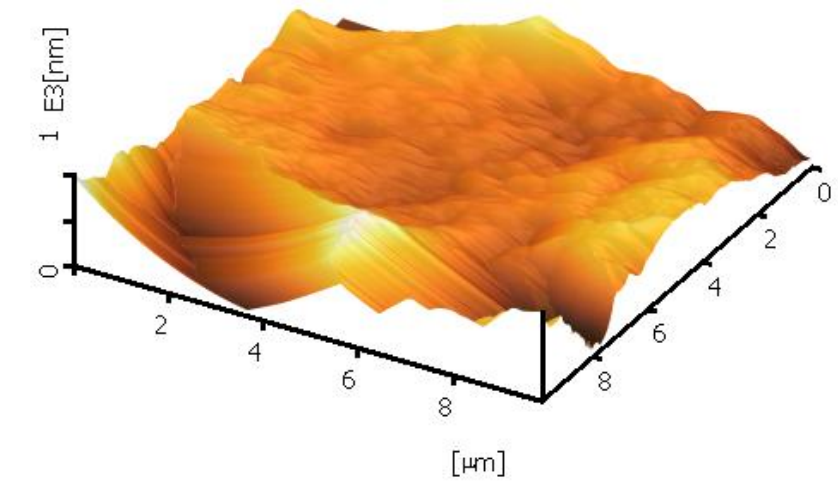



**Figure 4.11e: AFM Scans of Untreated Dum palm Fibre Composite**




File : s3.xqt
Com.1 :
Com.2 :
Com.3 :

**Figure 4.11f: AFM Scan of 5%Treated Dum palm Fibre Composite**

## 4.13 DSC RESULT OF THE COMPOSITES

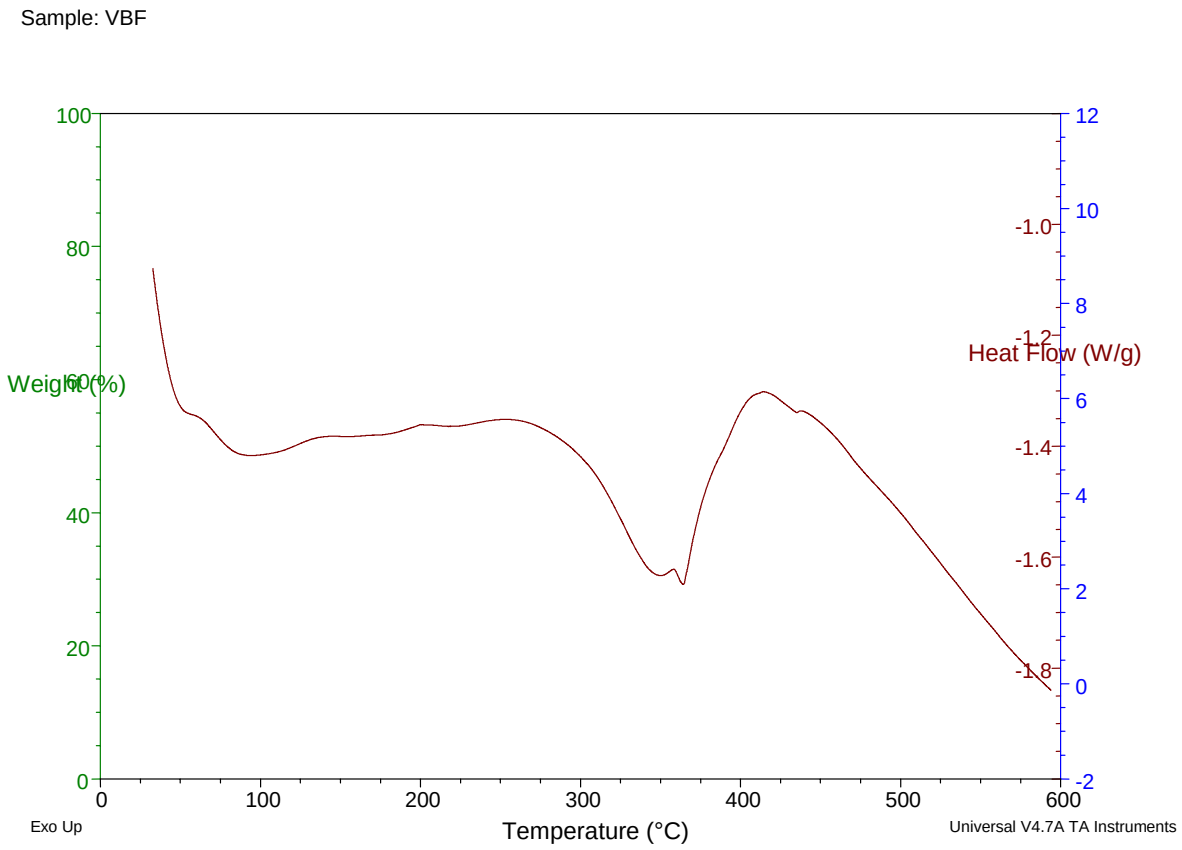



**Figure 4.12a: DSC of Untreated Baobab Fibre Composite**

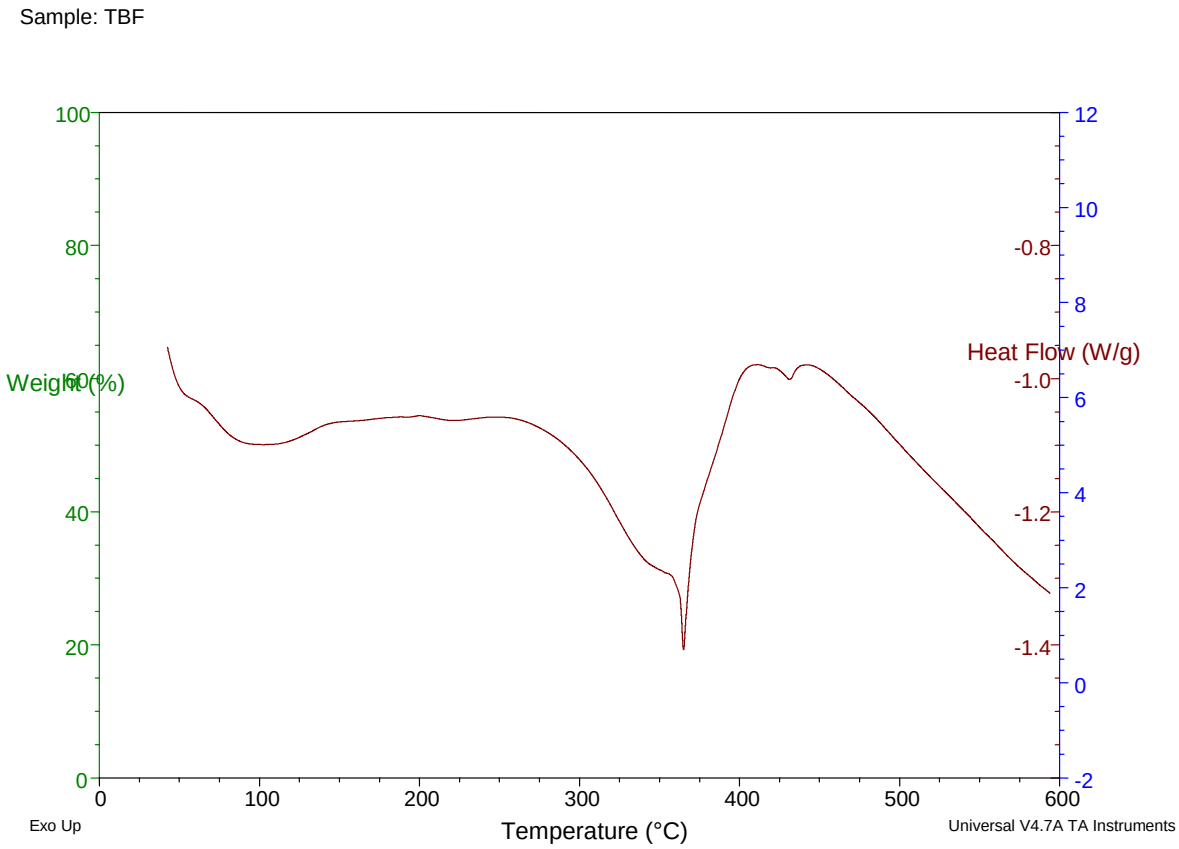



**Figure 4.12b: DSC of Treated Baobab Fibre Composites**

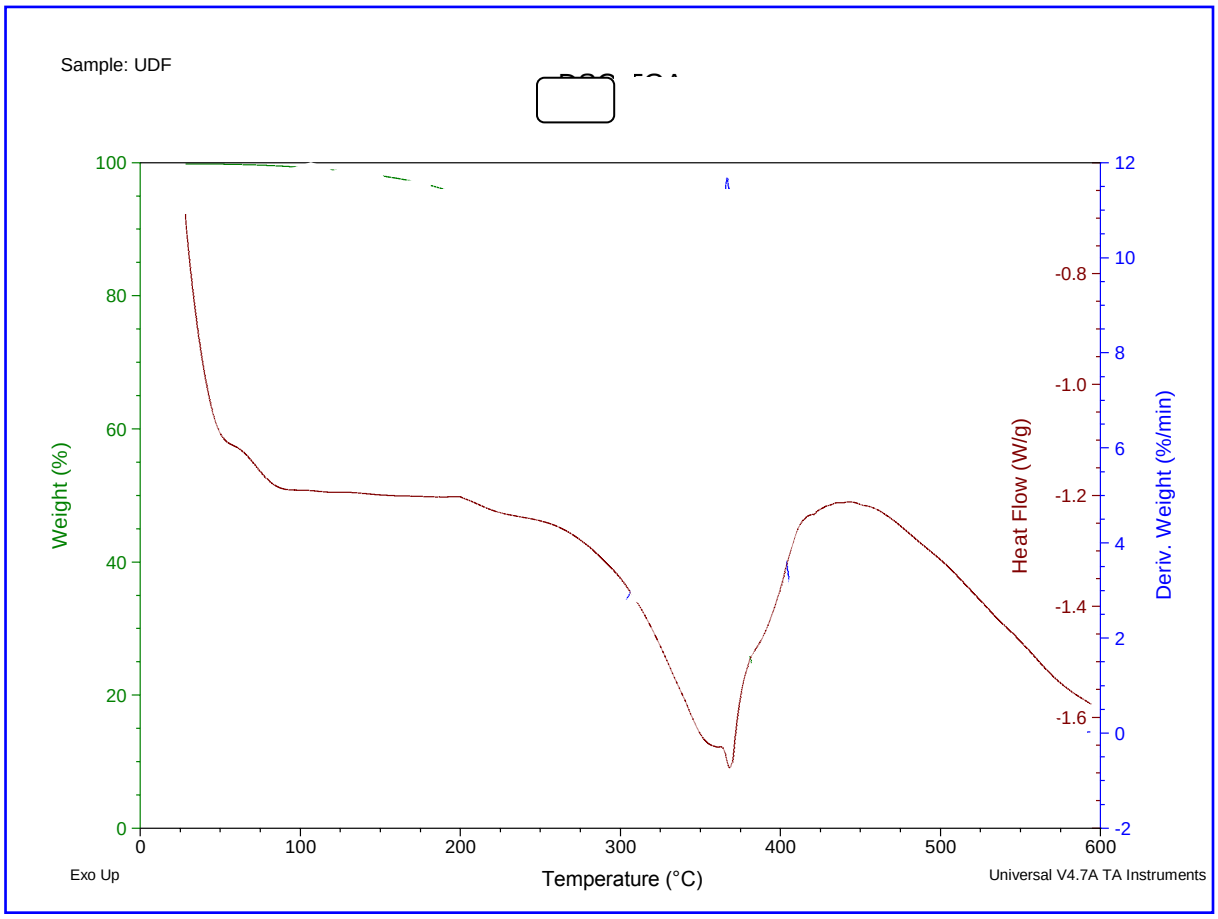



**Figure 4.12c: DSC of Untreated Dum palm Fibre Composites**

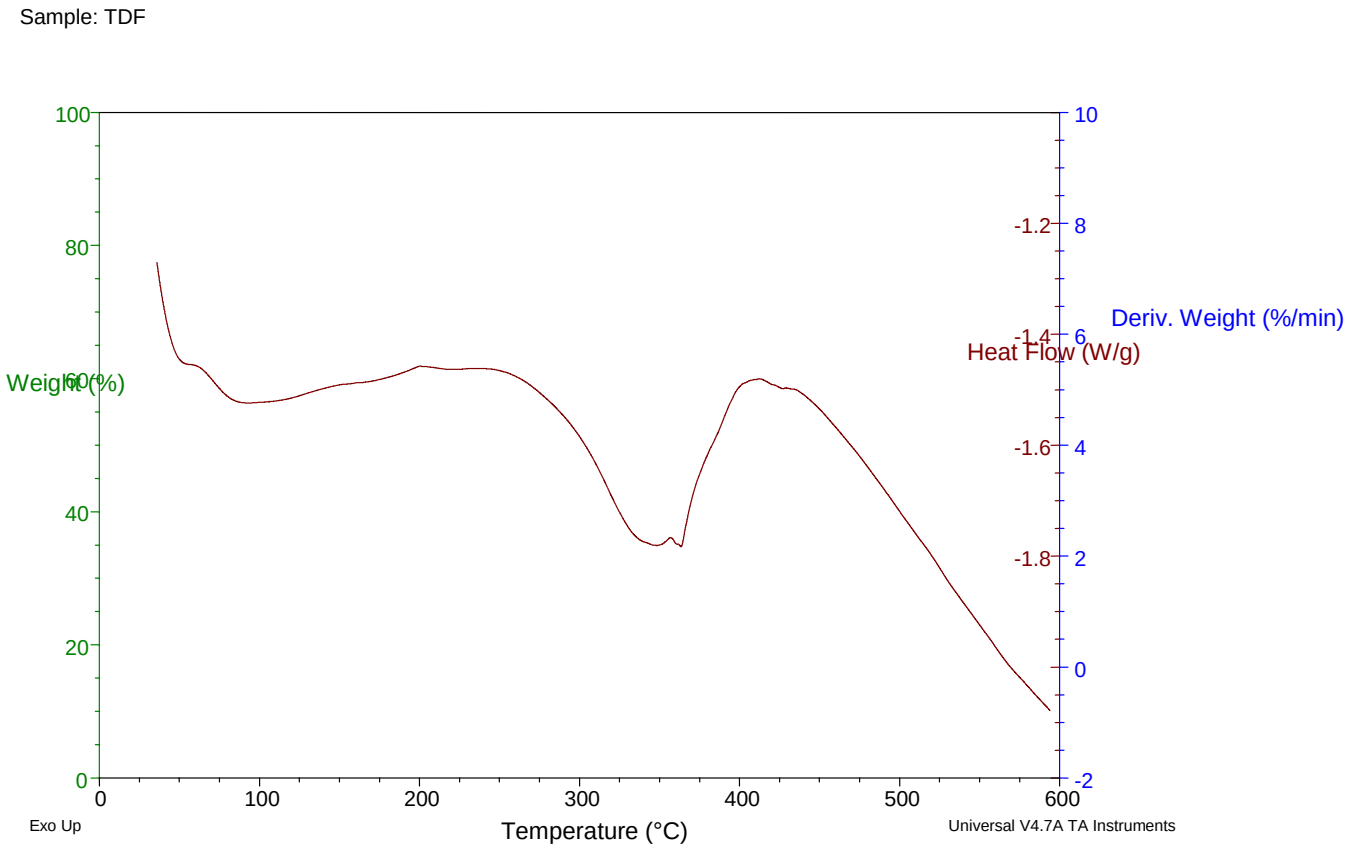



**Figure 4.12d: DSC of Treated Dum palm Fibre Composites**

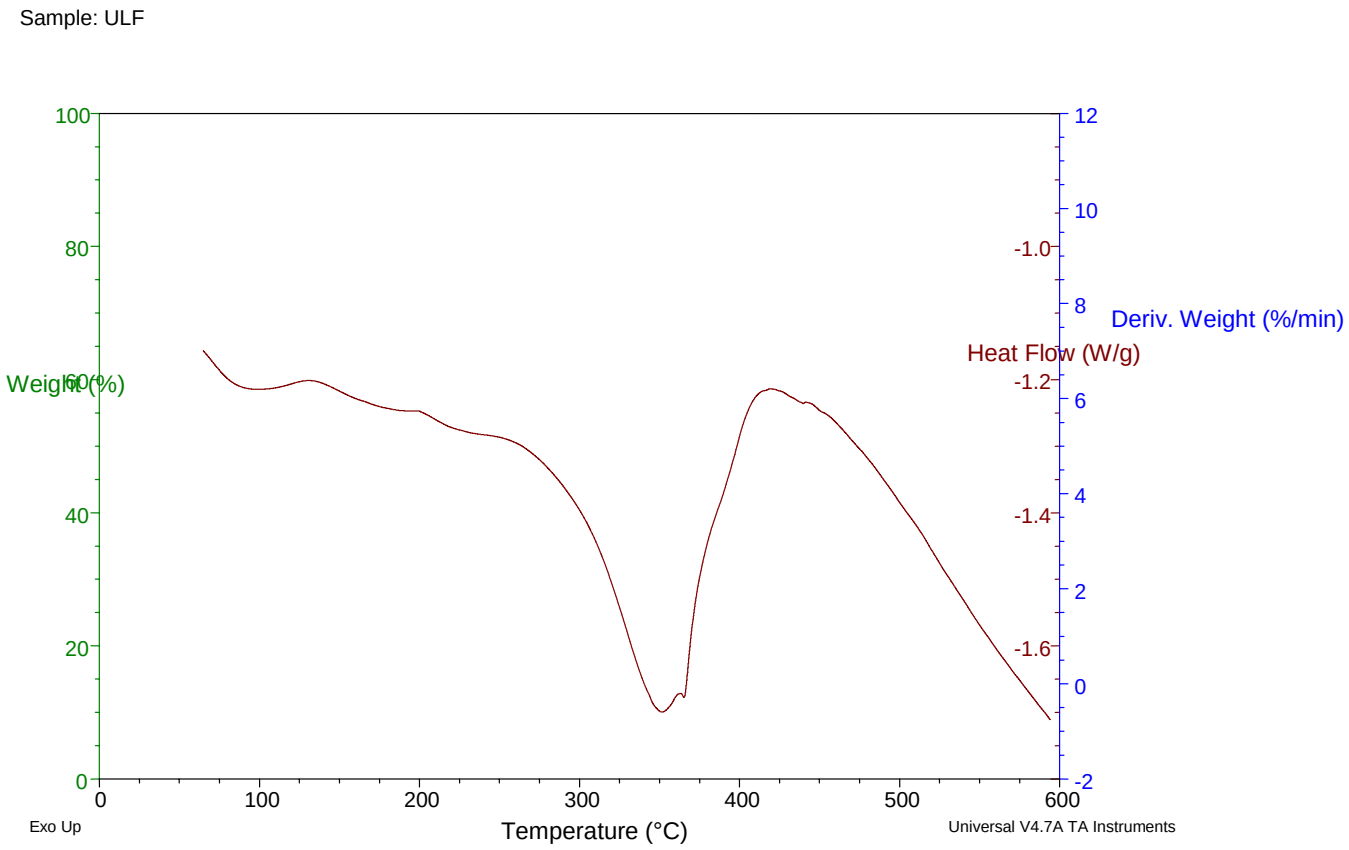



**Figure 4.12e: DSC of Untreated Luffa Gourd Fibre Composites**

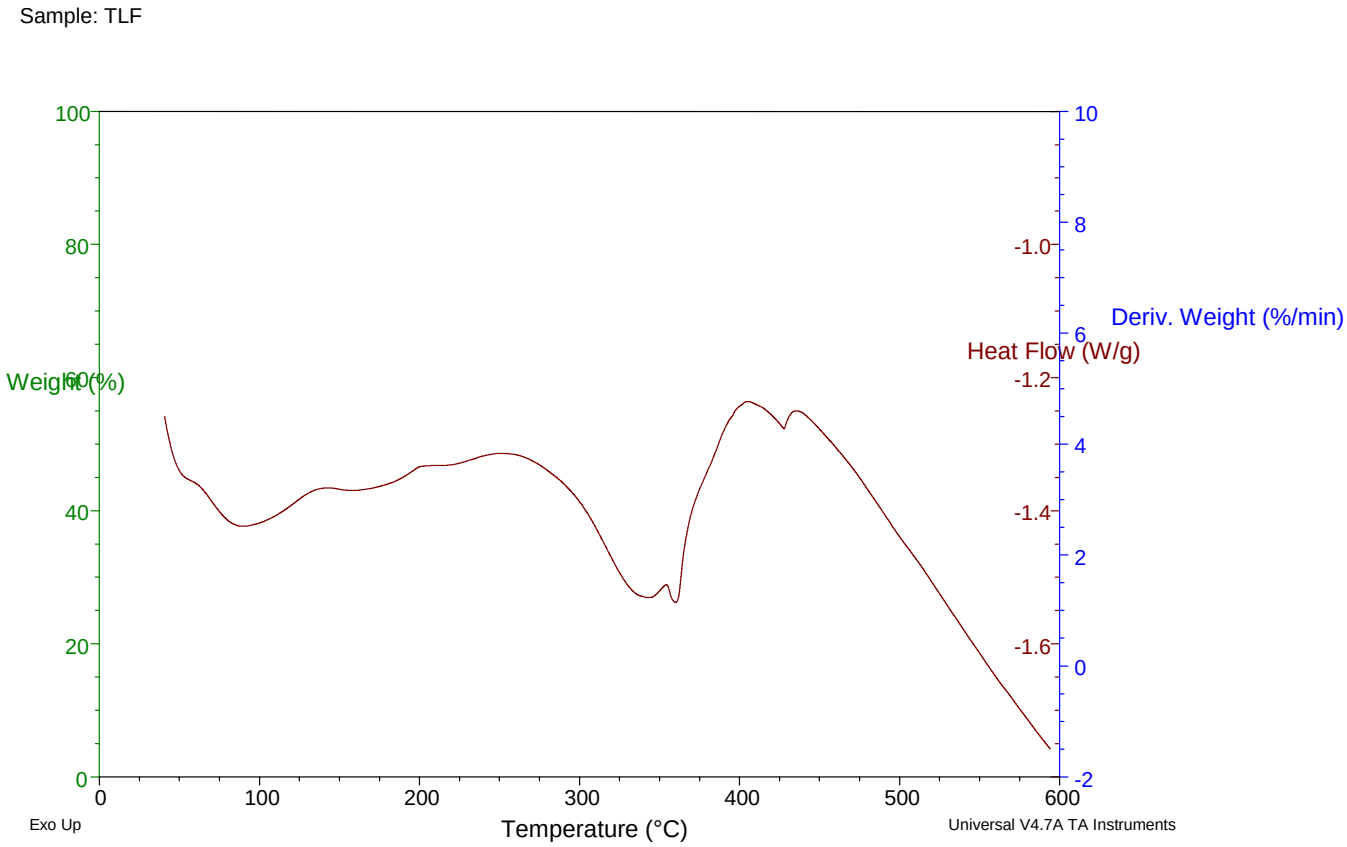



**Figure 4.12f: DSC of Treated Luffa Gourd Fibre Composites**

## 4.14 TGA REULTS OF THE COMPOSITES

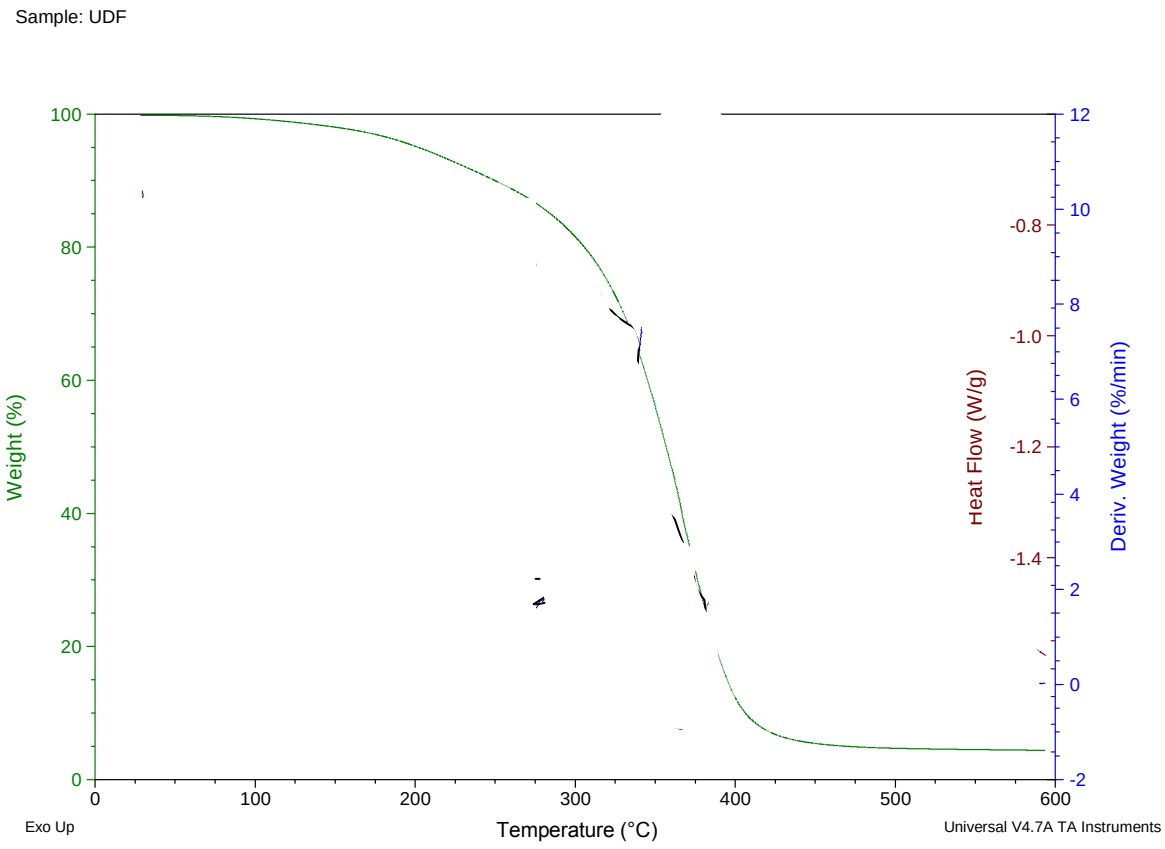



**Figure 4.13a: TGA of Untreated Dum palm Fibre Composites**

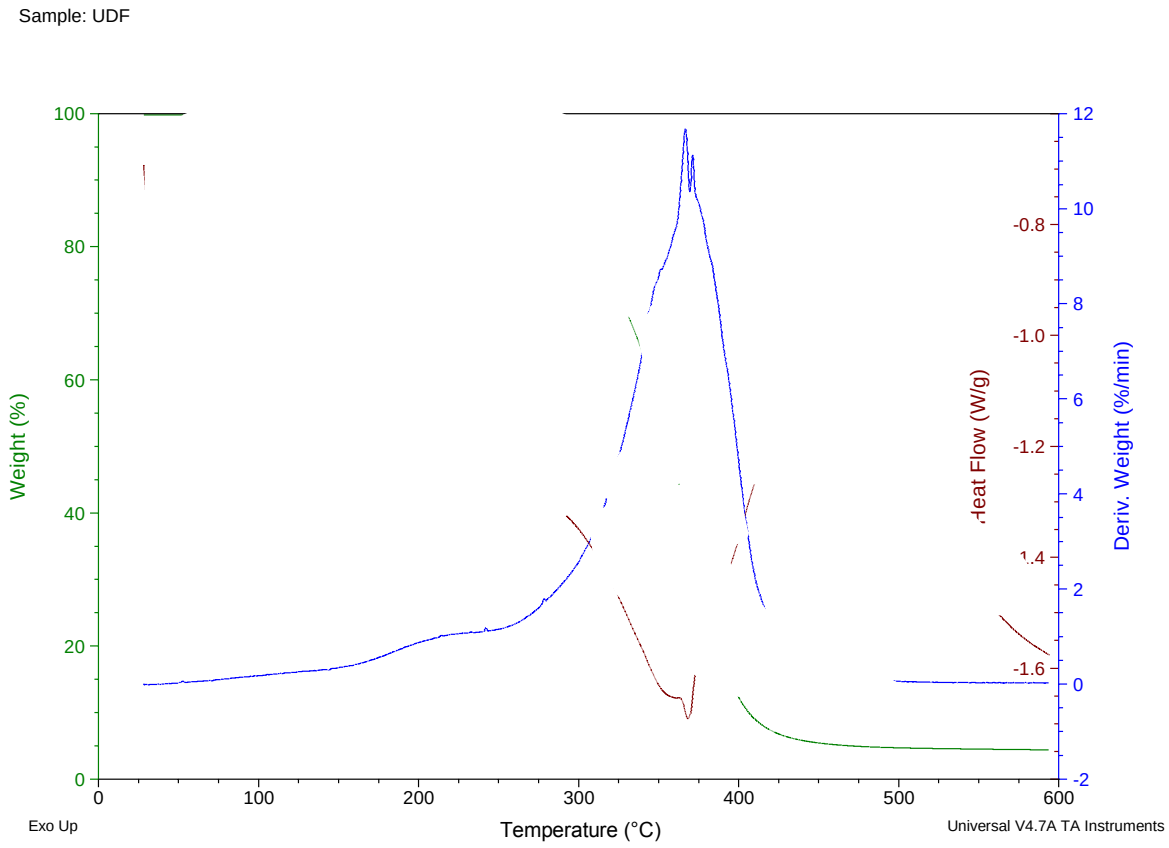



**Figure 4.13aa: TGA of Untreated Dum palm Fibre Composites**

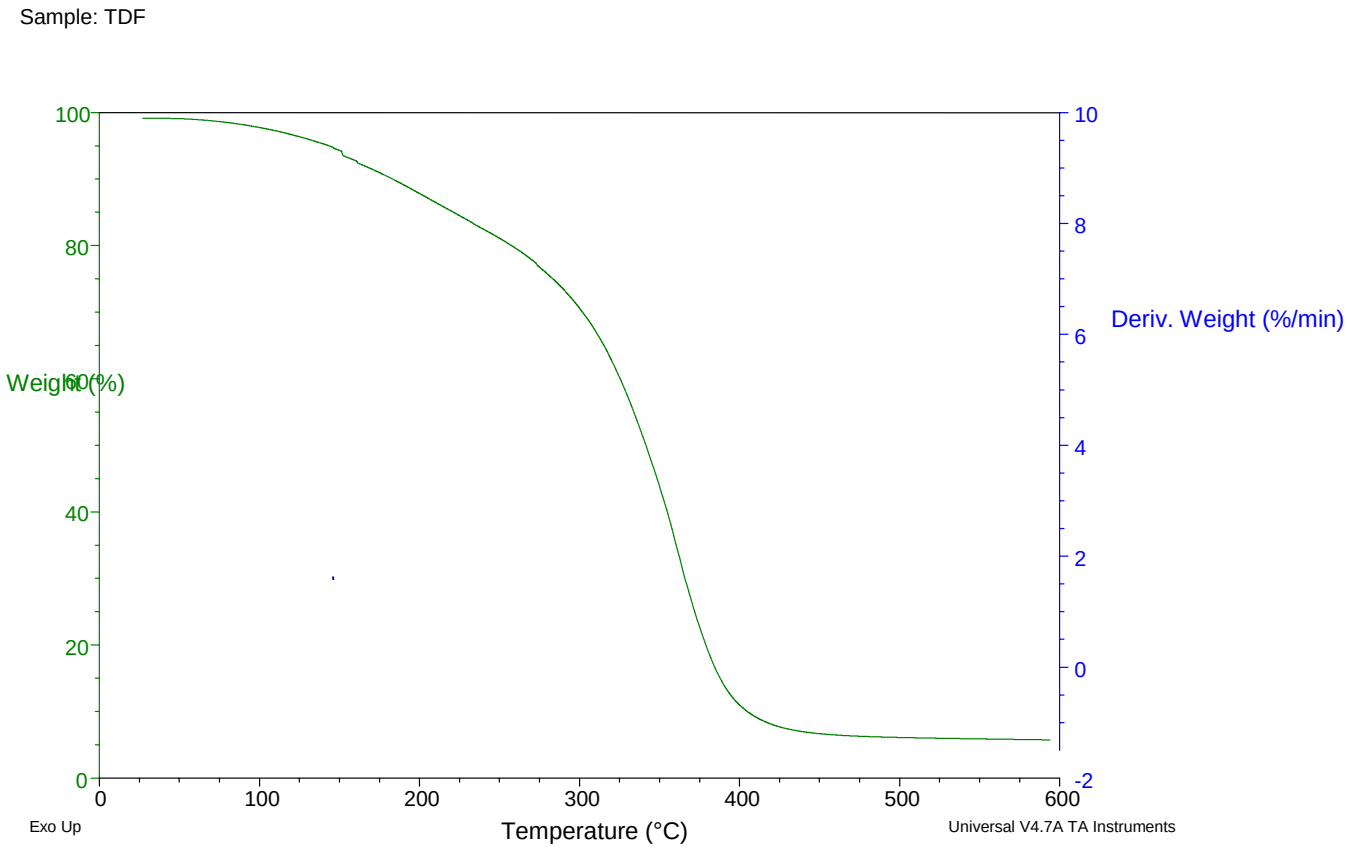



**Figure 4.13b TGA of Treated Dum palm Fibre Composites**

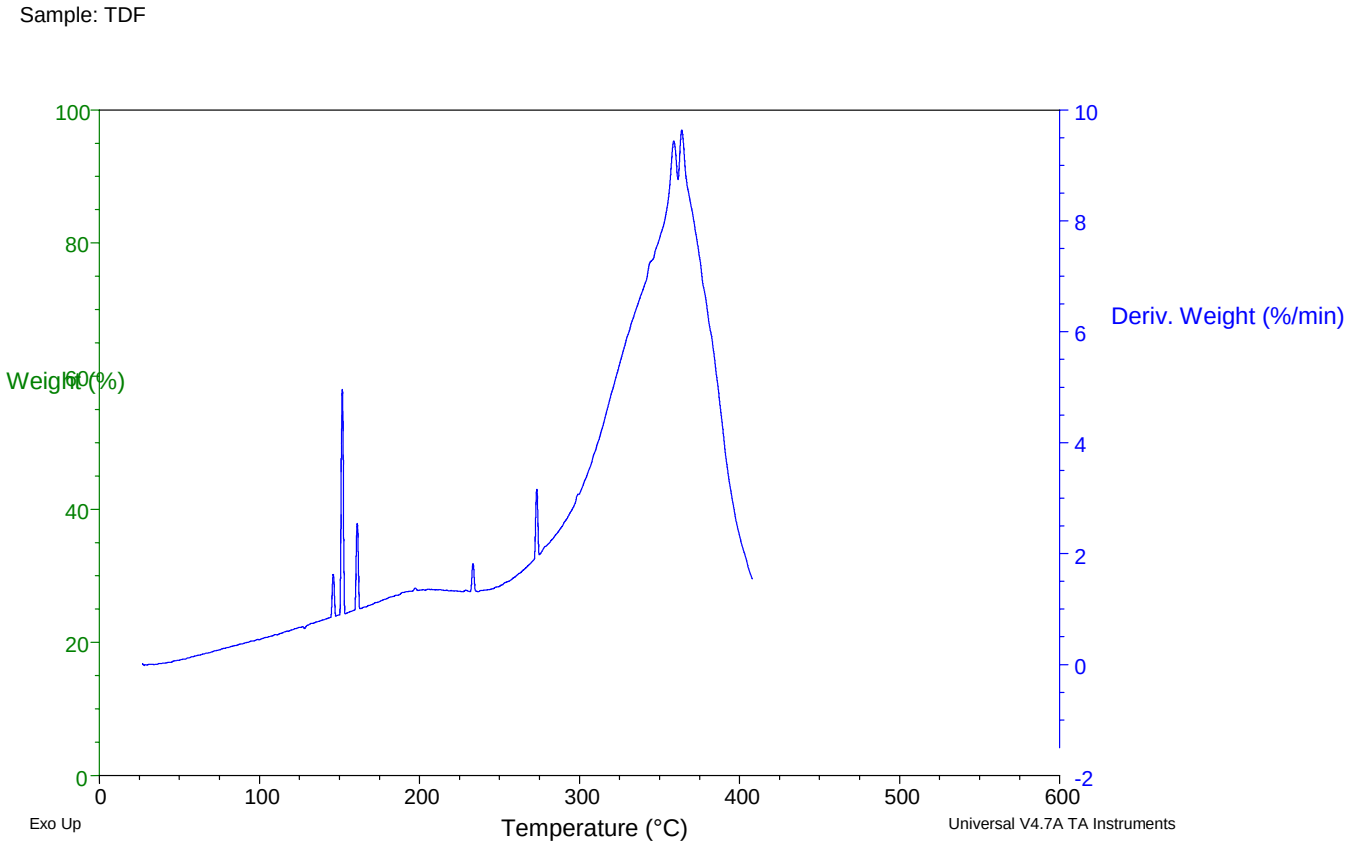



**Figure 4.13 bb: TGA of Treated Dum palm Fibre Composites**

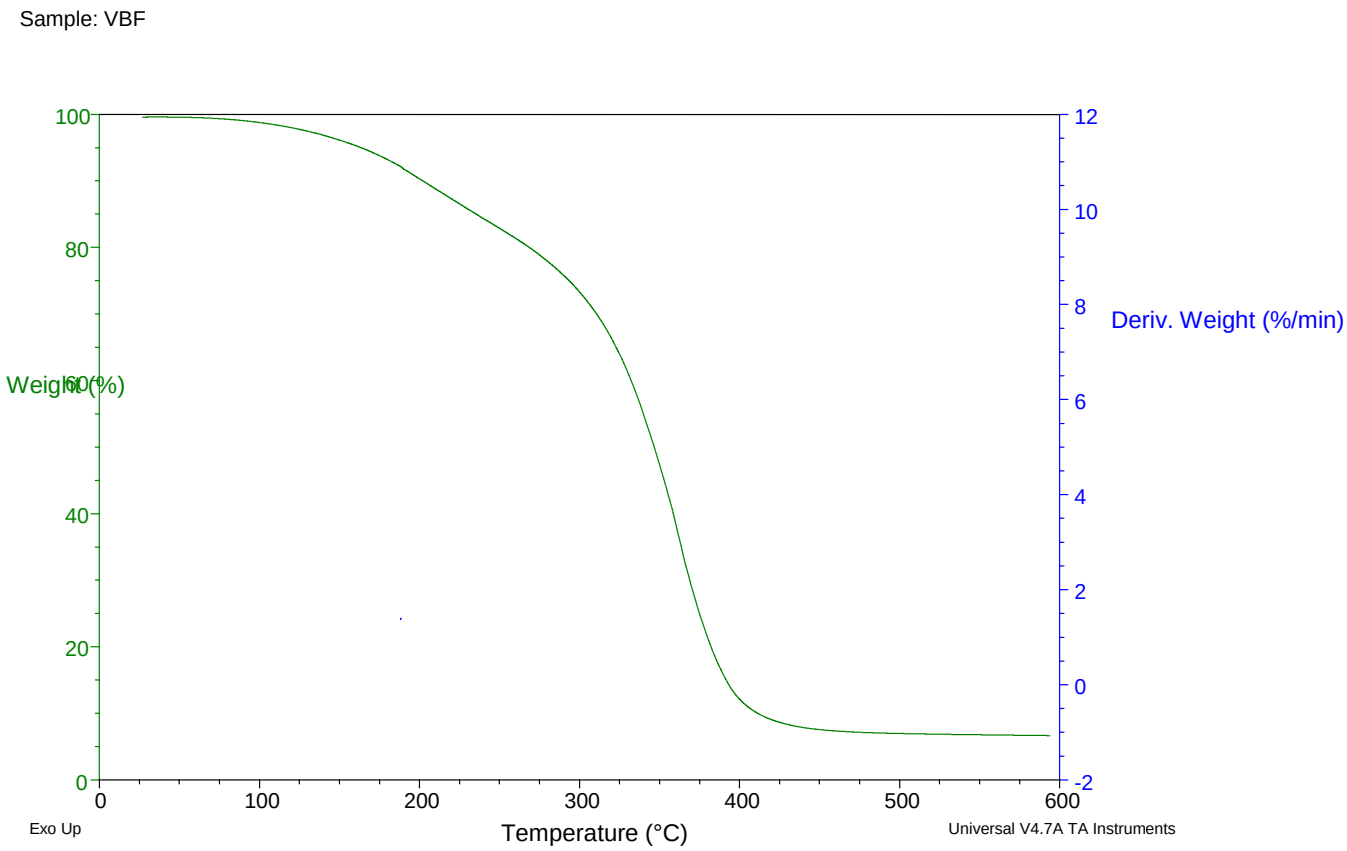



**Figure 4.13c TGA of Untreated Baobab Fibre Composites**

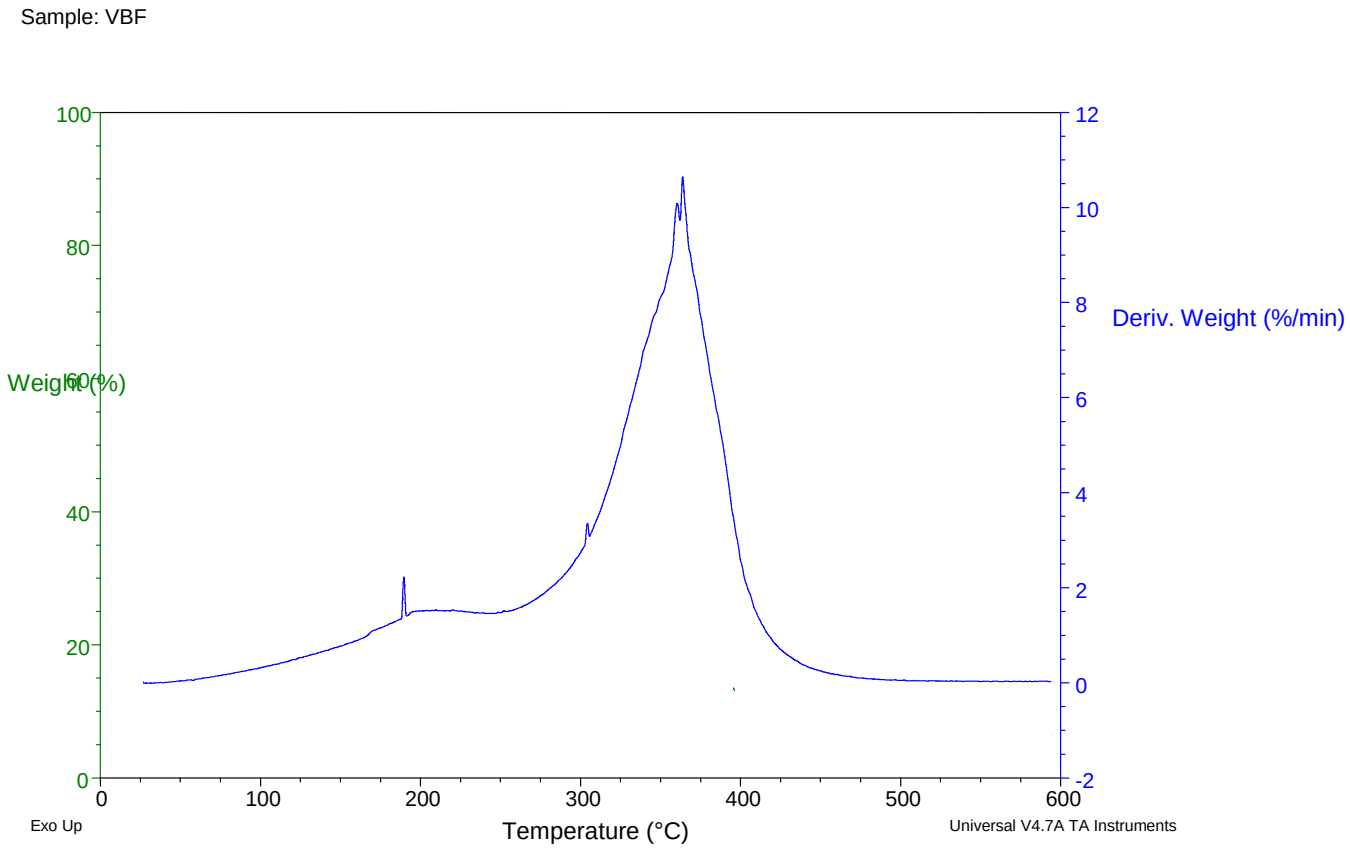



**Figure 4.13cc: TGA of Treated Baobab Fibre Composites**

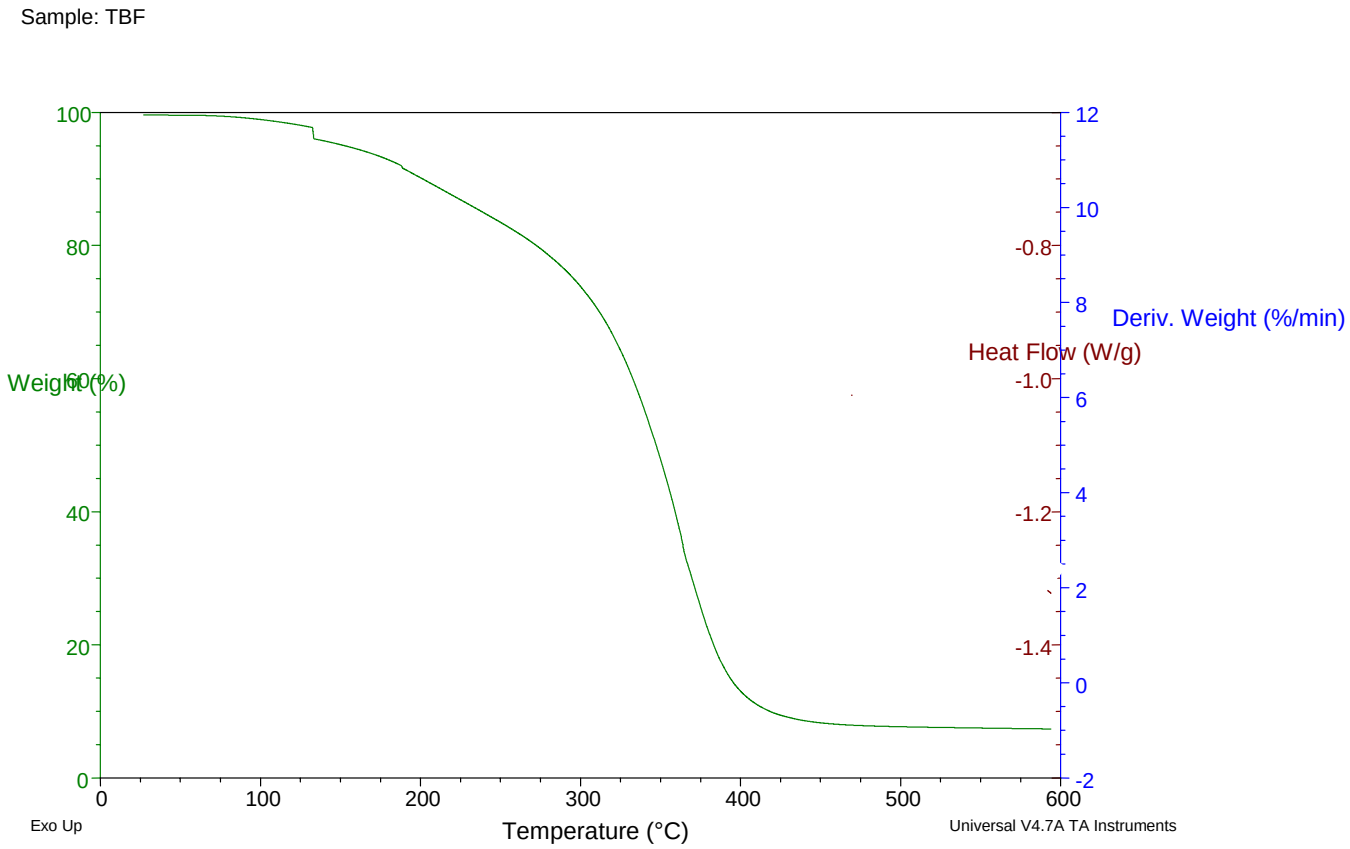



**Figure 4.13d: TGA of Treated Baobab Fibre Composites**

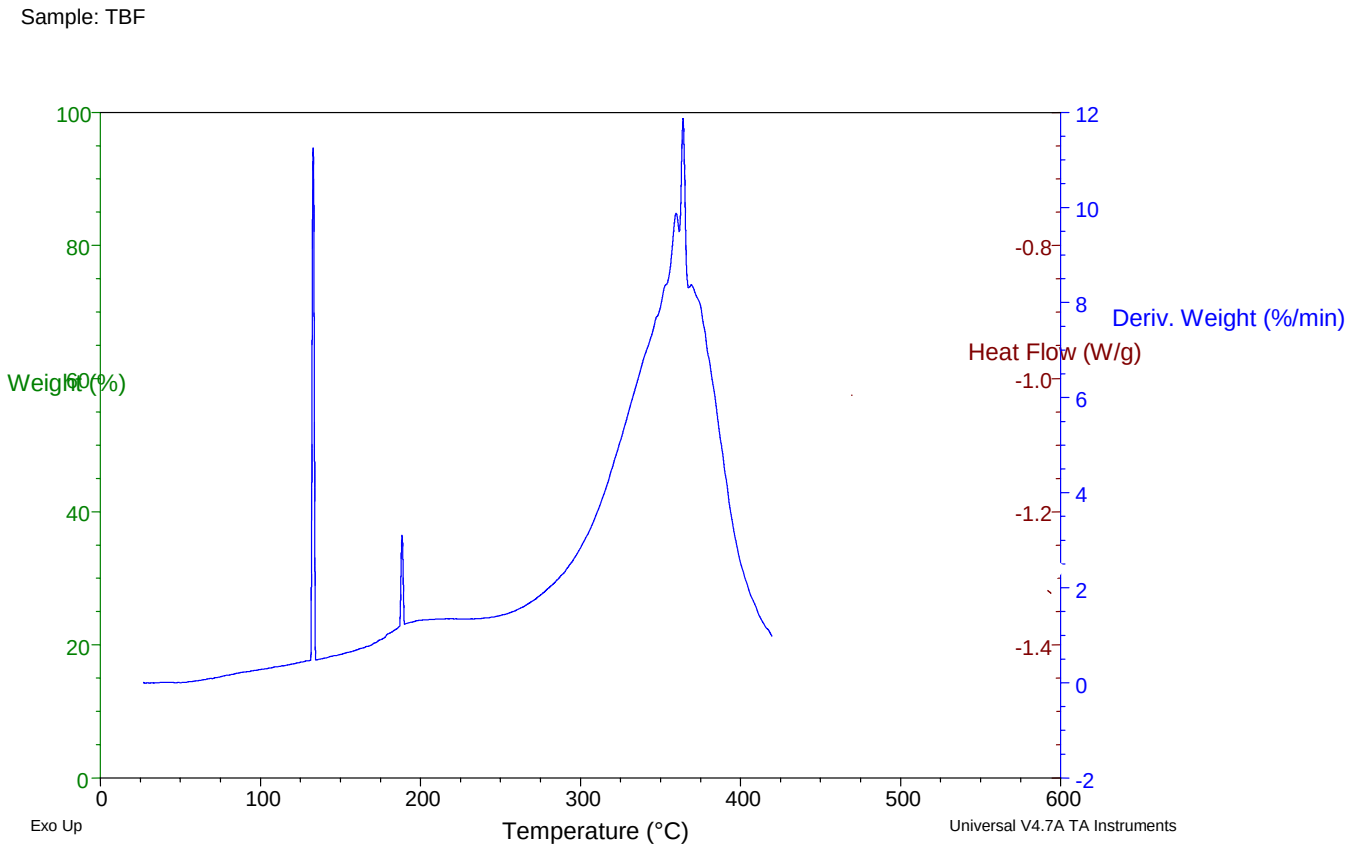



**Figure 4.13dd: TGA of Treated Baobab Fibre Composites**

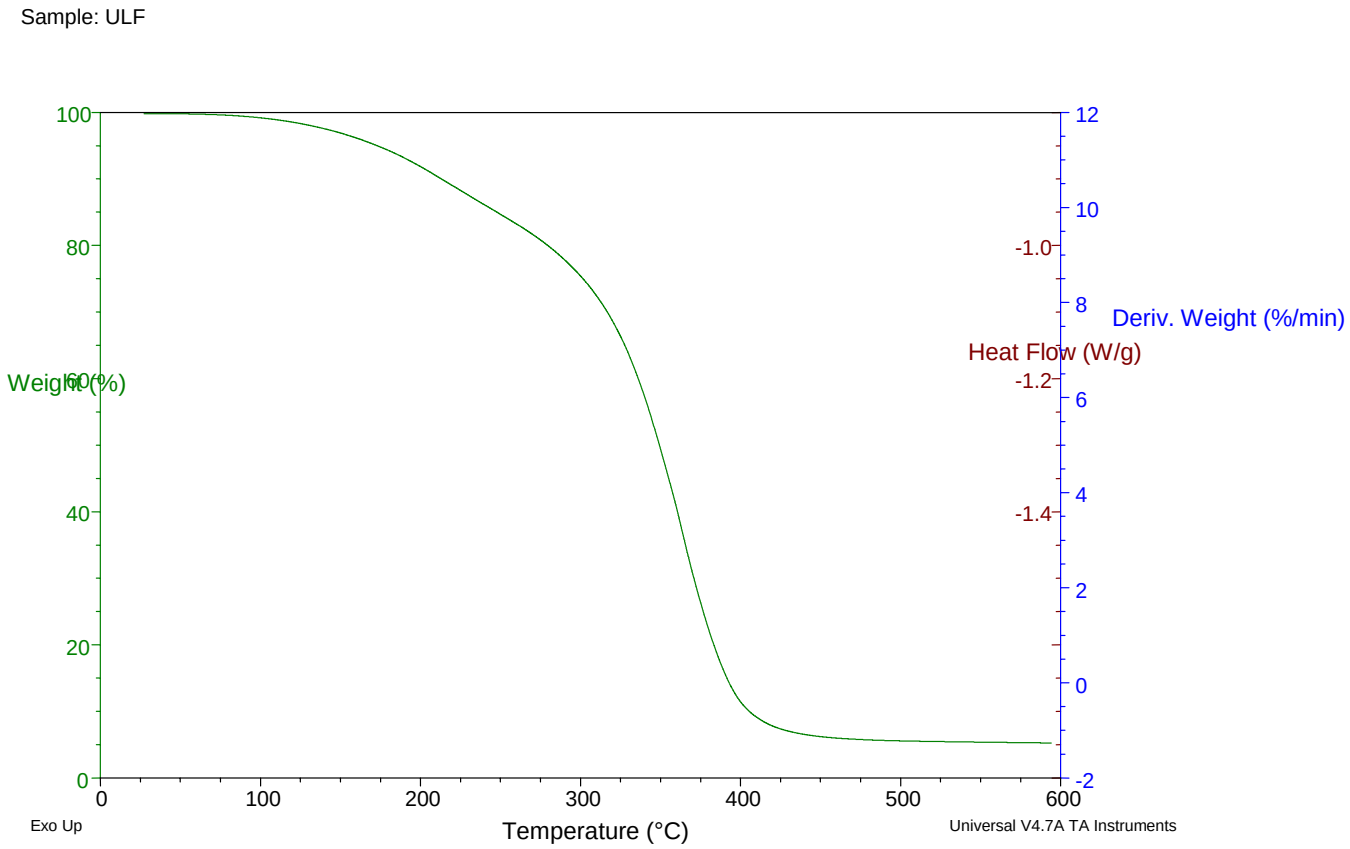



**Figure 4.13e: TGA of Untreated Luffa Gourd Fibre Composites**

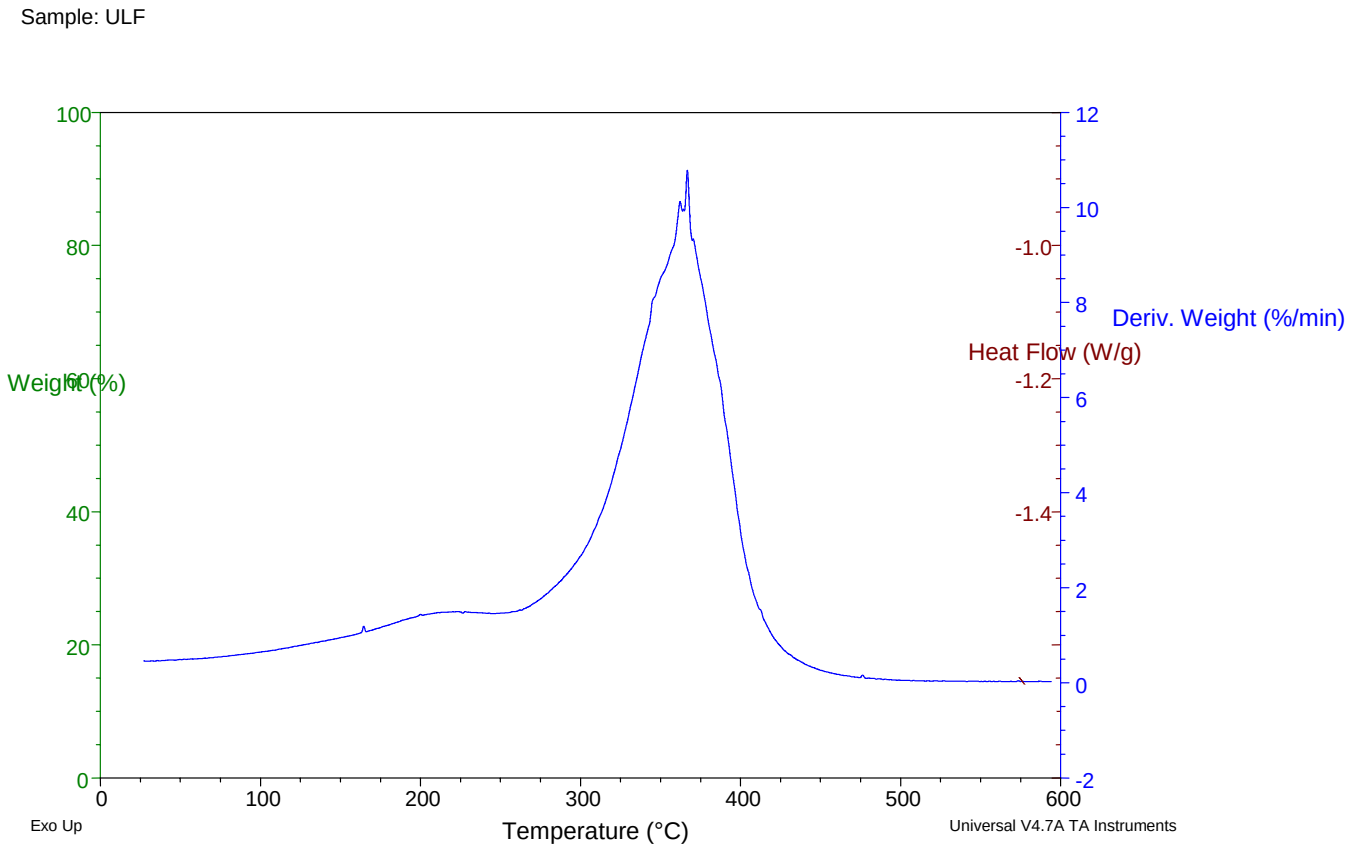



**Figure 4.13ee TGA of Untreated Luffa Gourd Fibre Composites**

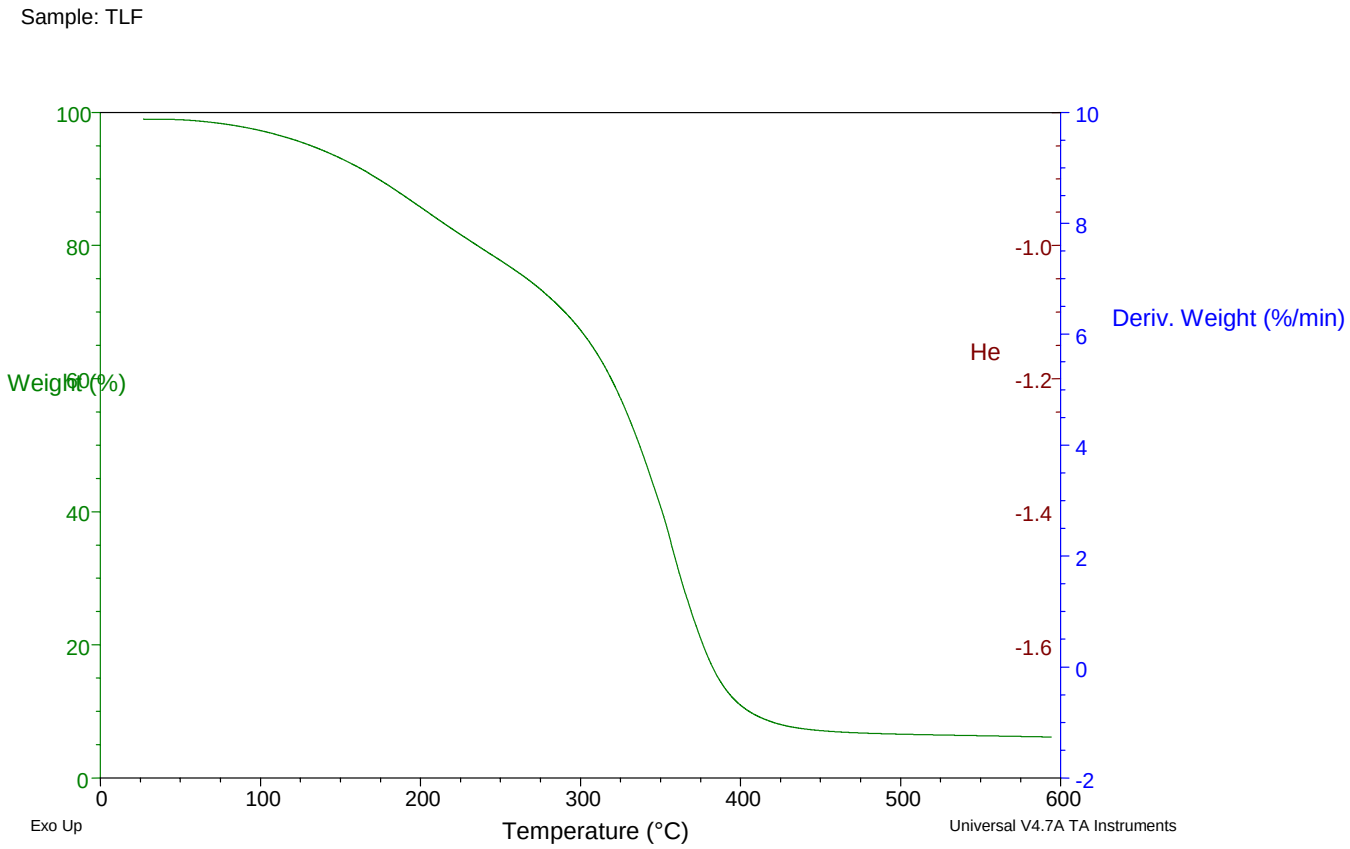



**Figure 4.13f TGA of Treated Luffa Gourd Fibre Composites**

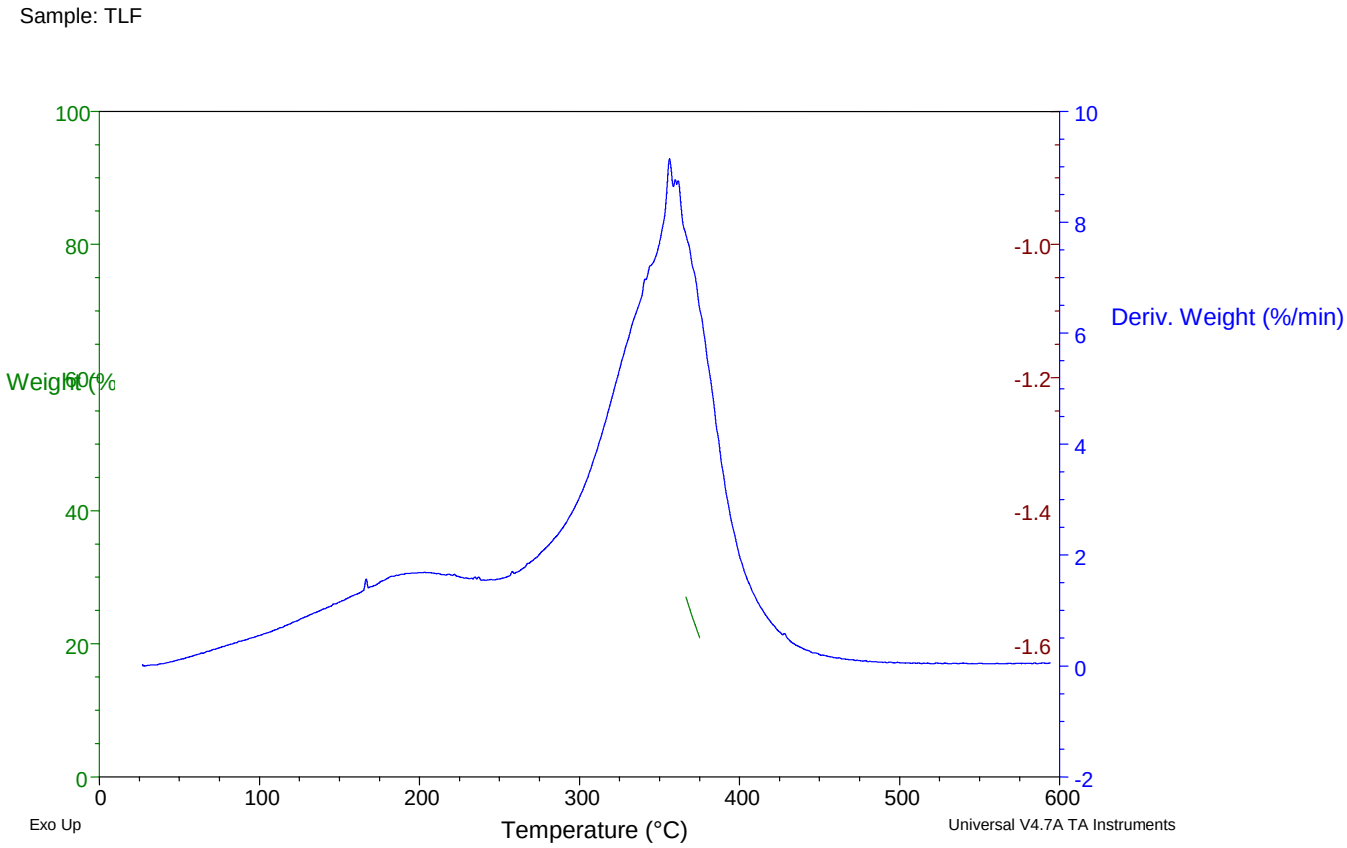



**Figure 4.13ff TGA of Treated Luffa Gourd Fibre Composites**

## 4.15 X-RAY DIFRACTION ANALYSIS RESULT OF COMPOSITES

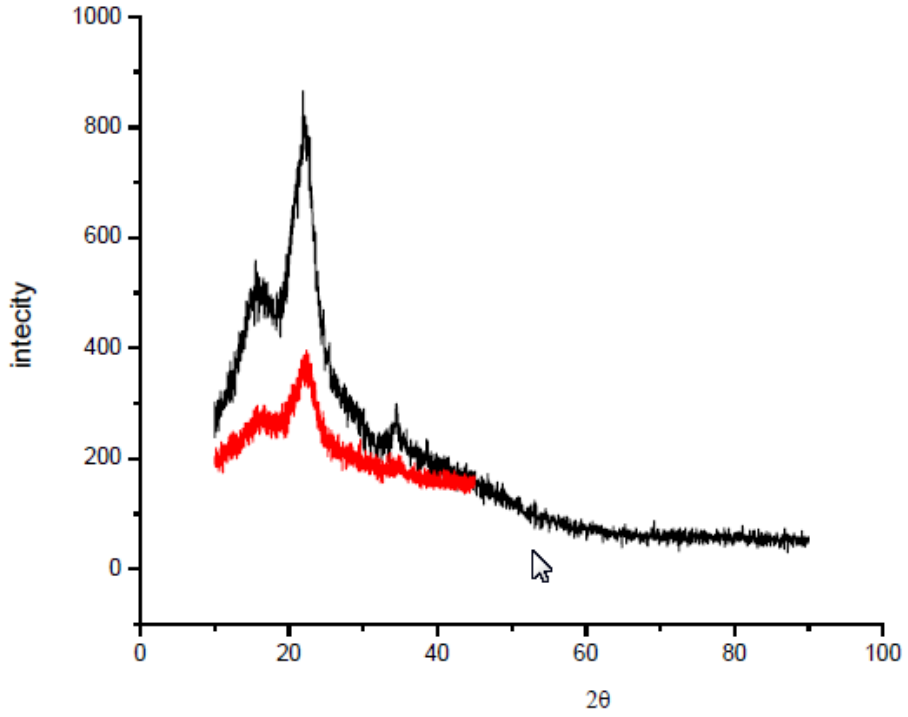



Figure 4.14a: XRD analysis of raw and treated fibre Baobab fibre composites

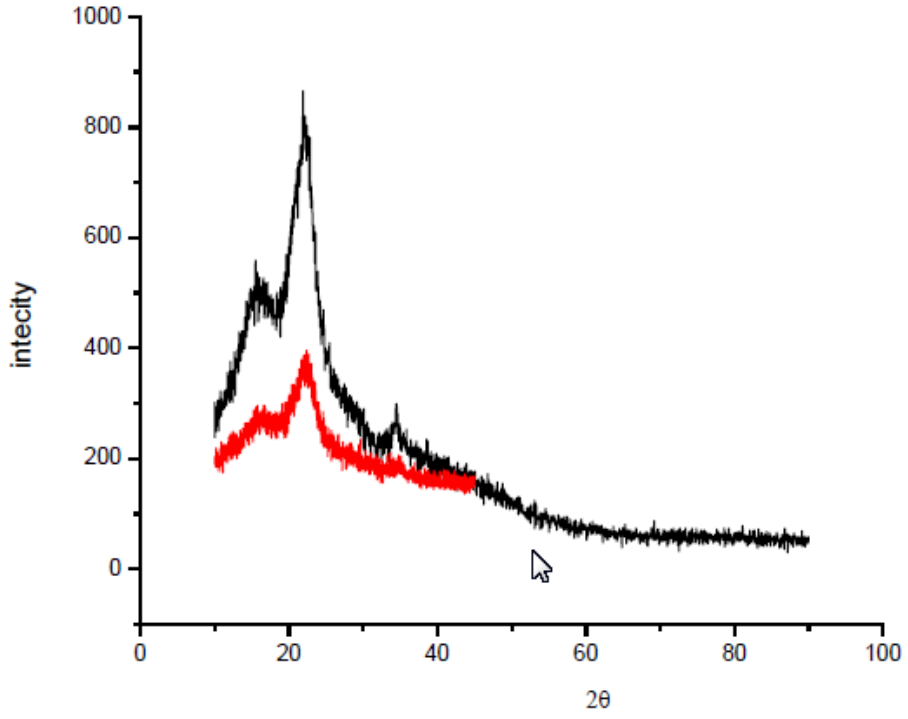



Figure 4.14b: XRD analysis of raw and treated fibre Dumpalm fibre composites

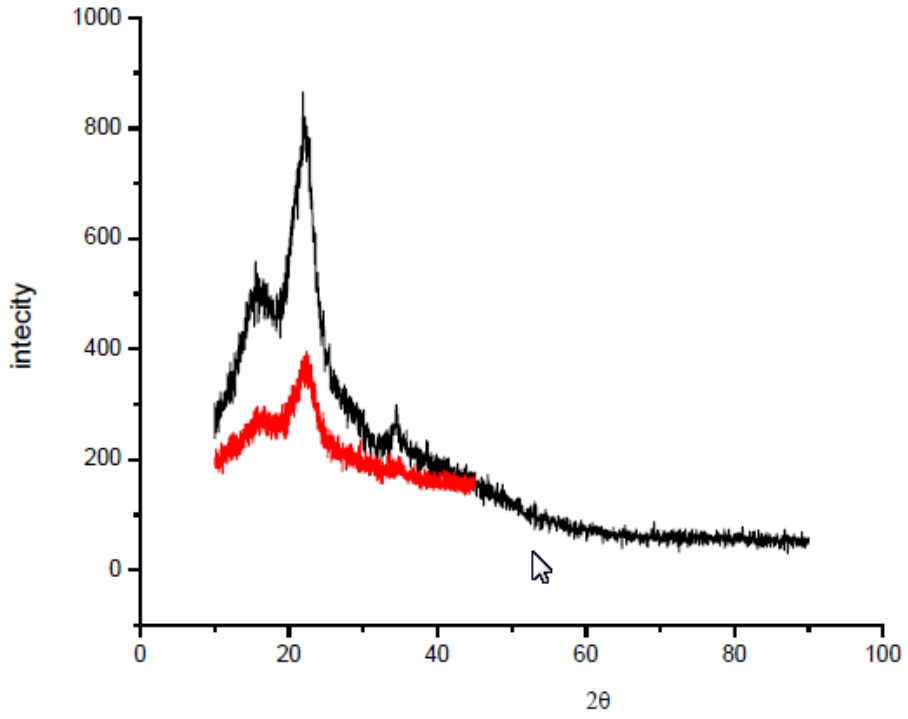



Figure 4.14c: XRD analysis of raw and treated fibre Luffa gourd fibre composites

# CHAPTER FIVE

## 5.0 DISCUSSION

Determination of the strength of the bundle fibres by the universal testing machine as shown in Table 4.1a-c reveal the breaking load of the differentbundle fibres. Dum palm fibres have the highest value of (105.90 N/ $cm^2$) followed by Luffa gourd fibres (68.96 N/$cm^2$) and lastly Baobab fibre (43.45 N/ $cm^2$). The extension at break shows Luffa gourd fibre to have 3.20% then Dum palm fibres 2.55% and finally Baobab fibres 2.12%.The results indicate that Dum palm fibre has the highest strength and it is expected to be more crystalline followed by Luffa gourd fibres and finally Baobab fibre which is less crystalline in nature.

The SEM micrographs of the fibres in Figures 4.2a-4.2i show the three different fibres. The SEM micrograph of untreated fibres shows the presence of natural waxy substances on the fibre surface i.e. pectins, lignins and ligno cellulose. Such waxy substances contributed to the ineffective fibre-matrix bonding and poor surface wet-out (Mohanty *et al*, 2001). Pectin, lignin and ligno cellulose are removed from surfaces of the treated fibres and there is an expectation of good adhesion between matrix and the fibre surfaces. The SEM micrographs of the treated fibres reveal that all chemical modifications smooth the fibre surface. This is probably due to the removal of cellulose admixtures from the surface of the fibres as a result of chemical treatment.

The results of tensile strength analysis of the composites in Tables 4.3a and 4.3b show the composites made with modified fibres have higher tensile strength in the following order : Baobab fibre composites (126.46 MPa), Luffa gourd fibre composites (114.12 MPa) and Dum palm fibre composites (106.35 MPa) these can be compared with the composites made with the unmodified fibres as follows : composites Baobab fibre composites (110.30 MPa), Luffa gourd fibre composites (107.95 MPa) and Dum palm fibre composites (95.83 MPa). Composite without reinforcement that is made of resin only have a tensile strength of (49.95 MPa). The

breaking extension of modified fibre composites is higher than the unmodified fibre composites. Baobab composites is has the highest value (3.2%) followed by Luffa gourd fibre composites (2.8%) and then Dum palm fibre composites (2.3%). The results of tensile properties of composites were strongly influenced by the adhesion between the fibre and matrix due to the presence of hydroxyl group in the natural fibres. Therefore composites produced have better strength. Modification of the fibre surface by chemicals usually makes the fibre more hydrophobic (Vilay et al, 2008).

On the other hand untreated fibre composites have lower tensile strength which is due to the weak compatibility between fibre and matrix. This is in agreement with the case of tensile properties of rubber reinforced Isora fibre composites. The properties of the composites filled with the treated fibres were higher than those filled untreated fibres at similar loading. To obtain good fibres reinforcement in the rubber composites, the adhesion between the rubber and the fibre was very important. From these results, it was clear that the aqueous alkali treated Isora fibre improved the fibre adhesion to rubber matrix (Lovely and Rani, 2006). The surface of the fibre could be modified by aqueous alkali treated at elevated temperatures and this improves the adhesion properties significantly. According to (Flodin and Zadorecki, 1985) fibre treatments can be used to prevent debonding at the fibre interface because covalent bonding can be formed between the rubber matrix and fibre. A strong force had to be used to overcome the shear strength, which resulted in a higher tensile strength. The elongation at break for the composites with the treated fibres was lower than composites with untreated fibres at similar loading. Alkali treatment of fibres further enhances the effect of the bonding agents such as shown by the higher modulus, tensile strength. The treated fibre provides a better surface for strong adhesion between the fibre and matrix, and the stress transfer become more efficient and consequently, there was

better enhancement in the properties. Treated fibres show lower elongation at break than untreated fibres.

The result of impact strength in Tables 4.4a and 4.4b show that the modified fibre composites have higher impact energies, Baobab fibre composites (23.972 KJ/$M^2$), Luffa gourd fibre composites (14.333 KJ/$M^2$) and Dum palm fibre composites (9.861 KJ/$M^2$), the unmodified composites Baobab fibre composites (19.759 KJ/$M^2$), Luffa gourd fibre composites (7.565 KJ/$M^2$) and Dum palm fibre composites (5.923 KJ/$M^2$). The impact strength of the resin without reinforcement was found to be (4.074 KJ/$M^2$). Baobab fibre composites have the highest impact energy followed by Luffa gourd and then Dum palm fibre composites. The impact strength of composites is influenced by many factors, which include the toughness properties and the nature of the interfacial region. This interfacial region is the most important and is directly related to the toughness of the composites. These aforementioned results show that treatment gave improved adhesion. The fibre/matrix adhesion determines the strength of the composites. Improved adhesion after treatment can lead to perfect bonding and thus composites will posses at relatively high impact strength. Composites having better interfacial bonding need absorbing capacity when the crack propagation occur along the fibre/matrix interface and this leads to an increase in impact strength

From the results, it is observed that 5% sodium hydroxide treated Baobab and Luffa gourd fibre treated composites have higher bending strength and higher percentage elongation than the untreated fibre composites. In the case of Dum palm fibre composites, untreated fibre composites have higher bending strength than the treated fibre composites. This signifies that treatment in that fibres did not improve the adhesion, bonding, transfer stress and wetttability between the fibre and the matrix. (Prasanna, et al).

In the assessment of hardness of the composites as shown in Tables 4. 6 the treatment improves the hardness property of the composites and this indicate that it improves the durability of the composites and also increase adhesion and strength at the fibre matrix interface. The result of chemical resistance of the composites (Table 4.6) shows that chemical treatment of natural fibres have been widely studied by many researchers (Li *et al*, 2007), but there is no agreement on which one is the best. Most chemical improved the tensile properties of natural fibre reinforced composites, Valadez Gonzalez and coworkers (1999). The chemical resistances of composite were determined by determining the weight and the thickness of the composites before and after chemical treatment. In the treatment of the composites with Dimethylformamide (DMF), Ethanol and 25 % Sulphuric acid ($H_2SO_4$), the composites dissolved completely leaving the fibres in the solvent. In the following solvents there is slight change in weight Acetone (0.44 %), Tetrahydrofuran (THF) 2.8 %, Sodium hydroxide (NaOH) 4.7 % and Hydrochloric acid (HCl) 5.7 % and change in thickness values Acetone 3.3%, Tetrahydrofuran (THF) 3.32 %, Sodium hydroxide (NaOH) 3.32 % and Hydrochloric acid (HCl) 3.32 %. This shows resistance of the composites to those chemicals. Rong and coworkers (2001) also found that chemical treatment (alkali treatment) removed hemicellulose and lignin in natural fibres. Cox and coworkers (1999) emphasized that hemicelluloses are readily hydrolyses by acid and even soluble in water at high temperature. Thus, removal of hemicellulose can decrease the water absorption in the final product. Cellulose with high crystallinity and cohesive density is insoluble in water and is more resistant to dilute acids; however they can swell and dissolve in strong acids, strong alkali or concentrated salt solutions (Cox *et.al*, 1999).

On the assessment of water absorption as shown in Table 4.8a-4.8f, the composites were found to have good water resistance, as the water absorbed by the composites was relatively small. The

densities of the fibres were found to be the same as shown in Tables 4.8a-f. The treatment did not make any change in weight of the composites (the untreated and treated composites). The percentage swelling in composites increase with the increase in the time of immersion up to a certain time when the swelling reaches maximum and does not increase (Jawaid et-al. 2012). Baobab fibre composites generally have higher percentage swelling than Luffa gourd and Dum palm fibre composites. This may be attributed to the fact that the fibres are more amorphous in nature than the other fibres.

The effect of chemical modifications on the fibre treated and untreated composites were studied using FTIR Spectrophotometer. Comparison of the representative spectra of untreated fibres and 5% sodiumhydroxide fibres treated composites are due to hydroxyl (OH) vibration in the cellulose structure of the fibres, for Baobab fibres composites the peaks in untreated Baobab fibre composites in Figure 4.10 are 612.4, 1317.4, 1600, 1948.17 and treated Baobab fibre composites in Figure 4.11 are 607.6, 1595.8, 1835.58 and 3264. 63 and that of untreated Luffa gourd fibre composites in Figure 4.12 are 619.17, 1124.54, 1599.04 and treated Luffa gourd fibre composite in Figure 4.13 are 483.18, 817.85 1284.63. And for Dum palm fibre composites untreated fibre composites in Figure 4.14 are 616.28, 1444.7, 1595.18 and that of treated Dum palm composites in Figure 4.15 are 612.42, 1314.53 and 1604.83.The most significant change are induced by acetylation and alkalization. The removal of hydroxyl group by the acetylation (as evidenced by disappearance of the strong absorption between 3200 $cm^{-1}$to 3600 $cm^{-1}$ caused by the OH group of the fibre constituents as a result of esterification) will drastically change the polarity of the surface and consequently their compatibility (reactivity) with polymer matrix (polar and non polar). The band in the spectrum of untreated fibres near1740$cm^{-1}$ which is also present in the spectra of the treated fibres, is assigned primarily to the C = O stretching vibration

of the carbonyl and acetyl group in the xylan component of hemicelluloses and also to chemical groups in lignin. Disappearance of some band in the spectra of alkali treated fibres is clearly seen. Indicating changes in the fibre surface chemical compositions.

Morphological characterization of the composite samples showed a relatively uniform distribution in the polymer matrix of modified fibre composite than the unmodified composites matrix (Bogoeva *et-al*, 2006). The SEM .micrographs of the composites shows untreated fibre composite to have less clarity than 5% sodium hydroxide fibre treated composites. The micrograph of the treated fibre composites shows pectin, lignin and ligno cellulose are removed from surfaces of the fibres and there is an indication of good adhesion between matrix and the fibre surfaces. In the case of untreated fibre composite, the presence of pectin, lignin, hemicelluloses and other impurities is responsible for the non clarity of the micrographs. In 5 % sodiumhydroxide treated fibre micrographs are clearer and much cleaner and open the fibre structure very well. The treatments carried out also reduce the number of hydroxyl group on the surface of the fibres. Reduction of hydroxyl group reduces the polarity and improves the compatibility with common thermosetting matrices. On treatment, the outer surface layer is removed and a rougher but more ordered structure is revealed.

The study of the composite morphology at nano level by AFM also indicated that the treated fibre composite to have a more uniform distribution compared to the untreated fibre composite. The fibres were found to have similarity in their micrographs at this level. This may be attributed to the fact that they are all cellulosic in nature. The topology of the fibres as shown in the Tables and Graphs shows treatment to give an improved property. The difference in height indicated by $Z_2$-$Z_1$=$\Delta Z$ in the Tables also shows treatment has improved properties of the composites as seen as follows; untreated baobab fibre composites $\Delta Z$ = 0.006100 nm and distance is152 nm and root

mean square (RMS) 1.24E+02 nm, treated Baobab fibre composites ΔZ = 0.006537, distance 203 nm and rms 7.768E+02 nm For untreated Luffa gourd fibre composites ΔZ = 0.003512 nm, distance 130 nm and RMS 4.591E+01 nm. Treated Luffa gourd fibre composites Δ Z = 0.00923 nm distance 554 nm and RMS 6.739E+08 nm and for Untreated Dum palm fibre composites Δ Z = 0.022890 nm, distance is 180 nm and RMS 2.762E01 nm treated Dum palm fibre composites ΔZ = 0.076727 nm, distance is 202 nm and RMS 1.561E+02 nm

Thermal analysis results obtained from DSC curve as shown in the Figures 4.12a-f. Heating was carried from 0 – 600 $^0$C at the heating rate of 10 $^0$C under Nitrogen atmosphere.

DSC analysis reveals that for Untreated Baobab fibre composite, $T_g$ is at 50 $^0$C. Melting is an endothermic progress. The heat flow becomes negative. Melting started at temperature above 275$^0$C because additional energy is absorbed. The peak melting is reached at 350 $^0$C. Degradation starts at temperature above 375$^0$ C. For treated Baobab fibre composites, Tg is at 50 $^0$C. Melting starts at 275 $^0$C and reached peak at 370 $^0$C, after 370 $^0$C degradation started. And at150 $^0$C – 210 $^0$C range, there is no endothermic or exothermic changes. This is the best processing temperature for the composites. For Dum palm untreated fibre composite, the melting started at 210 $^0$C and reach its peak at 375 $^0$C. Degradation starts after 375 $^0$C, $T_g$ is at 50 $^0$C. For treated Dumpalm fibre composite, melting starts at 250 $^0$C and reach its peak at 360 $^0$C. Degradation starts at 360 $^0$C. No endothermic or exothermic reactions at 80 - 200 $^0$C which is the best processing temperature. For Luffa gourd fibre untreated composite, the melting temperature starts at 210 $^0$C and reach its peak at 350 $^0$C. $T_g$ is at 80 $^0$C, degradation starts after 350 $^0$C. For treated Luffa gourd fibre composite, $T_g$ is at 50 $^0$C, melting starts at 260 $^0$C and reach its peak at 370 $^0$C. Degradation starts after 370 $^0$C.

TGA analysis of the composites reveals that Untreated Baobab fibre composites, different degradation temperatures were observed, that is 2 peaks which started at 180 $^0$C and reach peak at 350 $^0$C as seen in the Fig4.13cc. The percentage weight remaining at 425 $^0$C is 5 % that is final degradation temperature. Treated Baobab fibre composite shows 3 different degradation temperatures were observed which started 125 $^0$C, 190 $^0$C and peak 360 $^0$C. Degradation starts at a lower temperature. Untreated Dum palm fibre composite, one peak degradation temperature was seen, and is at 350 $^0$C and 5 % residue at 600 $^0$C. For treated Dum palm fibre composite, six different degradation peaks were seen, which started at 150 $^0$C and reach peak at350 $^0$C the residue is 5 % at 600 $^0$C. Untreated Luffa gourd fibre composite degradation peak seen is one that started at 275 $^0$C and reached peak at 350 $^0$C residue is 5 % at 600 $^0$C.Treated Luffa gourd fibre composites degradation is also one and started at 275 $^0$C and reached peak at350 $^0$C and residue is 5 % at 600 $^0$C.

XRD patterns of both raw and alkali treated fibres are shown in Figure4.14a-c. The full width at half maximum (FWHM) of the diffraction peaks of both raw and alkali treated samples are analyzed. The $K_\alpha 2$ components of the peaks are obtained from the data. The diffractograms of both treated and raw fibres display a well-defined main peak around $2\Theta = 22.5°$, which is the characteristic peak of cellulose-I. By using the formula Kc = Mc/Mt x 100, we can calculate the degree of crystallinity for both the samples. It is found that the degree of crystallinity is found to be 48.57 % and 46.26 % for raw and alkali treated Baobab fibre composite, and 71.2 % and 66.32 % for Dumpalm raw and alkali treated. And 69.59 % and 65.58 % raw and treated Luffa gourd fibre composite. This may be due to the swelling of the fibres due to the alkali treatment as this leads to the formation of new Na-cellulose-I lattice.

This treatment leads to the fibrillation which causes the breaking down of the composite fibre bundles into smaller fibres. So a better adhesion between the fibre and matrix occurred. This treatment gave rise to a decrease in the degree of crystallinity which is proved by the XRD analysis of the raw and alkali treated fibres i.e. a swelling of fibre is occurred in the fibre due to the treatment.

# CHAPTER SIX

## 6.0 SUMMARY, CONCLUSION AND RECOMMENDATIONS

### 6.1 SUMMARY

In this study, the effect of modification of the fibre surface was studied for the three fibres, which are Baobab fibres, Luffa gourd fibres and Dum palm fibres. The fibres were sourced from different plants and are retted in water for 7 days. Fibres were finally extracted and treated with different concentrations of caustic soda. The composites were made of untreated and 5 % sodiumhydroxide treated fibres. Properties of the composites such as physical, mechanical, morphological, resistance to chemicals and thermal were studied.

Determination of the fibre strength was carried out using universal testing machine and it reveals the breaking load of the different fibres with Dum palm fibre having the highest value of 103.90 N/$cm^2$, then Luffa gourd fibres 68.96 N/ $cm^2$ and Baobab fibres 43.45 N/ $cm^2$. Also extension at break is as follows Luffa gourd fibres 3.20 %, then Dum palm fibres 2. 55 % and Baobab fibres 2.12 %. The result indicated that Dum palm have the highest strength which is an indication of crystallinity, followed by Luffa gourd and lastly Baobab which is shows the fibre to be less crystalline in nature. The SEM micrographs of the treated fibres suggest that lignin, hemicelluloses and pectins have been removed from the surfaces hence a clean surface is revealed. The presence of lignin, pectins and hemi celluloses in untreated fibres, is also shown in the SEM micrographs.

The result of tensile strength of the composites shows the modified fibres to have a general improvement on the strength of the composites; Baobab composite has the highest value of 126.46 MPa then Luffa gourd114.12 MPa and Dum palm106.35 MPa, while the untreated composites values are Baobab 110.30 MPa, Luffa gourd 107.95 MPa and Dum palm 95.83 MPa. The breaking extension of the modified fibre composite is higher than that of unmodified. The

assessment of impact strength also indicated the modified fibre composite to have improved properties than the unmodified. Baobab is having the highest impact energy of 23.972 KJ, Luffa gourd 14.333 KJ and Dum palm 9.861 KJ while that of unmodified are Baobab 19.759 KJ, Luffa gourd 7.564 KJ and Dum palm 5.926 KJ.

The bending modulus of treated fibre composite shows a remarkable improvement in the bending modulus of Baobab and Luffa gourd while in Dum palm no improvement but drop down. On the assessment of hardness of the composite the treatment improves the hardness property and this shows it improves the durability of the composite and also increases adhesion, strength at the fibre matrix interface. In the assessment of chemical resistance of the composite, it shows the composite to have resistance to some chemicals and non-resistance to others. Dimethylformamide (DMF), Ethanol and Conc. Sulphuric acid ($H_2SO_{4)}$ dissolve the composite completely while Acetone, Tetrahydrofuran (THF), Sodiumhydroxide (NaOH) and Hydrochloric acid (HCl) did not damage the composites

In the assessment of water absorption the composites were found to be resistant to water, because they absorbed very little water. Water absorption is generally considered to be a disadvantage in composites. Yet natural thermoplastic biocomposites always have higher water absorption than the plastic polymer itself. Fibre surface modification reduce the hydroxyl group in the cell wall of cellulose molecules, is undertaking to reduce the water absorption of biocomposite. Chemical treatments have been used to decrease the water absorption of natural fibre reinforced composites as reported by many researchers (Mohanty et al, 2001 and Bledzki and Gassan, 1999). On density assessment of the composite, they are found to have a uniform density that is the treatment did not make any change in densities. On the morphological study of the composite by SEM, the fibre treated composites were found to be more uniform in distribution than the untreated. The FTIR analysis of the composites in which representative

samples of the spectra were compared shows change in peaks and is attributed to change in OH bond in the treated fibres.

The study of the composite morphology at nano level by AFM also indicated that the treated fibre composite to have a more uniform distribution compared to the untreated fibre composite. The fibres were found to have similarity in their micrographs at this level this may be attributed to the fact that they are all cellulosic in nature. The topology of the fibres as shown in the table and graph shows treatment to give an improved property. The difference in height indicated by $Z_2$-$Z_1$=$\Delta Z$ in the Table also shows treatment improves properties of the composite as seen as follows; Untreated Baobab$\Delta$ Z = 0.006100 nm, distance is152 nm RMS 1.24E+02 nm, Treated Baobab $\Delta$ Z = 0.006537, distance 203 nm and 7.768E+02. For Untreated Luffa gourd $\Delta Z$ = 0.003512 nm, distance 130 nm and RMS 4.591E+01 nm, treated Luffa gourd $\Delta$ Z = 0.00923 nm, distance 554 nm and RMS 6.379E+08 nm, and for untreated Dum palm$\Delta$ Z = 0.022890 nm, distance is 180 nm and RMS 2.762E+01 nm, treated Dum palm $\Delta$ Z = 0.076727 nm, distance is 202 nm and RMS 1.561E+02 nm. Thermal analyses of the composites were carried out using a Differential scanning calorimeter and Thermogravimetric analyzer. DSC analyses reveal the $T_g$, Tm and Degradation temperatures of the composites. On the TGA analyses, the rate of degradation and char content were determined for both untreated and treated fibre composites. On the analyses of composites by XRD treated and untreated fibre composites were characterized and percentage crystallinity was determined. Treatment was found to reduce the crystallinity of the composites.

## 6.2 CONCLUSION

From the research carried out, the bundle strength of fibres meets the minimum strength. Among the fibres Dum palm fibres has the highest strength followed by Luffa gourd which has medium strength and Baobab fibre with the least strength. It is concluded that Dum palm is highly crystalline, because crystallinity is an indication of strength, followed by Luffa gourd and Baobab fibres are less crystalline in nature. The trend in the extension percent varied, with Luffa gourd fibres 3.20 % as the highest followed by Dum palm fibres 2. 55 % and Baobab fibres 2.12 % with the least value.

The unmodified fibres, 5 % and 20 % modified fibres morphologies were studied using SEM.The SEM micrographs of the treated fibres suggest that cellulosis, hemicellulosis, pectins have been removed from the surfaces hence a clean surface is revealed, compared to untreated fibres. All chemically modified surfaces are smoother than the unmodified surfaces. The surface structure open the fibre structure very well, this is attributed to the fact that there is chemical dissolution during treatments at the outer surfaces layer. Thus chemical treatment improves fibre properties.

The unmodified fibres and modified fibres composites were produced for each type of fibres. On the assessment of tensile strength of the composites, Luffa gourd fibre composite has the highest tensile strength followed by Baobab and Dum palm fibre composites. For the untreated fibre composite. Treatment generally improves the tensile property. Baobab fibre composite respond better to chemical treatment followed by Luffa gourd and Dum palm fibre composite. The impact strength of the 3 composite studied both treated and untreated, reveals that Baobab fibre composites has the highest impact strength value followed by Luffa gourd and then Dumpalm But it was observed treatment improves the impact strength. Luffa gourd and Dum palm responded better with chemical treatment because there impact strength value almost double

after treatment. In the assessment of bending modulus, chemical treatment has influenced the property. In Baobab and Luffa gourd composite it increases the property whereas in the case of Dum palm the influence of treatment is negative that is bending stress dropped after treatment. The assessment of hardness of the composite reveals Luffa gourd and Dum palm composites hardness value to increase with chemical treatment but reduce on Baobab fibre composite.

On the assessment of chemical resistance of the composite, it shows resistance of the composites to some chemicals. Cellulose with high crystallinity and cohesive density is insoluble in water and is more resistance to dilute acids, however they can swell and dissolved in strong acids, strong alkalis or concentrated salt solutions (Cox *et.al,* 1999).The water absorption property of the composite shows no change with and without treatment. Generally the composites have low water absorption. The densities of the composites were found to be uniform irrespective of the fibre type. On the FTIR analysis treatment indicate a change in some peaks on the composites. On the determination of the morphology by SEM, 5 % caustic soda treated fibres show a more uniform distribution and a better adhesion than the untreated fibre composite. Morphological study at nano level by AFM indicated that modified fibre composites have a more uniform distribution compared to the unmodified fibre composites. The fibre reinforced composites were found to have similarity in their micrographs at nano level this may be attributed to the fact that they are all cellulosic in nature. Difference in topologies of the in the composites as shown in the tables and graphs as well as root mean square (rms) value also shows an improvement in the modified fibre composites than the unmodified fibre composites.

Thermal analysis of the composite by DSC reveals the $T_g$, $T_m$ and Decomposition temperature of the composites. It is shown that the $T_g$, $T_m$ and Decomposition temperatures of untreated and treated fibres is almost the same. This is because the matrix is concerned.

On TGA analysis, Untreated Dumpalm fibre composite one peak was seen while for treated fibre composite six peaks were seen. For untreated Baobab fibre composite two peaks were seen and for treated three peaks were observed. For Luffa gourd composite both untreated and treated one peak was seen at the same temperature.

XRD property of the composite was studied and the result shows treated and untreated fibre composites, The percentage crystallinity of the composite were calculated with Dumpalm composite been the most crystalline followed by Luffa gourd composite and lastly Baobab which is less crystalline. Untreated fibre composite are more crystalline than the treated fibre composites, hence alkali treatment lower the crystallinity of fibre composites.

After studying all these properties, areas of utilization of new outlets were determined. The areas include plastics such as in tanks, automotives industry for different parts of the automobiles, construction industry and for structural applications such as in partitions.

## 6.3 RECOMMENDATIONS

1. Other methods of composite fabrication should be used for example injection moulding technique.
2. Different matrices to be used and to determine the properties of the composite produced e.g. epoxy resin
3. Different method of chemical modification to be applied on the fibres such as silane treatment, permanganate treatment and plasma treatment.
4. The study can be extended to determine other properties not tested in this study such as ultraviolet resistance, electrical properties etc.
5. Hybrid composites recommended to be produced by combination of two or three of these fibres.
6. Additives for fibre-reinforced composites could be studied to improve special properties such as flow ability, resistant to microbial attacks.
7. Varying the fibre loading and determining the properties.

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# APPENDIX A

**Table 4.1a Breaking Load and Extension of Baobab Fibres**

| Test | Breaking Load ($N/cm^2$) | Extension at Break (%) |
|---|---|---|
| 1 | 43.81 | 2.29 |
| 2 | 43.25 | 1.90 |
| 3 | 43.50 | 2.19 |
| **Mean** | **43.45** | **2.12** |
| **SD** | **0.2921** | **0.2027** |

**Table 4.1b Breaking Load and Extension of Dum palm Fibres**

| Test | Breaking Load ($N/cm^2$) | Extension at Break (%) |
|---|---|---|
| 1 | 93.06 | 2.13 |
| 2 | 113.29 | 2.87 |
| 3 | 105.37 | 2. 65 |
| **Mean** | **103.90** | **2. 55** |
| **SD** | **7.751** | **0.380** |

**Table 4.1c Breaking Load and Extension of Luffa Gourd Fibres**

| Test | Breaking Load ($N/cm^2$) | Extension at Break (%) |
|---|---|---|
| 1 | 63.35 | 3.50 |
| 2 | 71.24 | 2.87 |
| 3 | 72.28 | 3.22 |
| **Mean** | 68.96 | **3.20** |
| **SD** | **4.883** | **0.3157** |

**Table 4.2a Tensile Properties of Untreated Composites**

| S.No | Control (UP without fibres) Pa | Baobab | | Luffa | | Dumpalm | |
|---|---|---|---|---|---|---|---|
| | | Tensile Strength (MPa) | %Extension | Tensile Strength (MPa) | % Extension | Tensile Strength (MPa) | % Extension |
| 1 | 49.56 | 104.92 | 1.03 | 81.47 | 1.98 | 131.94 | 0.94 |
| 2 | 50.00 | 99.53 | 0.98 | 84.97 | 1.68 | 85.83 | 0.84 |
| 3 | 50.03 | 110.31 | 1.28 | 157.42 | 1.27 | 95.83 | 0.98 |
| Mean | 49.68 | 104.92 | 1.07 | 107.95 | 1.60 | 104.54 | 0.93 |
| SD | | 5.39 | 0.16 | 4.29 | 0.36 | 2.43 | 0.07 |

**Table 4.2b Tensile Properties of Treated Composites**

| S.No | Baobab | | Luffa | | Dumpalm | |
|---|---|---|---|---|---|---|
| | Tensile Strength (MPa) | %Extension | Tensile Strength (MPa) | %Extension | Tensile Strength (MPa) | %Extension |
| 1 | 138.33 | 3.35 | 121.11 | 2.43 | 132.78 | 1. 63 |
| 2 | 111.056 | 2.82 | 115.97 | 3.06 | 90.17 | 2.75 |
| 3 | 130.00 | 3.32 | 105.28 | 3.00 | 96.11 | 2.54 |
| Mean | 126.46 | 3.20 | 114.12 | 2.80 | 106.35 | 2.30 |
| SD | 13.97 | 0.29 | 8.07 | 0.34 | 2.30 | 0.14 |

| Test | Control ($KJ/m^2$) | Baobab ($KJ/m^2$) | Luffa gourd ($KJ/m^2$) | Dumpalm ($KJ/m^2$) |
|---|---|---|---|---|
| **1** | 4.167 | 22.500 | 7.639 | 5.000 |
| **2** | 3.888 | 19.889 | 7.500 | 7.361 |
| **3** | 4.167 | 16.889 | 7.556 | 5.417 |
| **Mean** | 4.074 | 19.759 | 7.565 | 5.926 |
| **SD** | 4.50 | 2.80 | 6.99 | 1.26 |

**Table 4.3a Impact Strength of Untreated Composites**

| Test | Baobab ($KJ/m^2$) | Luffa gourd ($KJ/m^2$) | Dumpalm ($KJ/m^2$) |
|---|---|---|---|
| **1** | 27.806 | 13.333 | 11.194 |
| **2** | 22.278 | 17.472 | 9.861 |
| **3** | 21.833 | 12.194 | 8.528 |
| **Mean** | 23.972 | 14.333 | 9.861 |
| **SD** | 3.33 | 2.77 | 1.33 |

**Table 4.3b Impact Strength of Treated Composites**

**Figure 4.4a: Bending Modulus of Untreated Fibre Composites**

| Composite | Bending stress (Mpa) | Elongation (%) |
|---|---|---|
| **Baobab** | 73.35 | 11.15 |
| **Luffa** | 45.77 | 12.02 |
| **Dum** | 64.15 | 8.29 |
| **Control** | 14.02 | 10.56 |

**Table 4.4b Bending Modulus of 5% NaOH Fibre Treated Composite**

| Composite | Bending stress (Mpa) | Elongation (%) |
|---|---|---|
| **Baobab** | 109.92 | 14.10 |
| **Luffa** | 137.3 | 12.59 |
| **Dum** | 45.77 | 12. 62 |

**Table 4.5a Hardness property Untreated composites**

| Test | Baobab (shore D) | Luffa gourd (shore D) | Dum palm (shore D) | UPR |
|---|---|---|---|---|
| 1 | 59 | 53 | 6 0 | 42 |
| 2 | 6 0 | 50 | 50 | 43 |
| 3 | 6 0 | 57 | 58 | 43 |
| Mean | 6 0 | 53 | 56 | 43 |
| **SD** | **0.50** | **3.55** | **5.29** | **0.005** |

**Table 4.5b Hardness property of 5% NaOH treated composites**

| **Test** | **Baobab** (shore D) | **Luffa gourd** (shore D) | **Dum palm** (shore D) |
|---|---|---|---|
| **1** | 58 | 62 | 57 |
| **2** | 48 | 62 | 60 |
| **3** | 53 | 64 | 61 |
| **Mean** | 53 | 63 | 59 |
| **SD** | **5.0** | **1.22** | **2.12** |

| **Test** | **Mass (g)** | **Volume (cm3)** | **Density(g/cm3)** |
|---|---|---|---|
| **1** | 10.05 | 10.0 | 1.005 |
| **2** | 10.05 | 9.8 | 1.005 |
| **3** | 10.04 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7a Density of Untreated baobab composite**

| **Test** | **Mass (g)** | **Volume (cm3)** | **Density(g/cm3)** |
|---|---|---|---|
| **1** | 10.05 | 10.0 | 1.005 |
| **2** | 10.04 | 9.8 | 1.005 |
| **3** | 10.05 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7b Density of 5% NaOH treated Baobab treated composite**

| **Test** | **Mass (g)** | **Volume (cm3)** | **Density(g/cm3)** |
|---|---|---|---|
| **1** | 10.05 | 10.0 | 1.005 |
| **2** | 10.05 | 9.8 | 1.005 |
| **3** | 10.05 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7c Density of Untreated Luffa composite**

| Test | Mass (g) | Volume (cm3) | Density(g/cm3) |
|---|---|---|---|
| **1** | 10.05 | 10.0 | 1.005 |
| **2** | 10.05 | 9.9 | 1.005 |
| **3** | 10.05 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7d Density of 5% NaOH treated Luffa composite**

| Test | Mass (g) | Volume (cm3) | Density(g/cm3) |
|---|---|---|---|
| **1** | 10.04 | 10.0 | 1.005 |
| **2** | 10.05 | 10.0 | 1.005 |
| **3** | 10.05 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7e Density of Untreated Dum composite**

| Test | Mass (g) | Volume (cm3) | Density(g/cm3) |
|---|---|---|---|
| **1** | 10.05 | 10.0 | 1.005 |
| **2** | 10.05 | 10.0 | 1.005 |
| **3** | 10.04 | 10.0 | 1.005 |
| **Mean** | 10.05 | 10.0 | 1.005 |

**Table 4.7f Density of 5% NaOH treated Dum palm fibre treated composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.71 | 0.51 | 3.59 |
| **2** | 14.20 | 14.73 | 0.53 | 3.73 |
| **3** | 14.20 | 14.76 | 0.56 | 3.94 |
| **4** | 14.20 | 14.77 | 0.57 | 4.01 |
| **5** | 14.20 | 14.77 | 0.57 | 4.01 |
| 6 | 14.20 | 14.77 | 0.57 | 4.01 |

**Table 4.8a Water Absorption of Untreated Baobab fibre composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.26 | 0.06 | 0.42 |
| **2** | 14.20 | 14.27 | 0.07 | 0.49 |
| **3** | 14.20 | 14.30 | 0.10 | 0.70 |
| **4** | 14.20 | 14.32 | 0.12 | 0.85 |
| **5** | 14.20 | 14.32 | 0.12 | 0.85 |
| 6 | 14.20 | 14.32 | 0.12 | 0.85 |

**Table 4.8b Water Absorption of Untreated Luffa gourd fibre composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.27 | 0.07 | 0.49 |
| **2** | 14.20 | 14.28 | 0.08 | 0.56 |
| **3** | 14.20 | 14.29 | 0.09 | 0. 63 |
| **4** | 14.20 | 14.29 | 0.09 | 0. 63 |
| **5** | 14.20 | 14.29 | 0.09 | 0. 63 |
| 6 | 14.20 | 14.29 | 0.09 | 0. 63 |

**Table 4.8c Water Absorption of Untreated Dum fibre composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.50 | 0.30 | 2.11 |
| **2** | 14.20 | 14.51 | 0.31 | 2.18 |
| **3** | 14.20 | 14.52 | 0.32 | 2.25 |
| **4** | 14.20 | 14.53 | 0.33 | 2.32 |
| **5** | 14.20 | 14.53 | 0.33 | 2.32 |
| 6 | 14.20 | 14.53 | 0.33 | 2.32 |

**Table 4.8d Water Absorption of Treated Baobab fibre composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.24 | 0.04 | 0.28 |
| **2** | 14.20 | 14.25 | 0.05 | 0.35 |
| **3** | 14.20 | 14.26 | 0.06 | 0.42 |
| **4** | 14.20 | 14.26 | 0.06 | 0.42 |
| **5** | 14.20 | 14.26 | 0.06 | 0.42 |
| 6 | 14.20 | 14.26 | 0.06 | 0.42 |

**Table 4.8e Water Absorption of Untreated Luffa gourd fibre composite**

| Time (Hr) | Initial weight (g) | Final weight (g) | Difference in weight (g) | % difference |
|---|---|---|---|---|
| **1** | 14.20 | 14.24 | 0.04 | 0.28 |
| **2** | 14.20 | 14.25 | 0.05 | 0.35 |
| **3** | 14.20 | 14.26 | 0.06 | 0.42 |
| **4** | 14.20 | 14.27 | 0.07 | 0.49 |
| **5** | 14.20 | 14.27 | 0.07 | 0.49 |
| 6 | 14.20 | 14.27 | 0.07 | 0.49 |

**Table 4.8f Water Absorption of Treated Dum fibre composite**

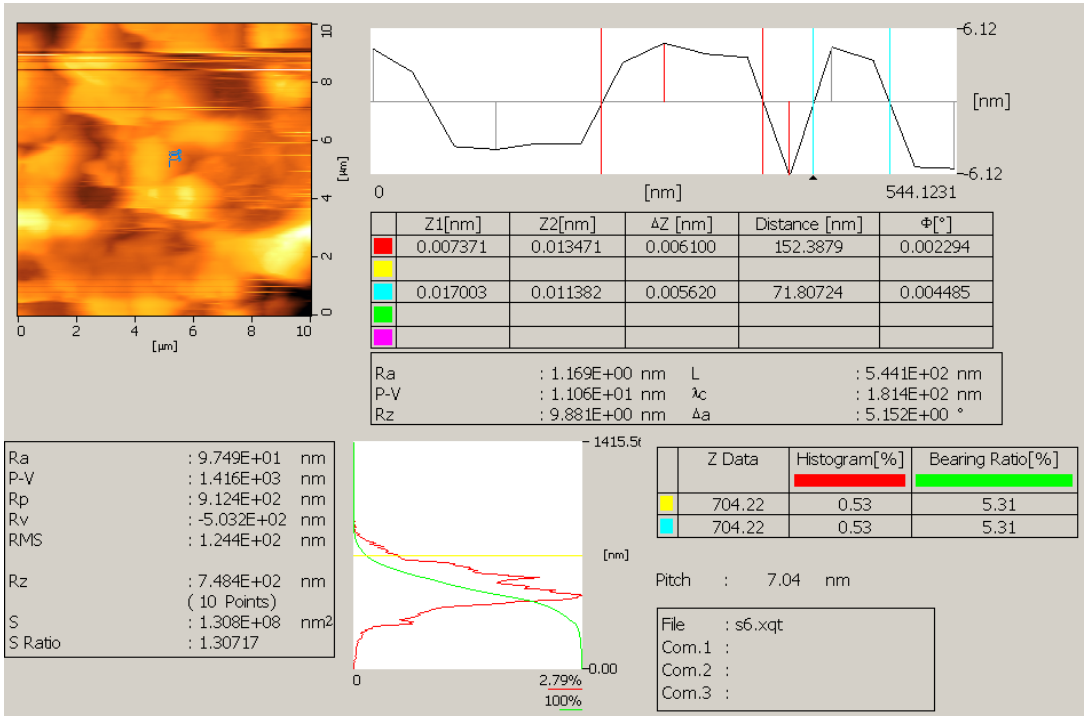



Table 4.9a AFM of Untreated Baobab Composites

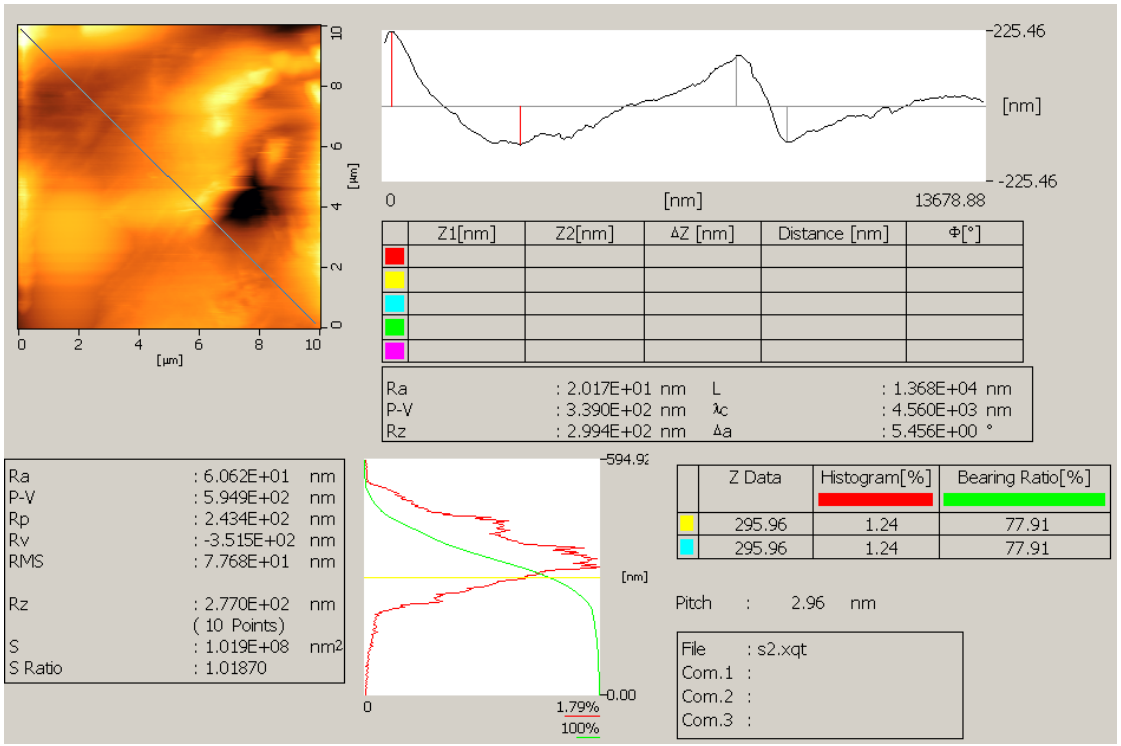



Table 4.9b AFM of Treated Baobab Composites

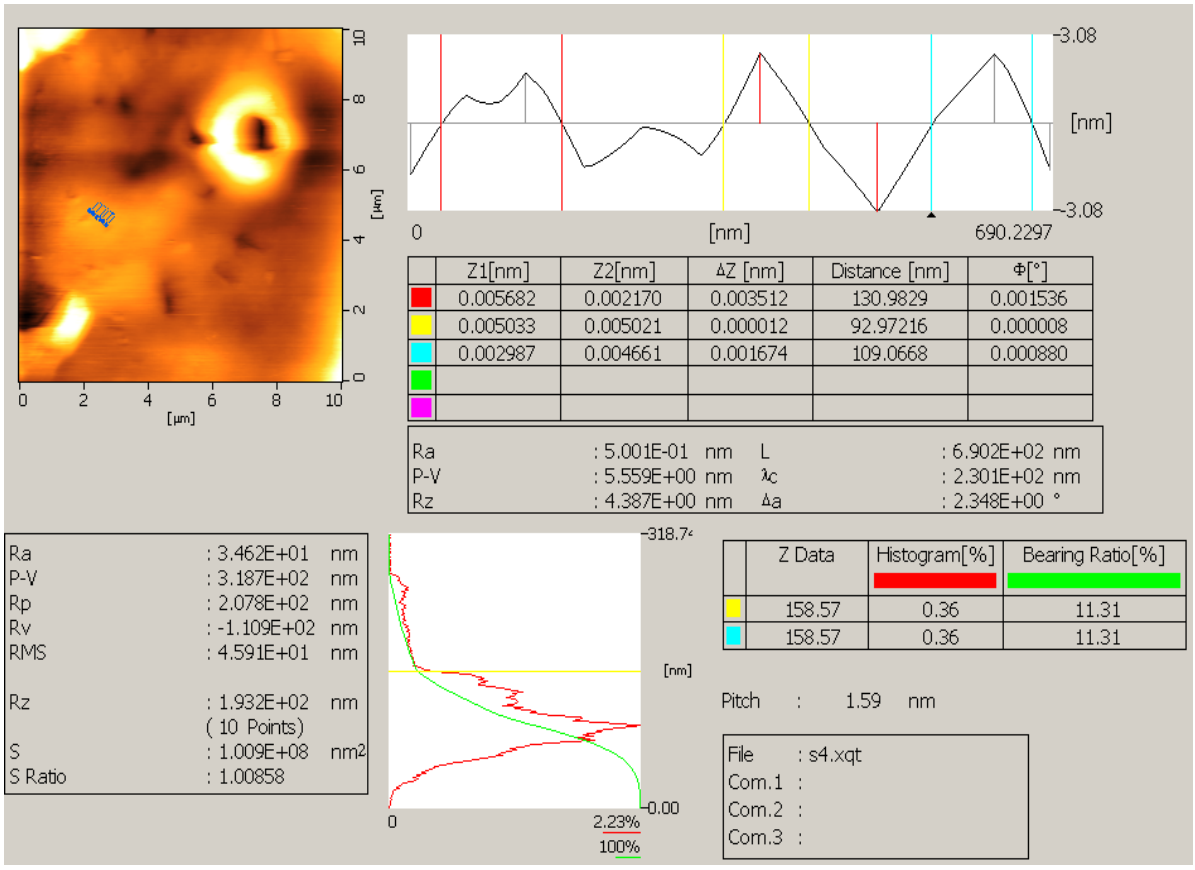



Table 4.9c AFM of Untreated Luffa Gourd Composites

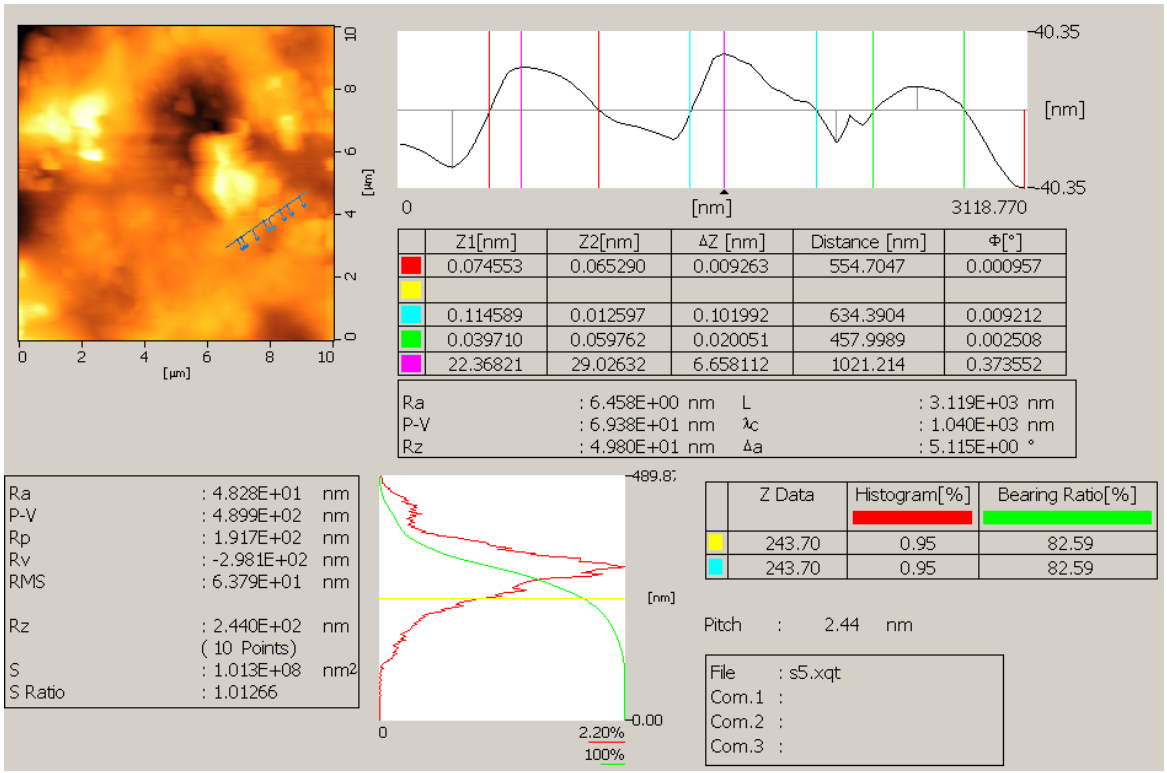



Table 4.9d AFM of Treated Luffa Gourd Composites

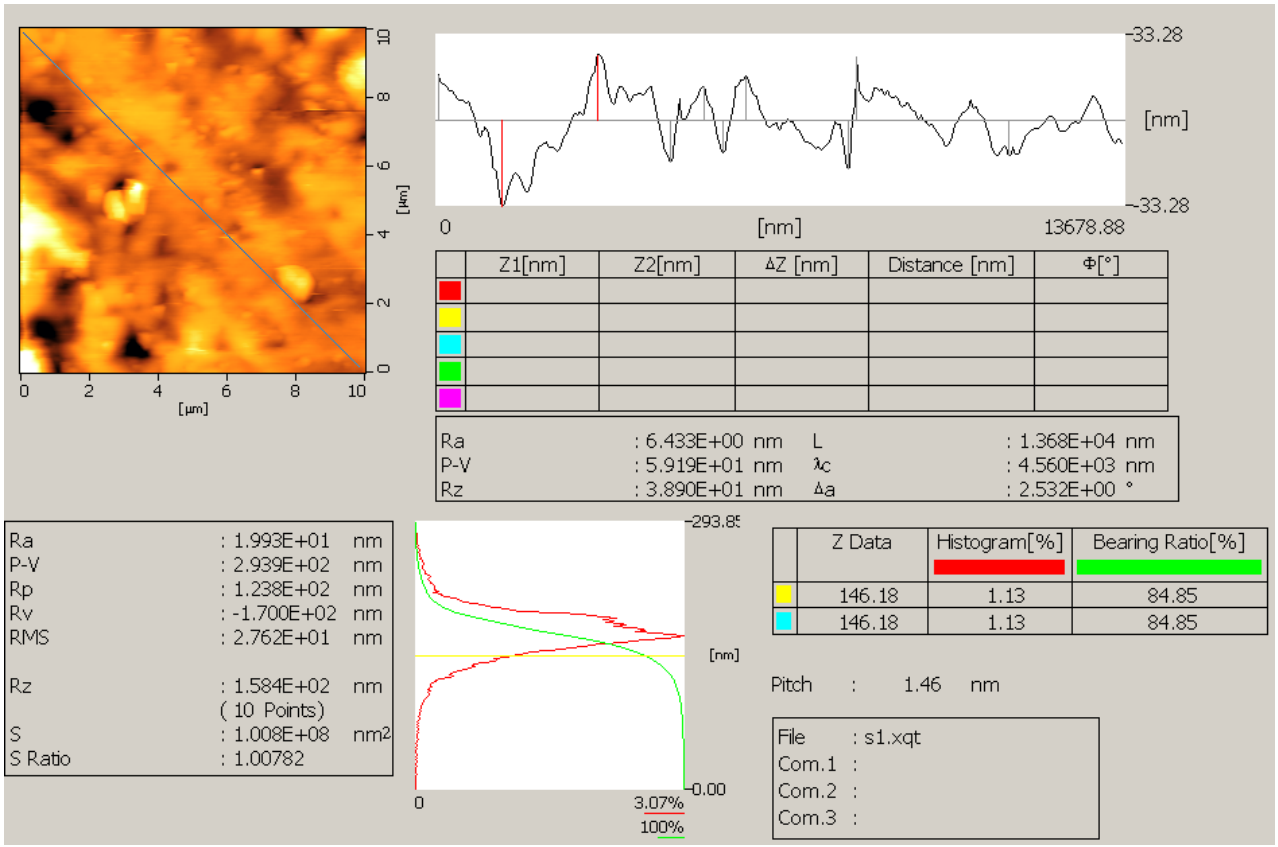



Table 4.9e AFM of Untreated Dum palm Composites

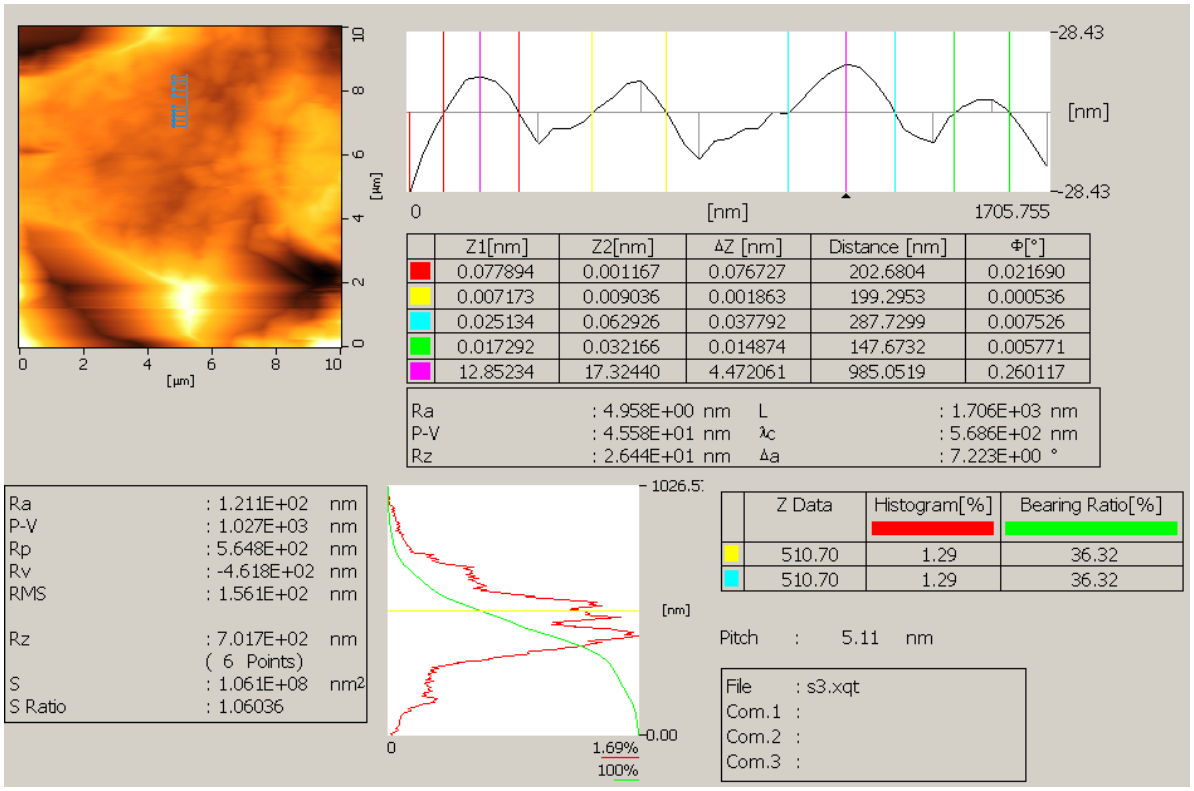



Table 4.9f AFM of Treated Dum palm Composites

| **Composites** | **Height(nm)** | **Distance (nm)** | **RMS (nm)** |
|---|---|---|---|
| Untreated Baobab | 0.006100 | 152 | 1.24E+02 |
| Treated Baobab | 0.006537 | 203 | 7.768E+02 |
| Untreated Luffa gourd | 0.003512 | 130 | 4.591E+01 |
| Treated Luffa gourd | 0.00923 | 554 | 6.379E+08 |
| Untreated Luffa gourd | 0.02289 | 180 | 2.762E+01 |
| Treated Luffa gourd | 0.076727 | 202 | 1.561E+02 |

**Table 4. 10: Analysis of AFM of Composites**

| | No of peak(s) | On set temperature(0c) | Peak deg. Temperature(0c) | Final deg. Temperature(0c) | Residue (%) |
|---|---|---|---|---|---|
| UDF | 1 | 275 | 350 | 420 | 5 |
| TDF | 6 | 150 | 350 | 410 | 5 |
| UBF | 2 | 180 | 350 | 425 | 5 |
| TBF | 3 | 125 | 360 | 425 | 5 |
| ULF | 1 | 275 | 360 | 425 | 5 |
| TLF | 1 | 275 | 350 | 425 | 5 |

**Table 4. 11 TGA analyses of composites**



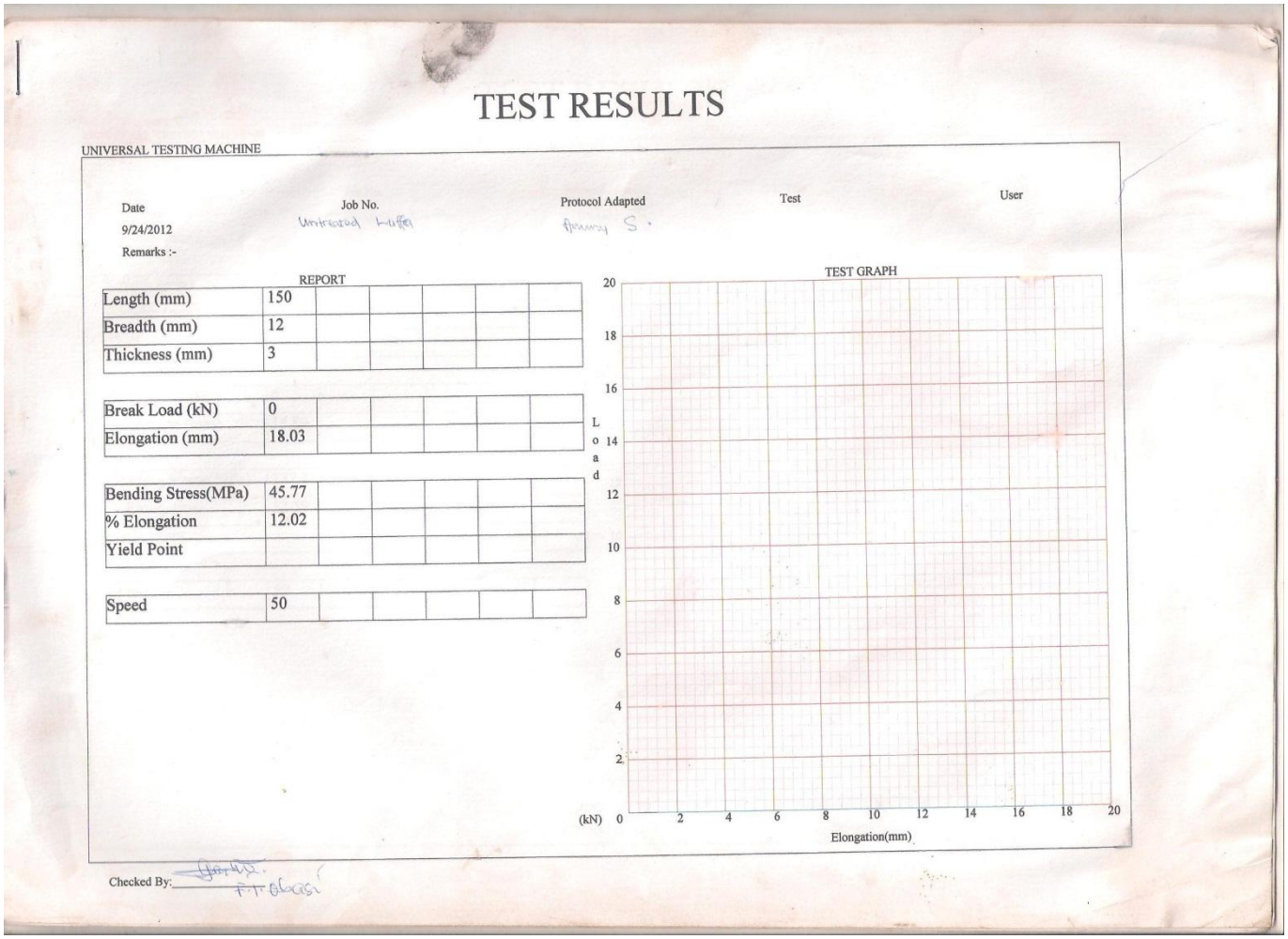
Figure M 1: Retted fibres inside container




Figure M 2: Water bath




Figure M3: Analytical balance



TEST RESULTS

UNIVERSAL TESTING MACHINE

Date: 9/24/2012 | Job No.: Untreated Luffa | Protocol Adapted: Ammu S. | Test: | User:

Remarks :-

REPORT

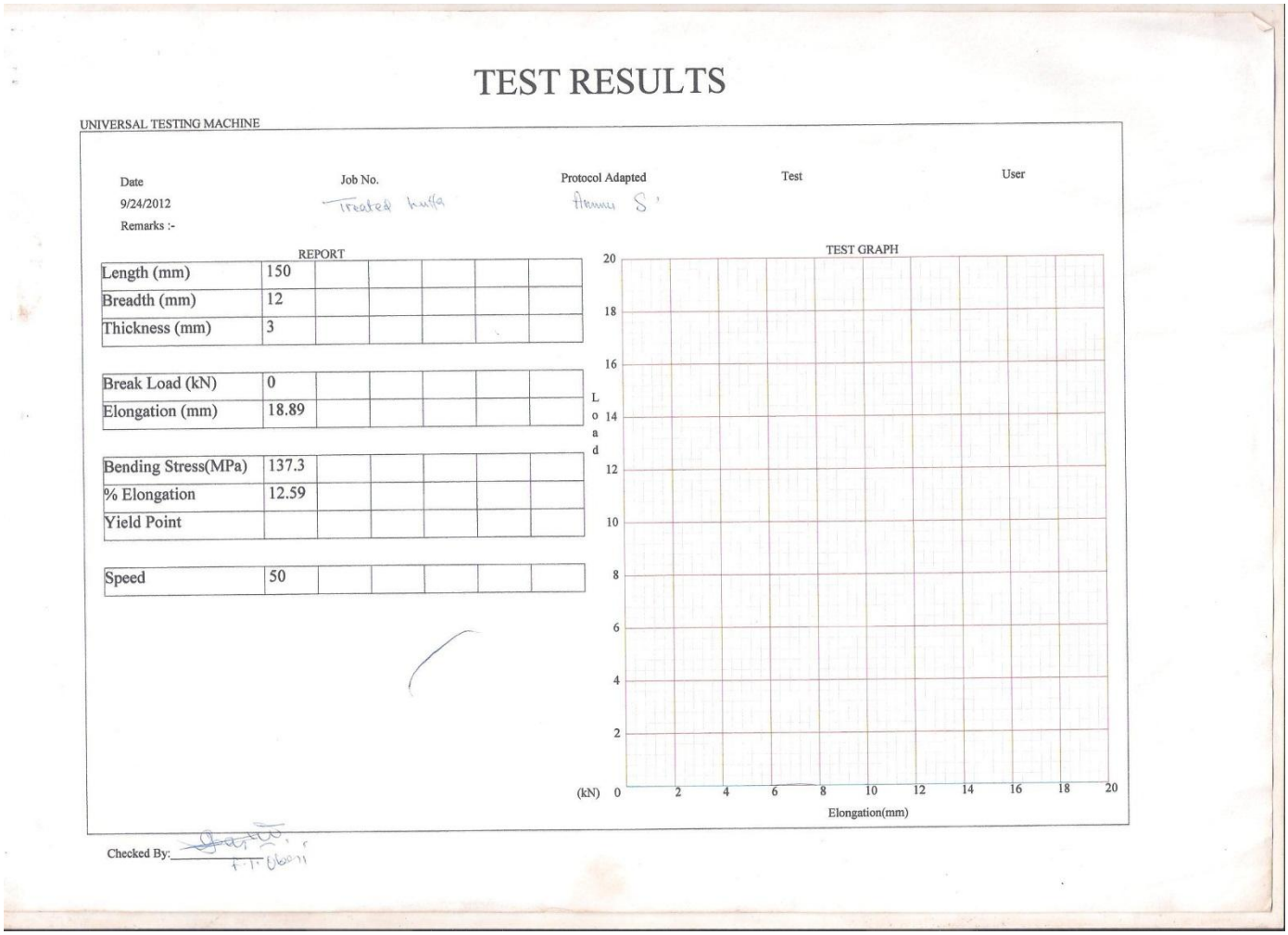
| | |
|---|---|
| Length (mm) | 150 |
| Breadth (mm) | 12 |
| Thickness (mm) | 3 |
| Break Load (kN) | 0 |
| Elongation (mm) | 18.03 |
| Bending Stress(MPa) | 45.77 |
| % Elongation | 12.02 |
| Yield Point | |
| Speed | 50 |

TEST GRAPH




Checked By:

Figure M 4: Untreated Luffa gourd composites



TEST RESULTS

UNIVERSAL TESTING MACHINE

Date: 9/24/2012 | Job No.: Treated luffa | Protocol Adapted: Flexure S' | Test: | User:

Remarks :-

REPORT

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Length (mm) | 150 | | | | | | |
| Breadth (mm) | 12 | | | | | | |
| Thickness (mm) | 3 | | | | | | |
| Break Load (kN) | 0 | | | | | | |
| Elongation (mm) | 18.89 | | | | | | |
| Bending Stress(MPa) | 137.3 | | | | | | |
| % Elongation | 12.59 | | | | | | |
| Yield Point | | | | | | | |
| Speed | 50 | | | | | | |

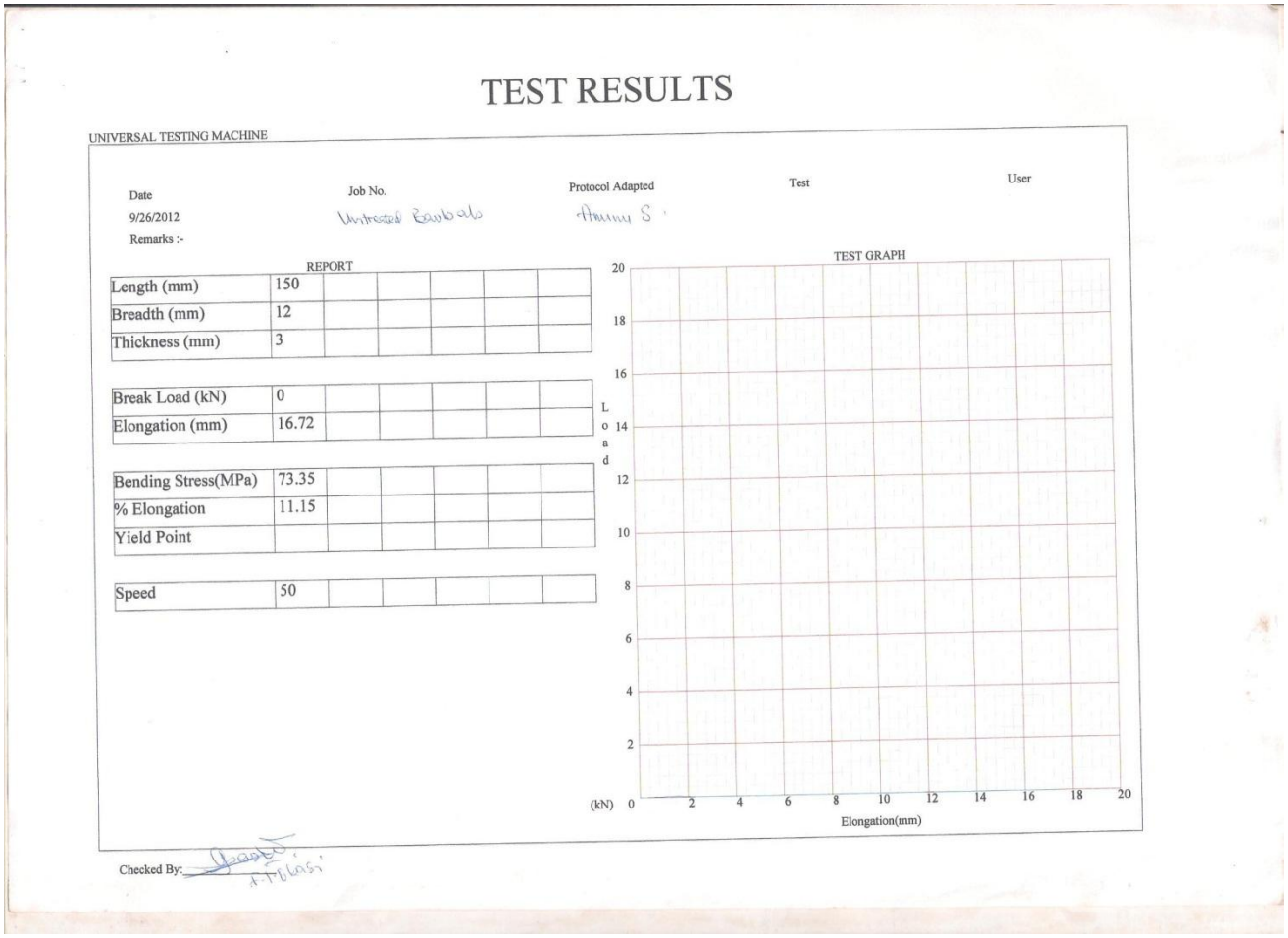
TEST GRAPH

Load (kN): 0, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20

Elongation(mm): 0, 2, 4, 6, 8, 10, 12, 14, 16, 18, 20

Checked By:

Figure M 5: Treated Luffa gourd composites



# TEST RESULTS

UNIVERSAL TESTING MACHINE

| Date | Job No. | Protocol Adapted | Test | User |
|---|---|---|---|---|
| 9/26/2012 | Untreated Baobab | Ahmu S. | | |

Remarks :-

REPORT

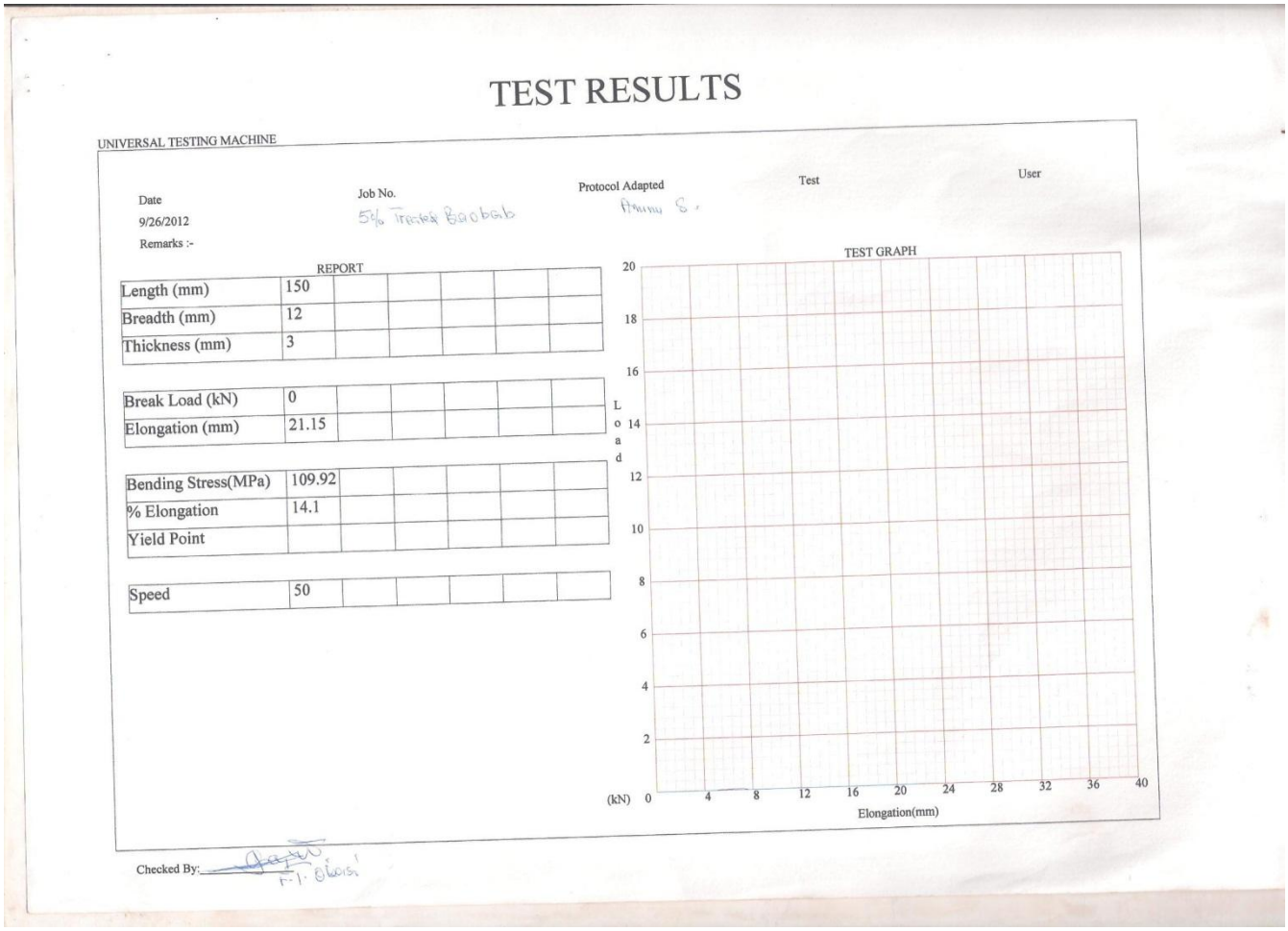
| | |
|---|---|
| Length (mm) | 150 |
| Breadth (mm) | 12 |
| Thickness (mm) | 3 |
| Break Load (kN) | 0 |
| Elongation (mm) | 16.72 |
| Bending Stress(MPa) | 73.35 |
| % Elongation | 11.15 |
| Yield Point | |
| Speed | 50 |

TEST GRAPH




Checked By:

Figure M 6: Untreated Baobab composites



# TEST RESULTS

UNIVERSAL TESTING MACHINE

Date: 9/26/2012

Job No.: 5% Treated Baobab

Protocol Adapted: ASTM S.

Test

User

Remarks :-

REPORT

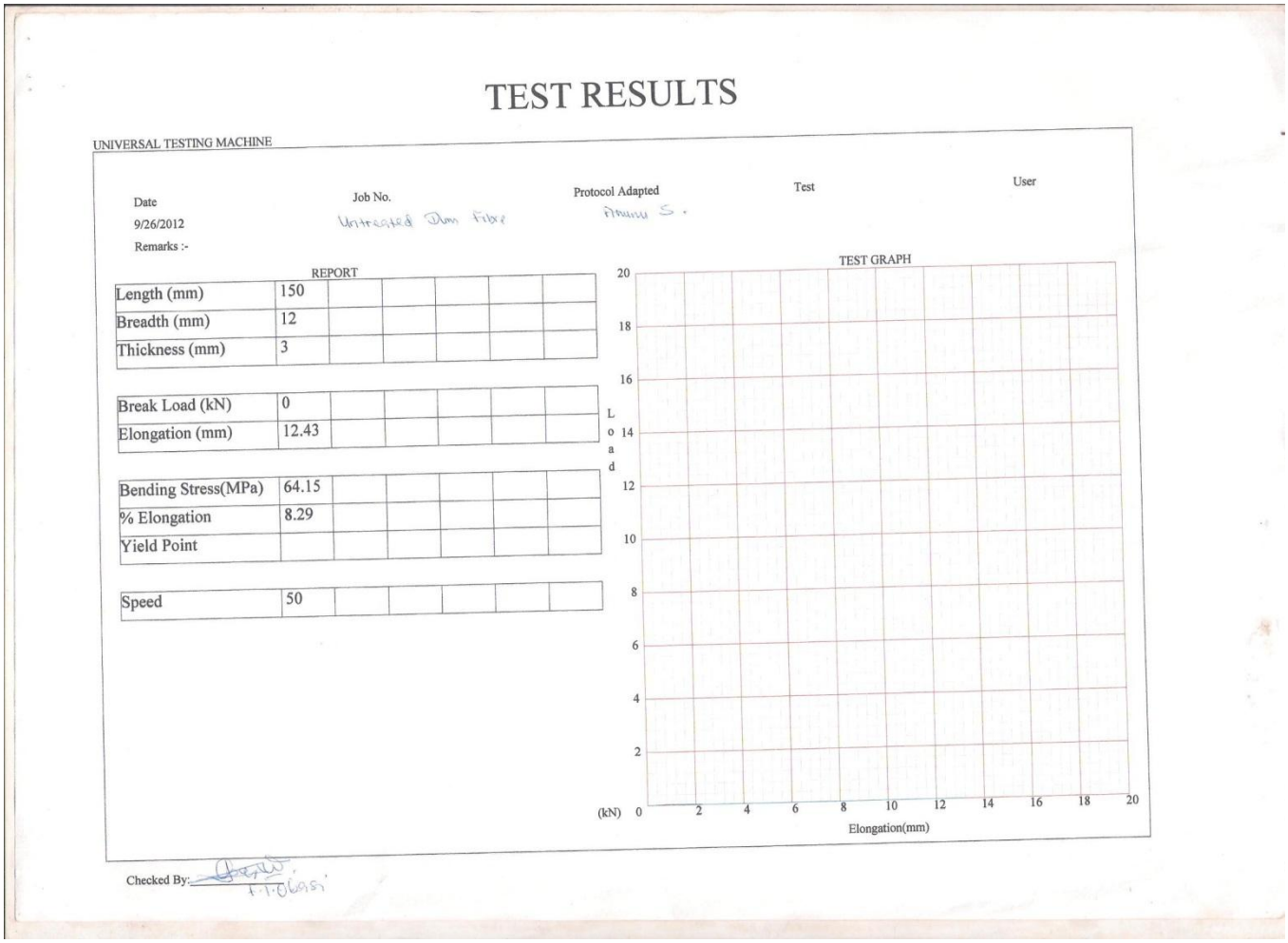
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Length (mm) | 150 | | | | | | |
| Breadth (mm) | 12 | | | | | | |
| Thickness (mm) | 3 | | | | | | |
| Break Load (kN) | 0 | | | | | | |
| Elongation (mm) | 21.15 | | | | | | |
| Bending Stress(MPa) | 109.92 | | | | | | |
| % Elongation | 14.1 | | | | | | |
| Yield Point | | | | | | | |
| Speed | 50 | | | | | | |

TEST GRAPH



Checked By: F.I. Owoi

Figure M 7: Treated Baobab composites



TEST RESULTS

UNIVERSAL TESTING MACHINE

Date: 9/26/2012 | Job No.: Untreated Dum Fibre | Protocol Adapted: Manu S. | Test | User

Remarks :-

REPORT

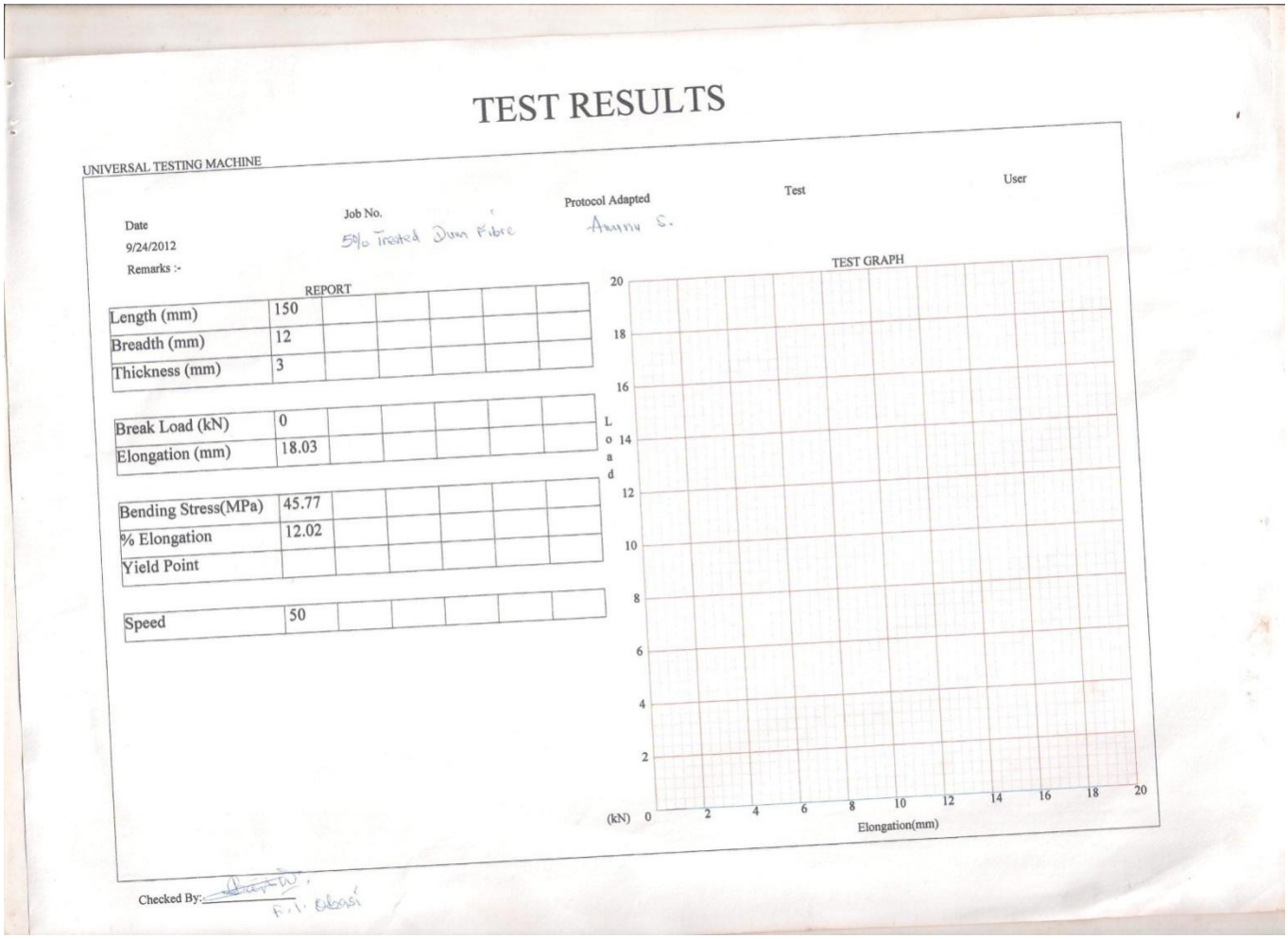
| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Length (mm) | 150 | | | | | | |
| Breadth (mm) | 12 | | | | | | |
| Thickness (mm) | 3 | | | | | | |
| Break Load (kN) | 0 | | | | | | |
| Elongation (mm) | 12.43 | | | | | | |
| Bending Stress(MPa) | 64.15 | | | | | | |
| % Elongation | 8.29 | | | | | | |
| Yield Point | | | | | | | |
| Speed | 50 | | | | | | |

TEST GRAPH



Checked By:

Figure M 8: Untreated Dum palm composites



TEST RESULTS

UNIVERSAL TESTING MACHINE

Date: 9/24/2012

Job No.: 5% Treated Dum Fibre

Protocol Adapted: Ammy C.

Test

User

Remarks :-

REPORT

| | | | | | | |
|---|---|---|---|---|---|---|
| Length (mm) | 150 | | | | | |
| Breadth (mm) | 12 | | | | | |
| Thickness (mm) | 3 | | | | | |
| Break Load (kN) | 0 | | | | | |
| Elongation (mm) | 18.03 | | | | | |
| Bending Stress(MPa) | 45.77 | | | | | |
| % Elongation | 12.02 | | | | | |
| Yield Point | | | | | | |
| Speed | 50 | | | | | |

TEST GRAPH



Checked By: F.I. Obasi

Figure M 9: Treated Dum palm composites



Figure M 10:  Dum palm bundle fibres



Figure M11: Baobab bundle fibres

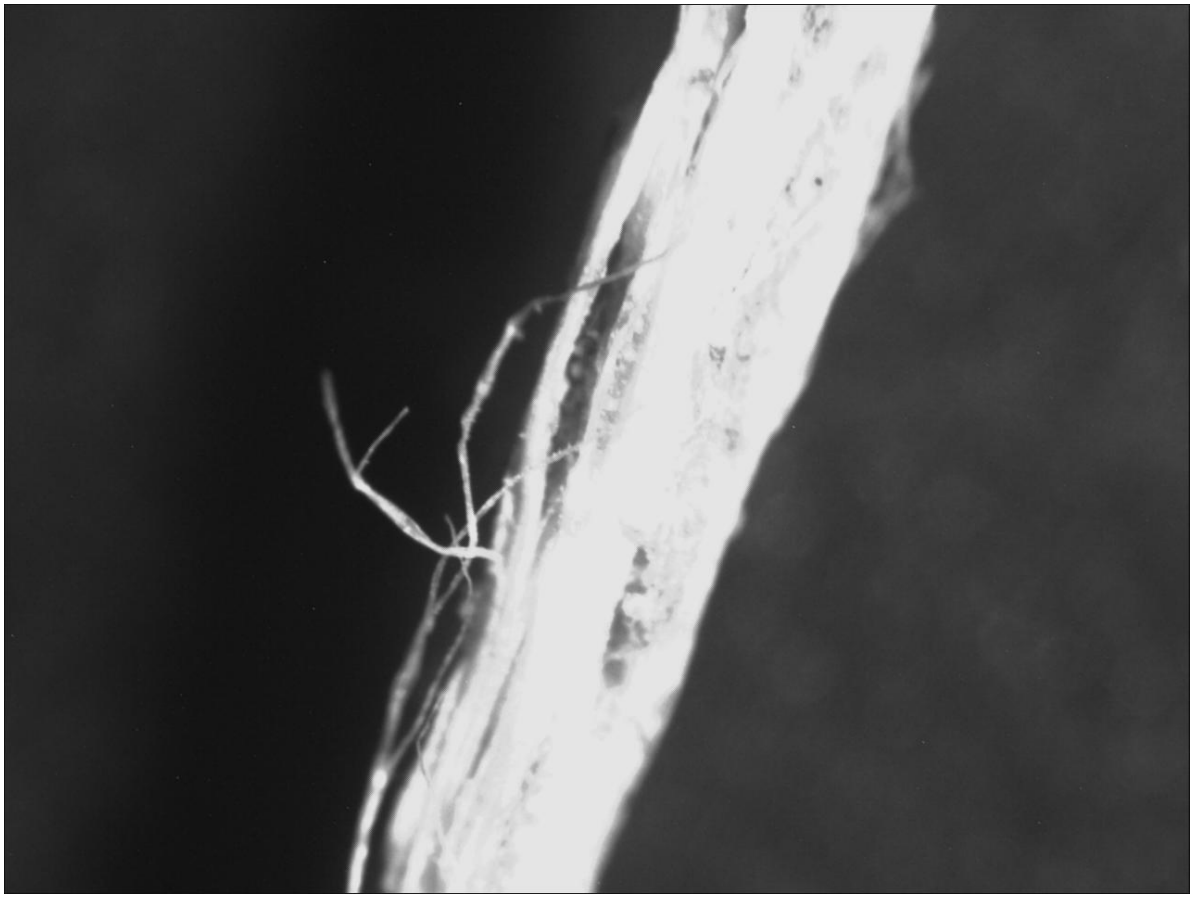


Figure M12: Baobab fibre viewed under high resolution microscope

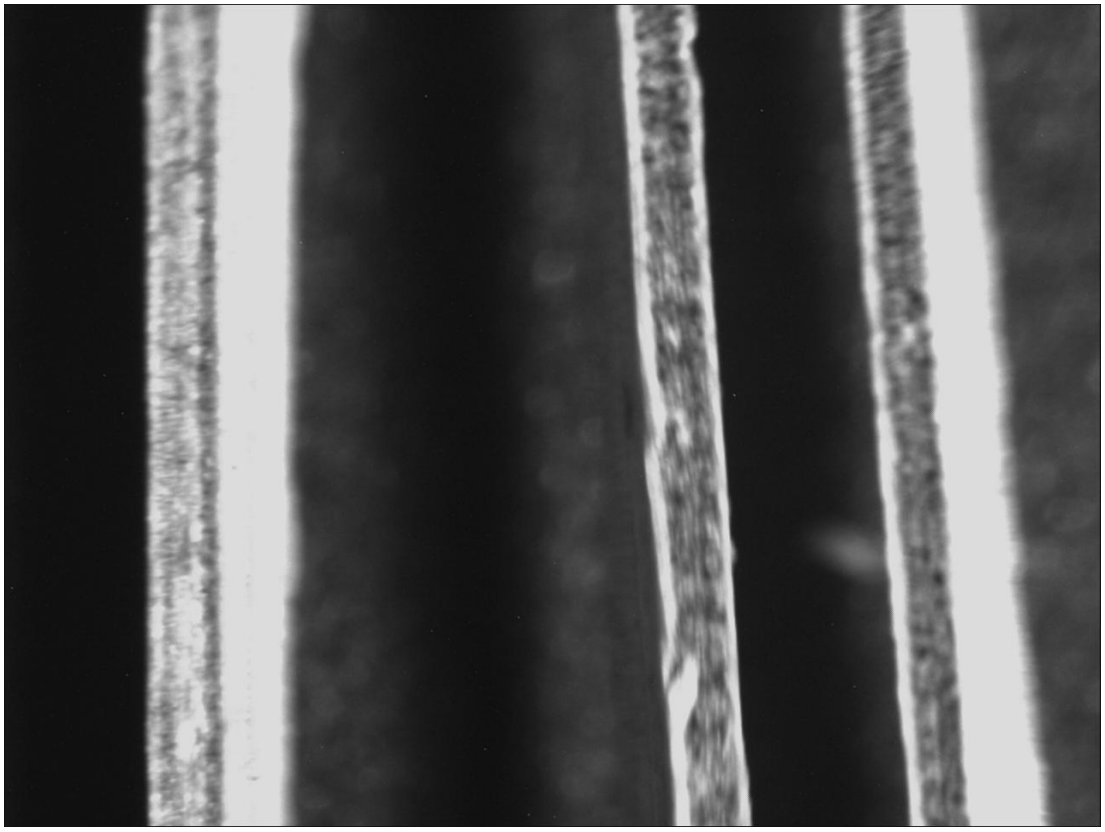


Figure M13: Dum palm fibres viewed under high resolution microscope

## APPENDIX B

The relation determines the degree of crystallinity according to Hermans and veidinger,

$$K = 100\, Sc/S$$

S = is the total area

Sc = is the area of the crystalline field.

But S is to be replaced by M and Sc by Mc

Where M = the weight of the total area

Mc = the weight of the crystalline area.

Hence,

$$K = 100\, Mc/M$$

**For Untreated Baobab fibre**,

Ma – Weight of Amorphous area = 0.0239g

Mc – Weight of Crystalline area = 0.0253g

M –Weight of Total area = 0.0492g

Kc = 0.0239/0.0492 x 100 = 48.57%

**For Untreated Dumpalm fibre**

Ma – Weight of Amorphous area = 0.0159g

Mc – Weight of Crystalline area = 0.0393g

M –Weight of Total area = 0.0552g

Kc = 0.0393/0.0552x 100 = 71.2%

**Untreated Luffa gourd fibre**

Ma –Weight of Amorphous area = 0.0157g

Mc – Weight of Crystalline area = 0.0359g

M – Weight of Total area = 0.0516g

Kc  0.0359/0.0516 x100 = 69.59%

**For treated fibres**

**For Treated Baobab fibre composites**

Ma =0.0230g

Mc = 0.0198g

M = Total area = 0.0428g

Kc = 0.0198/0.0428x 100 = 46.26%

**For Treated Dum palm fibre composites**

Ma = 0.0164g

Mc = 0.0323g

M =Total area = 0.0428g

Kc = 0.0323/0.0428 x 100 = 66.32%

**For Untreated Luffa gourd fibre composite**

Ma = 0.0159g

Mc = 0.0303g

M =Total area = 0.0462g

Kc = 0.0303/0.0462 x100 = 65.58%