

**DEVELOPMENT AND CHARACTERISATION OF EPOXY/BORASSUS PALM
(*Borassus aethiopum* Mart.) LEAF STALKFIBRE REINFORCED
COMPOSITE**

BY

AYO SAMUEL ALABI

**THE DEPARTMENT OF METALLURGICAL AND MATERIALS
ENGINEERING,
FACULTY OF ENGINEERING,
AHMADU BELLO UNIVERSITY,
ZARIA**

FEBRUARY, 2018

**DEVELOPMENT AND CHARACTERISATION OF EPOXY/BORASSUS PALM
(*Borassus aethiopum* Mart.) LEAF STALK FIBRE REINFORCED
COMPOSITE**

BY

**Ayo Samuel ALABI, B. ENG. (ABU) 2009
P15EGML8007**

**A THESIS SUBMITTED TO THE SCHOOL OF POSTGRADUATE STUDIES,
AHMADU BELLO UNIVERSITY, ZARIA IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE AWARD OF MASTER OF SCIENCE IN
METALLURGICAL AND MATERIALS ENGINEERING**

**DEPARTMENT OF METALLURGICAL AND MATERIALS ENGINEERING,
FACULTY OF ENGINEERING,
AHMADU BELLO UNIVERSITY,
ZARIA, NIGERIA**

FEBRUARY, 2018

Declaration

I declare that the work in this thesis,entitled: “Development and Characterisation of Epoxy/borassus Palm (*Borassus aethiopum* Mart.) Leaf Stalk Fibre Reinforced Composite” has been carried out by me in the Department of Metallurgical and Materials Engineering. Information from literatures were duly acknowledged in the text and a list of references provided. No part of this work was previously presented for another degree or diploma at this or any other institution.

Ayo Samuel ALABI _____
(Student) Signature Date

Certification

This thesis, entitled: “Development and Characterisation of Epoxy/Borassus Palm (*Borassus aethiopum* Mart.) Leaf Stalk Fibre Reinforced Composite” by Ayo Samuel ALABI meets the regulations governing the award of M.Sc. degree in Metallurgical and Materials Engineering of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

Dr. U. Shehu (Signature) _____ Date

(Chairman, Supervisory Committee)

Prof. T. Ause (Signature) _____ Date

(Member, Supervisory Committee)

Dr. A. Kasim (Signature) _____ Date

(Head of Department)

Prof. S.Z. Abubakar (Signature) _____ Date

(Dean, School of Postgraduate Studies)

Acknowledgements

All praise and thanks to God for the gift of life, good health and His enablement for the success of this work. I count it a privilege to have been supervised by Dr. U. Shehu and Prof. T. Ause to whom my profound gratitude goes for guiding me and offering meaningful contributions, making valuable suggestions and prompt corrections of my various write-ups, May God reward them abundantly.

I wish to express my candid appreciation to the Head of Department, Dr. A. Kasim, for his encouragement, assistance, advice and significant contributions. My sincere appreciation also goes to the entire staff of the Department of Metallurgical and Materials Engineering, notable among whom are Dr. M. Abdulwahab, Mr. I. Usman, Mr. S. A. Abubakar, Mr. Y. Mohammed for their counsel, assistance, support and contribution to the success of this study.

The immense assistance rendered by colleagues and friends at various stages of this work are appreciated. Names that readily came to mind include Mal. Hashimu Dankasa, Mr. I. N. Mbada Pst. Cletus Emeka and Mr. Afam Ani. May God bless and reward every one of them richly for contributing to the success of this study.

I owe gratitude and appreciation to Prof. J. O. Ayo for his selfless effort, advice and encouragement in persuading me to embark on this feat at the moment of my discouragement. May the good Lord reward his kindness.

Lastly, I acknowledge the support and encouragement of my lovely wife, Mrs. Omotola Alabi and daughters, Ailsa and Natanela. Thank them for their patience and understanding. I love them all.

Abstract

The search for new engineering materials that addresses ecological and environmental issues is generating renewed interest in utilisation of agricultural wastes to explore value-added application avenues. The aim of the study was to develop and characterise epoxy/borassus palm (*Borassus aethiopum* Mart.) leaf stalk fibre (BPLSF) reinforced composite to establish the potential of BPLSF as a reinforcement in epoxy, a thermosetting polymeric material, for maritime craft applications. Fibres were extracted from the stalk by water retting and treated with sodium hydroxide (NaOH) and natural latex coating. The fibre was characterised using Fourier Transform Infrared Spectroscopy and Scanning Electron Microscopy (SEM). The composites were produced by hand lay-up method, aligning the continuous fibres longitudinally. The composites produced were characterised by density evaluation and mechanical testing such as tensile, flexural, impact and hardness tests. Others included water absorption test, soil burial and natural weather exposure tests using weight gain or loss as criteria to evaluate the degradation of the composites. Dynamic mechanical analysis (DMA) was used to study the thermo-mechanical properties of the composites. SEM was used to study the impact fractured surfaces and water absorption specimen surfaces. Average fibre strengths of 1250 MPa, 1040 MPa and 820 MPa were obtained for untreated, alkali treated and latex treated fibres, respectively. The density results for untreated, alkali treated and latex treated fibre reinforced composites lie between 0.7967 g/cm³ and 1.0381 g/cm³, 0.8827 g/cm³ and 1.0498 g/cm³ and 0.9192 g/cm³ and 1.0786 g/cm³, respectively. Density decreased generally as fibre contents increased. Tensile test results showed a general increase in tensile strengths and moduli with increasing fibre contents. Epoxy exhibited tensile strength of 23.69 MPa compared to the composites having maximum tensile strengths at 25 wt. % fibre contents of 53.59 MPa for untreated, 55.74 MPa for alkali treated and 50.48 MPa for latex treated fibre reinforced composites. Corresponding tensile moduli for epoxy and composites at 25 wt. % fibre contents were 256.11 MPa, 1363.79 MPa, 1258.50 MPa and 1431.72 MPa, respectively. The morphological studies of impact fractured surfaces revealed brittle fracture for unreinforced matrix but ductile failure for the various composites. The water absorption tests showed a general increase in water absorption with increasing fibre content, the maximum water absorption at 25 wt% fibre contents were 51.3% for untreated, 19.4% for alkali treated, and 50.2% for latex treated fibre reinforced composites in river water; while in distilled water, lower values for epoxy and all composites were obtained compared to those in river water. Morphological study of water absorption sample surfaces revealed fibre debonding for untreated fibre and loss of surface integrity for latex coated fibre-reinforced composites. DMA results showed a decrease in storage and loss modulus with increasing fibre content, tangent delta reduced with fibre content. It was concluded that BPLSF is a potential reinforcement for epoxy matrix and may be beneficial in complementing/replacing the man-made fibre.

Table of Contents

Declaration.....	iii
Certification.....	iv
Acknowledgements.....	v
Abstract.....	vi
Table of Contents.....	vii
List of Figures.....	x
List of Tables.....	xi
List of Plates.....	xii
List of Appendices.....	xiii
List of Abbreviations.....	xv

CHAPTER ONE: INTRODUCTION

1.1 Background of the Study.....	1
1.2 Statement of Research Problem.....	3
1.3 Aim and Objectives.....	4
1.4 Justification of the Research.....	5
1.5 Scope of Work.....	5
1.6 Contribution to Knowledge.....	6

CHAPTER TWO : LITERATURE REVIEW

2.1 Composite Material.....	7
2.2 Polymers.....	8
2.2.1 Thermoplastics.....	10
2.2.2 Thermosetting polymers.....	10
2.2.3 Elastomers.....	10
2.3 Polymer matrix composites (PMCs).....	11
2.4 Epoxy Resins.....	12
2.5 Natural Fibres.....	14
2.5.1 Classification of natural fibres.....	15
2.5.2 Ligno-cellulosic fibres.....	16
2.6 Modification of Natural Fibres.....	18
2.6.1 Physical methods.....	18

2.6.2 Chemical modification and use of adhesion promoters.....	20
2.7 Retting	23
2.7.1 Dew (field) retting	24
2.7.2 Water retting.....	24
2.7.3 Stand retting	25
2.7.4 Chemical retting.....	25
2.7.5 Enzyme retting	25
2.7.6 Other retting processes.....	26
2.8 <i>Borassus aethiopum</i> Mart. <i>Areaceae</i>	27
2.9 Mechanical Properties of Natural Fibre Reinforced Polymer Composites	29
2.10 Water Absorption Properties of Natural Fibre Reinforced Polymer Composites	30
2.11 Thermal Properties of Natural Fibre Reinforced Polymer Composites	31
2.11.1 Dynamic mechanical analysis (DMA).....	31
2.12 Degradation of Natural Fibre Reinforced Polymer Composites	32
2.13 Review of Previous Works.....	33
CHAPTER THREE: MATERIALS AND METHODS	
3.1 Materials.....	38
3.3 Methodology.....	41
3.3.1 Water retting.....	41
3.3.2 Surface treatment	41
3.3.3 Production of epoxy/BPLSF reinforced composite	42
3.3.4 Curing condition	42
3.4 Physical Properties.....	43
3.4.1 Determination of density	43
3.4.2 Water absorption test.....	43
3.4.3 Degradability test.....	43
3.5 Mechanical Tests	44
3.5.1 Single fibre tensile test.....	45
3.5.2 Tensile test.....	45
3.5.3 Flexural test.....	46
3.5.4 Impact test.....	47
3.5.5 Hardness test	48

3.6 Single Fibre Diameter Measurement.....	49
3.7 Dynamic Mechanical Analysis (DMA)	49
3.8 Scanning Electron Microscopy (SEM)	50
3.9 Fourier Transform Infrared (FTIR) Spectroscopy	50
CHAPTER FOUR: RESULTS AND DISCUSSION	
4.1 Fibre Characterisation	52
4.1.1 Fibre diameter measurement	52
4.1.2 Single fibre strength	52
4.1.3 Fourier transform infrared spectroscopy (FTIR) characterisation	54
4.1.4 Fibre morphology	60
4.2 Composite Characterisation.....	61
4.2.1 Physical properties.....	61
4.2.2 Mechanical properties	68
4.2.3 Degradability test.....	78
4.2.4 Thermo-mechanical Analysis	83
CHAPTER FIVE: CONCLUSION AND RECOMMENDATION	
5.1 Conclusion	88
5.2 Recommendation	89
REFERENCES	90

List of Figures

Figure 4.1: Average fibre strength at fracture and fibre diameter.....	52
Figure 4.2: FTIR spectrum for untreated fibre.....	55
Figure 4.4: FTIR spectrum for latex treated fibre.....	57
Figure 4.5: Effect of fibre content (wt%) on the density of treated and untreated fibre reinforced composite.....	61
Figure 4.6: Effect of distilled water exposure duration (hrs) on under absorption of 5 wt% treated and untreated fibre reinforced composite.....	63
Figure 4.8: Effect of river water exposure duration (hrs) on water absorption of 5 wt% treated and untreated fibre reinforced composite.....	64
Figure 4.9: Effect of river water exposure duration (hrs) on water absorption of 25 wt% treated and untreated fibre reinforced composite.....	64
Figure 4.11: Effect of fibre content (wt.%) on tensile modulus of treated and untreated fibre reinforced composite.....	70
Figure 4.12: Effect of fibre content (wt.%) on flexural strength of treated and untreated fibre reinforced composite.....	71
Figure 4.13: Effect of fibre content (wt.%) on flexural modulus of treated and untreated fibre reinforced composite.....	72
Figure 4.14: Effect of fibre content (wt.%) on impact strength of treated and untreated fibre reinforced composite.....	73
Figure 4.15: Effect of fibre content (wt.%) on hardness value of treated and untreated fibre reinforced composite.....	76
Figure 4.16: Effect of fibre content (wt.%) on weight loss of treated and untreated fibre reinforced composites buried for 90 days in harmatan season.....	78
Figure 4.17: Effect of fibre content (wt.%) on weight loss of treated and untreated fibre reinforced composite buried for 90 days in rainy season.....	79
Figure 4.19: Effect of fibre content (wt.%) on weight gain of treated and untreated fibre reinforced composite exposed to natural weather for 90 days in rainy season.....	82
Figure 4.20: Storage modulus against temperature of epoxy and 5 wt.% composites.....	83
Figure 4.21: Loss modulus against temperature of epoxy and 5 wt.% composites.....	83
Figure 4.22: Tan δ against temperature of epoxy and 5 wt.% composites.....	84
Figure 4.24: Loss modulus against temperature of epoxy and 25 wt.% composites.....	85
Figure 4.25: Tan δ against temperature of epoxy and 25 wt.% composites.....	85

List of Tables

Table 2.1a: Review of Previous Works and Findings	36
Table 3.1a: List of Equipment Used in the Study	39
Table 4.1: Untreated Fibre Functional Groups, Nature and Types of Vibration.....	58
Table 4.2: Alkaline Treated Fibre Functional Groups, Nature and Types of Vibration	58
Table 4.3: Latex Treated Fibre Functional Groups, Nature and Types of Vibration	59

List of Plates

Plate I: <i>Borassus aethiopum M.</i> (a) trees; (b) leaf stalks	28
Plate II: Wooden moulds	42
Plate III: Fractured tensile test samples	46
Plate IV: Ruptured flexural test samples	47
Plate V: Impact test samples.....	48
Plate VI: Hardness test samples.....	49
Plate VII: Dynamic mechanical analysis samples	50
Plate VIII:Micrographs showing fibre diameter.....	51
Plate IX: SEM of fibre surfaces morphology	60
Plate X: SEM of Water Absorption Samples.....	67
Plate XI: SEM of impact fractured surfaces of composite	75

List of Appendices

Appendix A: Single Fibre Strength and Diameter..... 98
Appendix B: Density Values of the Composite Formulations 98
Appendix C: Water Absorption Test after 30 Days..... 99
Appendix D: Mechanical Properties of Composites..... 102
Appendix E: Degradability Test Data..... 105

List of Abbreviations

ACS	American Chemical Society
ASTM	American Standard of Testing Material
ATR	Attenuated Total Reflectance
BPA	Bisphenol A
BPLSF	Borassum palm leaf stalk fibre
CMCs	Ceramic matrix composites
CSP	Coconut shell powder
DGEBA	Diglycidyl ether of bisphenol A
DMA	Dynamic mechanical analysis
DMTA	Dynamic mechanical thermal analysis
DTA	Differential Thermal Analysis
DTG	Differential thermo gravimetry
EB	Electron beam
ECD	Epichlorohydrin
EDTA	Ethylenediaminetetraacetic acid
FTIR	Fourier transform infrared
ISO	International standards organization
LDPE	Low density polyethelene
MMCs	Metal matrix composites
MVH	Micro Vickers hardness
PBS	Polybutelene succinate
PC	Polycarbonate
PE	Polyethene
PEEK	Polyether ether ketone

pH	Potential of hydrogen
PLA	Poly lactic acid
PMCs	Polymer matrix composites
PP	Polypropylene
PS	Polystyrene
PVC	Polyvynyl chloride
RH	Rice husk
rHDPE	Recycled high-density polyethelene
RHF	Rice husk flour
SCB	Sugar cane bagasse
SEM	Scanning electron microscopy
TDI	Toluene-2,4-diisocyanate
T _g	Glass transition temperature
TGA	Thermo gravimetric analysis
TPU	Thermoplastic polyurethane
UP	Unsaturated polyester
UV	Ultraviolet

CHAPTER ONE

INTRODUCTION

1.1 Background of the Study

Interest is on the increase in the use of ligno-cellulosic fibre as potential replacement of its man-made counterpart for reinforcement in polymeric material. Increasing environmental awareness globally has imparted greatly on materials engineering and design. Renewed interest in the utilisation of natural materials addresses ecological issues such as recyclability, non-toxicity and environmental safety.

In the last two decades, interest in ligno-cellulosic fibres as polymeric reinforcements has greatly increased. Researchers in Asia, Europe and other parts of the globe have shown kin interest in this research area due the advantages of these fibres over their synthetic counterparts (Bledzki and Gassan, 1999; Ansell and Aziz, 2004; Tserki *et al.*, 2006; Abdelmoulehet *et al.*, 2009; Kabir *et al.*, 2012; Rassiah *et al.*, 2012; Fakhrul and Islam, 2013; Ramesh *et al.*, 2013). The advantages include low density, low cost, high specific strength and stiffness, low emission of toxic fumes when subjected to heat during incineration at the end of life and low hazard manufacturing processes (Pickering *et al.*, 2016).

Attempts have been made to use natural fibre in place of fibre-glass reinforced composites mostly in non-structural applications. So far, a good number of automotive components previously made with glass-fibre composites are now being manufactured using environmentally friendly composites (Wambua *et al.*, 2003).

Currently, synthetic fibres such as glass, carbon and aramid are widely used in polymer-based composites because of their high stiffness and strength properties. However, these

Comment [R21]: Please provide references for these researches

fibres have serious draw-backs in terms of their biodegradability, initial processing costs, recyclability, energy consumption, machine abrasion and health hazards (Kabiret *al.*, 2012). The draw-backs have contributed to shifting research focus from synthetic fibres to natural/renewable fibres. Natural fibres from renewable resources are now popularly used as reinforcements in polymer matrices. They provide benefits to the environment with respect to degradability and utilisation of natural materials (Kabiret *al.*, 2012). Today, the growing environmental awareness throughout the world has triggered a paradigm shift from synthetic fibres and their composites to composites made from natural reinforcing constituents; natural fibre and natural particulate fillers, which are more environmental friendly (Araoye, 2015).

Commercial and industrial applications of fibre-reinforced polymer composites include aircraft, space, military, automobile, sporting goods, marine, oil industry, medicine, oxygen tanks, and power transmission shafts to mention but a few. Glass fibre-reinforced polyesters have been used in different types of boats; for example, sail boats, fishing boats, dinghies, life boats, yachts and ships, ever since their introduction as commercial materials in the 1940s. Today, nearly 90% of all recreational boats are constructed from either glass fibre-reinforced polyester or glass fibre-reinforced vinyl ester resin. Among the applications are hulls, decks, and various interior components (Mallick, 2007).

Boats and canoes in most riverine settlements in Nigeria are exclusively used for fishing, transportation, sand digging and recreation. Most canoes used in the lagoon are generally tied to the planked jetties and left in water infested with insects, such as marine borers, beetles, termites, carpenter ants and ship worms, and microbes (algae, bacteria and fungi) throughout the year. The wood absorbs a lot of water, which adds more to the weight and

eventually reduces the speed of the canoe when propelled (Emmanuel,2010). Insects and microbes attack differently the three main chemical components of wood: cellulose, hemicellulose and lignin, causing wood-degradation (rot and decay) by their metabolic activities, leading to decrease in strength or utter destruction (Shupeet *al.*, 2016).

Application of fibre reinforced polymer composites to maritime crafts was initially driven by a need for lightweight (particularly the topside weight of ships), strong, corrosion resistant and durable materials. Most of the early applications were driven by the need to overcome corrosion problems experienced with steel or aluminum alloys and environmental degradation suffered by wood. The high acoustic transparency of composites also resulted in their use in ships and sonar domes on submarines(Selvaraju and Ilaiyavel,2011).

1.2 Statement of Research Problem

Most developing countries like Nigeria are yet to explore the potential that renewable agricultural by-product portends for gainful engineering production. This is why the best that could be done with the fibre-rich borassus palm leaf stalk waste in the environment is to burn as firewood which further aggravates the greenhouse effect on our changing climate. Therefore, there is need to develop more ways of reducing the amount of the waste in the environment, but of course, not by burning. One of the easiest ways of solving the problem is to explore value-added avenue of using borassus palm leaf stalk fibre as a reinforcement in epoxy matrix to form epoxy/borassus palm leaf stalk fibre reinforced composite. Epoxy-based composites can be used in marine applications such as ship and boat hulls, decks, propellers and interiors. Researchers are faced with the problem of developing materials that have low cost, light weight, eco-friendly and enhanced

mechanical properties for insatiable engineering applications. Metals which have good strength are insidiously affected by corrosion and heavy weight, likewise the use of wood is bedeviled with water absorption and degradation, making the search for alternative materials like epoxy-based composites that can be a substitute for metals/wood in marine/riverine applications inevitable. Fibre reinforced composites are gaining wide research interest due to their low-cost and light weight advantages over their counterpart synthetic fibre composites. In the light of this, and also in line with the current administration policy of looking inward and producing what is locally consumed, this research is geared towards developing and characterizing a light weight, eco-friendly, renewable and low-cost epoxy/borassus palm leaf stalk fibre reinforced composites with less water absorption and reduced deterioration with time. The composite may serve as an indigenous replacement to wood and metals used in maritime crafts, thereby alleviating the problems of riverine/marine communities.

1.3 Aim and Objectives

The aim of this research was to develop and characterise epoxy/BPLSF reinforced composite.

The objectives of the research to be pursued in achieving this aim were to:

- i. To carry out surface treatment and characterisation of extracted fibre from Borassus palm leaf stalk.
- ii. To develop epoxy matrix composite reinforced with treated and untreated BPLS fibre varying the composition from 5 – 25 wt% at 5wt% interval.

- iii. To examine the physical, mechanical and thermo-mechanical analysis of the epoxy/fibre reinforced composite.
- iv. To evaluate the degradability of the epoxy/BPLSF reinforced composite by soil burial and exposure to natural weather.

1.4 Justification of the Research

The high demand for light weight, eco-friendly, renewable and low-cost engineering materials has ushered in the use of natural fibres as reinforcements in polymeric matrices.

Therefore, the justifications for carrying out this research include:

- i. Low-cost and light-weight advantages of natural fibres in comparison to the synthetic fibres.
- ii. Addressing environmental and ecological issues since fibres are renewable resources.
- iii. Enhancing job creation since it will encourage production of more plants from which fibre could be extracted and also because of the low capital, energy and machinery requirement for composite production.
- iv. It is important to the society because it will enhance the development of a new material with better performance in riverine and marine environments.
- v. Boosting our indigenous technology.

1.5 Scope of Work

The scope of this research was to extract fibres from borassus palm leaf stalk by water retting, carry out surface treatments on some part of the extracted fibre and to characterise

the treated and untreated fibres. The work also study the fibre weight percent in the composite that gives optimum physical, mechanical and thermo-mechanical properties. This was followed by production of epoxy/BPLSF reinforced composite for riverine applications. Composite characterisation include physical (density determination and water absorption), mechanical (tensile, flexural, impact and hardness tests), degradability test (soil burial and exposure to natural weather) and thermo-mechanic test using dynamic mechanical analysis.

1.6 Contribution to Knowledge

The following contributions to knowledge were made by this work:

- i. The use of BPLSF as a reinforcement in polymeric material was established.
- ii. The extent to which fibre content wt.% affects physical, mechanical, degradability and thermo-mechanical properties of the composite was established.

CHAPTER TWO

LITERATURE REVIEW

2.1 Composite Material

Composite material can be defined as a combination of two or more monolithic materials that have been bonded together to maximise their useful properties and minimise their weaknesses. The term “composite material” collects under one heading a very large and now ubiquitous class of materials. Their common and defining trait is that they are combinations of phases that are bonded together instead of being created by phase transformation within bulk materials. Composites can therefore, contain widely different and interacting materials combined at a scale and with a micro-structural architecture that are controlled with considerable latitude. This enables generation of materials with special and controlled properties, making composites unique among materials (Mortensen, 2007).

It is the act of artificially bonding distinct phases that makes a material a composite; this also creates a distinction between composite processing and bulk material processing. Many multi-phased materials, such as high-carbon steel or porcelain, can indeed be produced by conventional bulk processing; however, these various phases are produced by internal phase transformation. Most composite fabrication processes comprise two main steps:

- i. separate production of the two phases to be combined, and
- ii. combination of these two phases.

The second step can be viewed as composite production in the stricter sense. Often, among the phases making a composite, one is distinguished as “reinforcement”, which is

generally the stronger phase of the constituent. In most cases, the reinforcement is also geometrically distributed as discrete elements (fibres, particles) within the composite. The other phase, which bonds the reinforcement elements together and percolates throughout the composite, is known as the matrix. Although generally the weaker phase, the matrix influences markedly the properties of the composite. Composites are therefore classified according to the nature of their matrix: polymer-, metal-, and ceramic-matrix composites, often designated as PMCs, MMCs, and CMCs, respectively. Reinforcements are distinguished by their shape. They can be:

- i. long, essentially continuous fibres,
- ii. short fibres,
- iii. whiskers, which are small elongated single crystals of high perfection, or
- iv. equiaxed particles of a wide variety of materials and forms.

Processes by which the reinforcements are produced vary widely, and depend strongly on the reinforcement shape (Mortensen,2007). Fibre-reinforced composite materials consist of fibres embedded in or bonded to a matrix with distinct interfaces (boundaries) between them. In this form, both fibres and matrix retain their physical and chemical identities, yet they produce a combination of properties that cannot be achieved with either of the constituents acting alone. In general, fibres are the principal load-carrying members, while the matrix keeps them in the desired location and orientation, acts as a load transfer medium between them, and protects them from environmental damages (Mallick, 2007).

2.2

Polymers

In recent years, as a result of better understanding of polymer structure–property relationships, introduction of new polymerisation techniques and availability of new and low-cost monomers, the concept of a truly tailor-made polymer has become a reality. Today, it is possible to create polymers from different elements with almost any quality desired in an end-product. Some polymers are similar to existing conventional materials, but with greater economic values, some represent significant improvements over existing materials, and some can only be described as unique materials with characteristics unlike any previously known to man. Polymer materials can be produced in the form of solid plastics, fibres, elastomers or foams. They may be hard or soft or may be films, coatings or adhesives. They can be made porous or non-porous or can melt with heat or set with heat (Ebewele, 2000).

There are a number of polymers available depending on the starting raw ingredients with several broad categories, each having numerous variations. The most common of these are polyesters, vinyl esters, epoxies, phenolics, polyimides, polyamides, polypropylene, polyether ether ketone (PEEK) and others. The major advantages of polymers as matrix materials are low cost, easy processability, good chemical resistance and low specific gravity. Some of their disadvantages include low strength, low modulus and low operating temperatures, which limit their use. Polymers can be classified in many different ways. The most obvious classification is based on the origin of the polymer, that is, natural or synthetic.

Other classifications are based on the polymer structure, polymerisation mechanism, preparative techniques, or thermal behaviour (Ebewele, 2000). Varieties of polymers for

composites production are thermoplastic polymers, thermosetting polymers, elastomers as well as their blends (Shehu,2015).

2.2.1 Thermoplastics

Thermoplastics are polymers consisting of linear or branched-chain molecules having strong intramolecular bonds, but weak intermolecular bonds. They can be reshaped by application of heat and pressure and are either semicrystalline or amorphous in structure. Examples include polyethylene (PE), polypropylene (PP), polystyrene (PS), nylons, polycarbonate (PC), polyether ether ketone (PEEK), polysulfone, and polyphenylene sulphide(Shehu, 2015).

2.2.2 Thermosetting polymers

Thermosets are those polymers with cross-linked or network structures having covalent bonds in all their molecules. They do not soften but decompose on heating because, once they are solidified by the cross-linking process, they cannot be reshaped. Common examples are epoxies, polyesters, phenolics, ureas, melamine, silicone and polyimides (Shehu, 2015).

2.2.3 Elastomers

An elastomer is a polymer with the property of viscoelasticity, generally having low Young's modulus and high yield strain compared to other materials. Each of the monomers that link to form the polymer is usually made of carbon, hydrogen, oxygen and silicon. Elastomers are amorphous polymers existing above their glass transition temperature (T_g), and their primary uses are for seals, adhesives and moulded flexible parts. Examples include natural rubber, synthetic polyisoprene, polybutadiene, chloroprene rubber, butyl

rubber, ethylene propylene rubber, silicone rubber, fluoroelastomers and thermoplastic elastomers (Shehu, 2015).

2.3 Polymer-Matrix Composites (PMCs)

These are among the best entrenched engineering composite materials. The three classes of polymers namely; thermoplastics, thermosets and rubbers are all employed as matrices. PMCs are very popular due to their low cost and simple fabrication methods. Reinforcement of polymers by strong fibrous network permits fabrication of PMCs, which is characterised by: High specific strength; High specific stiffness; High fracture resistance; Good abrasion resistance; Good impact resistance; Good corrosion resistance; Good fatigue resistance and Low cost (Shehu, 2015).

Fakhrul and Islam (2013) developed a polypropylene matrix composite by blending it with small additions (5%) of wood sawdust and wheat flour to improve its biodegradability. The experimental results revealed that addition of both sawdust and wheat flour considerably improved the biodegradability of polypropylene.

Shukor *et al.* (2013) conducted a research to investigate the effect of NaOH concentration (3%, 6% and 9%) for treating kenaf fibre used as reinforcement in poly lactic acid matrix bio-composite. They found that 6% alkali treatment at ambient temperature is the optimum condition to obtain maximum flexural modulus and impact strength of the composite.

Surata *et al.* (2014) produced rice husk fibre reinforced polyester composite and reported that tensile and flexural strengths of the composite increased when the fibre weight fraction increased. Tong *et al.* (2014) also produced recycled high density polyethylene (rHDPE)/rice husk (RH) filler composite and studied its mechanical and morphological

properties. They reported that tensile and flexural properties improved with increasing RH loading. However, the impact strength of the composite decreased as RH loading increased. SEM revealed good interfacial bonding between the fibre and polymer matrix.

Hague *et al.* (2015) studied fibre-matrix adhesion and properties evaluation of sisal/polyester composite. They investigated the effects of acetylation, acrylation, silanisation, alkalisation and permanganate treatment on physical, chemical and mechanical parameters. From their findings, they concluded that treatments such as acetylation, acrylation and silanisation can increase interfacial strength, wetting and compatibility between fibre and matrix.

2.4 Epoxy Resins

Epoxy resins are a class of thermosetting materials, characterised by two or more oxirane rings or epoxy groups within their molecular structure. The commonest epoxy resin is the diglycidyl ether of bisphenol A (DGEBA), which is prepared by the reaction of epichlorohydrin (ECD) and bisphenol A (BPA). The ECD is prepared from polypropylene (PP) by reacting chlorine with sodium hydroxide. It is allowed to react with BPA in the presence of sodium hydroxide. The first step is cleavage of the oxirane ring of ECD by the hydroxyl group of BPA. The second step is cyclisation in base medium, leading to the formation of an epoxy-ended intermediate. The intermediate then undergoes chain extension with BPA to produce an epoxy resin. A wide variety of resins can be produced by adjusting the concentration of the reactants. A liquid resin can be further chain-extended with BPA to make a solid resin of higher molecular weight. Thus, epoxy resins are available in various consistencies from low viscous liquid to a tack-free solid. The broad interest in epoxy resins originates from the versatility of epoxy group towards a wide

variety of chemical reactions and the useful properties of the network polymers, such as high strength, very low creep, excellent corrosion and weather resistance, elevated temperature service capability, and adequate electrical properties (Ratna, 2009).

An epoxy formulation must contain a suitable curing agent and some optional ingredients, which are decided after considering the application in which the resin is going to be used. Examples of such ingredients are diluents, fillers or extenders. Diluents are used in an epoxy formulation to reduce the viscosity or to eliminate the need of solvents (Ratna, 2009).

Cross-linked epoxies exhibit outstanding properties that have placed them as the standard option for a variety of applications such as adhesives, coatings, composites for structural applications, and so on. Some of the reasons for these are:

- i. No emission of volatile products in the polymerisation reaction.
- ii. Flexibility in the selection of monomers and co-monomers enabling one to obtain a variety of products from low glass-transition temperature (T_g) rubbers to high T_g materials.
- iii. Possibility to use and combine different polymerisation chemistries appropriate for the particular processing needs.
- iv. Very low contraction during polymerisation (for some formulations contraction may be entirely eliminated).
- v. Very high adhesion to a variety of surfaces due to the polar groups present in the structure.

- vi. Possibility of introducing different modifiers to generate optical properties (as in dispersions of droplets of liquid crystals in an epoxy matrix), or to increase toughness (rubber-or thermoplastic-modified epoxies)(Pascault and Williams, 2010).

Due to the versatility of epoxy resins towards a wide variety of chemical reactions, epoxy resins can be cured using a range of materials with different types of curing conditions. The choice of curing agents (also called ‘hardeners’) depends on the curing conditions applicable and the final application of the resin. Epoxies can be cured with amines, thiols, and alcohols. The reaction proceeds through cleavage of the oxirane ring through a nucleophilic addition reaction (Ratna, 2009).

Singh *et al.* (2013) developed epoxy matrix/coconut shell powder (CSP) composites and studied the mechanical properties and water absorption behaviour of the composite. They concluded that the composite is suitable for application in the interior part of aircraft and automobile where materials with good tensile strength and low density are required.

Mishra (2014) reinforced epoxy resin with jute fibre and investigated the physical, mechanical and abrasive wear behaviour of the composites. The study revealed that abrasive wear performance of needle punched non-woven jute based composite was better than that of the short and bi-directional jute fibre reinforced composites. Results of physical and mechanical properties of the composites also revealed that these properties are affected by fibre type, fibre content and void content.

2.5 Natural Fibres

Natural fibre encompasses all forms of fibres from woody plants, grasses, fruits, agriculture crops, seeds, water plants, palms, wild plants, leaves, animal feathers, and animal skins.

By-products of pineapple, banana, rice, sugarcane, coconut, oil palm, kenaf, hemp, cotton, abaca, sugar palm, sisal, jute and bamboo are among the fibres known to be used to make composites. Wool and silk are strong fibrous materials, for over 3500 years wool had been used in textile industry and silk from at least 5,000 years (Salit, 2014). The plant-based fibres contain cellulose and non-cellulose materials such as hemicelluloses, pectin and lignin; thus, they are also known as ligno-cellulosic or cellulosic fibres. Natural cellulose fibres are extracted from ligno-cellulosic by-products using biological retting (bacteria and fungi), chemical retting (boiling in chemicals), mechanical retting (hammering, decortications), and water retting. Natural fibres can be used in the form of particulate or filler, short fibres, long fibres, continuous roving, woven fabric and non-woven fabric. The natural fibre-reinforced polymer composites performance depends on several factors, including fibres chemical composition, cell dimensions, microfibrillar angle, defects, structure, physical and mechanical properties, and also the interaction of a fibre with the polymer. In order to expand the use of natural fibres for composites and improve their performance, it is essential to know the fibre characteristics (Faruk *et al.*, 2012).

2.5.1 Classification of natural fibres

Natural fibres can be classified according to their origin (Bongarde and Shinde, 2014) as:

- i. Animal fibres: these include wool, silk, and avian fibres.
- ii. Mineral fibres: are naturally-occurring fibres or slightly modified fibres obtained from minerals, for example, asbestos.
- iii. Plant fibres: generally comprised mainly cellulose. These fibres can be further categorised as follows:

- a. Seed fibres: fibres collected from seeds and seed cases, for example, cotton and kapok.
- b. Leaf fibres: fibres collected from leaves, for example, sisal and agave.
- c. Bast fibres: fibres collected from skin or bast surrounding the stem of their respective plants. These fibres have higher tensile strength than other fibres. Therefore, these fibres are used for durable yarn, fabric, packaging, and paper. Some examples are flax, jute, banana, hemp, soybean, and kenaf.
- d. Fruit fibres: fibres collected from fruits of plants, for example, coconut (coir) fibre, and borassus palm fruit fibre.
- e. Stalk fibres: fibres are actually the stalks of the plants such as straws of wheat, rice, barley, and other crops, including bamboo and grass. Tree wood is also such a fibre.

2.5.2 Ligno-cellulosic fibres

Ligno-cellulosic fibres consist of bundles of hollow cellulose fibrils. Their cell walls are reinforced with spirally-oriented cellulose in a hemicellulose and lignin matrix. The composition of the external surface of the cell wall is a layer of lignaceous material and waxy substances that bond the cell to its adjacent neighbours. These components are to be considered in the study of the interaction with polymer matrices. Thus, the discussion of their spectra and modifications or their interactions with different polymers, one should begin by identifying their corresponding absorption bands by the use of Fourier transform infrared (FTIR) spectroscopy (Zafeiropoulos, 2011).

Ligno-cellulosic natural fibres, such as sisal, coir, jute, ramie, pineapple leaf (PALF), and kenaf have the potential to be used as replacement for glass or other traditional reinforcement materials in composites. These fibres have many properties which make

them attractive alternative to traditional materials. They have high specific properties (specific strength and modulus). In addition, they are available in large amounts, renewable and biodegradable. Other desirable properties include low cost, low density, less equipment abrasion, less skin and respiratory irritation, vibration damping capability, and enhanced energy recovery (Sgriecia *et al.*, 2008). Ligno-cellulosic fillers/fibres, depending on their performance when incorporated in a plastic matrix, can be classified as:

- i. Flours which can be considered as particulate fillers that enhance (increase) tensile and flexural moduli of the composites, while having little effect on the composite strengths.
- ii. Fibres which have relatively higher aspect ratios and contribute to an increase in moduli of the composite as well as its strength, when suitable additives are employed to improve stress transfer between the matrix and the fibres (Shehu, 2015).

Begum and Islam (2013) reported that natural fibre-reinforced composites can attain equivalent mechanical strength as that of the glass fibre-reinforced composites on using higher volume fraction of natural fibres than glass fibres during production of the composites. They also reported that the natural fibre-reinforced polymer composites have already been proven to be an alternate to the synthetic fibre reinforced composites in applications such as automotive, packaging and construction industries. Verma *et al.* (2013) also reported that various natural fibre composites can favourably compete with the mechanical properties of glass fibre composites and have already found many industrial applications.

2.6

Modification of Natural Fibres

The main disadvantages of natural fibres as reinforcements in composites are the poor compatibility between fibre and matrix due to their relatively high moisture absorption property as a result of hydroxyl (–OH) group that is present between the macromolecules in the fibre cell wall. When moisture from the atmosphere comes in contact with the fibre, the hydrogen bond breaks and hydroxyl groups form new hydrogen bonds with water molecules. The cross-section of the fibre becomes the main access of water penetration. The interaction between hydrophilic fibre and hydrophobic matrix causes fibre swelling within the matrix. This results in weakening the bond strength at the interface, which leads to dimensional instability, matrix cracking and poor mechanical properties of the composites. The quality of fibre–matrix interface is significant for application of natural fibres as reinforcement fibres for plastics. Therefore, natural fibre modifications are considered in order to improve their adhesion with different matrices (Faruk *et al.*, 2012). Additionally, most natural fibres have low degradation temperatures (~200°C), which make them incompatible with polymers of higher melting temperatures. This also restricts natural fibre composites to relatively low temperature applications (Sgriccia *et al.*, 2008). Physical and chemical methods can be used to optimise this interface. These modification methods are of different efficiency for adhesion between the matrix and the fibre (Bledzki and Gassan, 1999).

2.6.1 Physical methods

Physical methods include stretching, calendaring, corona, plasma, electron beam and the production of hybrid yarns for modification of natural fibres. Physical treatments change structural and surface properties of the fibre and influence mechanical bonding of the

polymers. Physical treatments do not extensively change the chemical composition of the fibres. Therefore, the interface is generally enhanced via an increased mechanical bonding between the fibre and the matrix (Faruk *et al.*, 2012).

Corona treatment is one of the most interesting techniques for surface oxidation activation. This process changes the surface energy of cellulose fibres. Corona discharge treatment on cellulose fibre and hydrophobic matrix was found to be effective for improvement of the compatibilisation between hydrophilic fibres and a hydrophobic matrix (Faruk *et al.*, 2012). Amirou *et al.* (2013) modified date palm fibre by using corona discharge treatment, which results in a surface oxidation and reported that for composites containing treated fibres, there was a significant increase in tensile strength and Young's modulus compared to the composites with untreated fibres. They concluded that the interfacial contact between both components is improved because of higher mechanical keying, enhanced as a consequence of the etching effect.

Plasma treatment is another physical treatment method and is similar to corona treatment. The property of plasma is exploited by this method to induce changes on the surface of a material. A variety of surface modifications can be achieved depending on the type and nature of the gases used. Reactive free radicals and groups can be produced, the surface energy can be increased or decreased and surface cross-linking can be introduced (Faruk *et al.*, 2012). Bhattacharyya *et al.* (2004) modified wood fibre surface with argon and air plasma treatment and reported that the tensile strength and tensile modulus of the composite sheet reinforced with wood fibre improved to some extent after the plasma treatment.

Electron Beam (EB) irradiations are conducted to create a hydrophobic group, cause cross-linking and increase the interfacial surface area of fibres. The EB irradiation technique is increasingly being utilized to alter the surfaces of various materials, such as fibres, textiles and films. Cotton fabrics have been coated with pigment colours using EB to improve colour fastness, and create resistance to water absorption. Han *et al.* (2006) irradiated natural fibres with EB to improve adhesion between fibres and thermoplastics and reported that EB irradiation is effective in both impurity removal and functional group development on the surface of natural fibres for better bonding between natural fibre and polymer matrix.

2.6.2 Chemical modification and use of adhesion promoters

In many cases, it is possible to induce compatibility in two incompatible materials by introducing a third material that has properties intermediate between those of the other two. There are several coupling mechanisms in materials, for example, weak boundary layers, deformable layers, restrained layers, wettability, chemical bonding, and acid–base effect. The development of a definite theory for the mechanism of bonding using coupling agents in composites is a complex problem. Hence, the consideration of other concepts appears to be necessary. These include morphology of the inter-phase, acid–base reactions in the interface, surface energy and the wetting phenomena (Faruk *et al.*, 2012). Fibre surface modification relies on chemical techniques, mainly focused on grafting chemical groups, capable of improving the interfacial interactions between filler particles and polymer matrix. The main techniques may be summarised as follows:

- i. Alkali treatment (also called mercerisation): treatment of natural fibres with sodium hydroxide (NaOH) is widely being used to modify the cellulosic molecular structure. It

changes the orientation of highly packed crystalline cellulose order and forms an amorphous region. This provides more access to penetrating chemicals. In the amorphous region, cellulose micro-molecules are separated at large distances and the spaces are filled by water molecules. Alkali sensitive hydroxyl (OH) groups present among the molecules are broken down, which then react with water molecules (H-OH) and move out from the fibre structure. The remaining reactive molecules form fibre-cell-O-Na groups between the cellulose molecular chains. Due to this, hydrophilic hydroxyl groups are reduced, and they increase the fibre moisture resistance property. It also takes out a certain portion of hemicelluloses, lignin, pectin, wax and oil covering materials; consequently, the fibre surface becomes clean. In other words, the fibre surface becomes more uniform due to elimination of microvoids and the stress transfer capacity between the ultimate cells improves. In addition, it reduces fibre diameter and thereby increases the aspect ratio (length/diameter). This increases effective fibre surface area for good adhesion with the matrix. Mechanical and thermal behaviours of the composites are improved significantly by this treatment. If the alkali concentration is higher than the optimum condition, excess delignification of the fibre can take place, which results in weakening or damaging the fibres (Kabir *et al.*, 2012). Fibres are immersed in 5% solution of NaOH for 48 hours at room temperature (25 – 26 °C), rinsed with distilled water and then neutralised in 1% glacial acetic acid solution. The fibre is then oven dried for 5 hours at 110°C (Mwaikambo and Ansell, 1999; Ansell and Aziz, 2004).

- ii. Acetylation: fibres are usually immersed in glacial acetic acid for 1 hour, then immersed in a mixture of acetic anhydride and few drops of concentrated sulphuric acid for 5

- minutes, then filtered, washed and dried in ventilated oven (La Mantia and Morreale, 2011).
- iii. Benzoylation: the fibres are immersed in 10% NaOH and then stirred with benzoyl chloride for 1 hour, filtered, washed and dried, then immersed in ethanol for 1 hour, rinsed and dried in oven (La Mantia and Morreale, 2011).
 - iv. Toluene-2,4-diisocyanate (TDI) treatment: fibres are immersed in chloroform with 3 drops of a catalyst (based on dibutyltin dilaurate) and stirred for 2 hours after adding toluene-2,4-diisocyanate. Finally, fibres are rinsed in acetone and dried in oven (La Mantia and Morreale, 2011).
 - v. Peroxide treatment: fibres are immersed in a solution of dicumyl (or benzoyl) peroxide in acetone for about half an hour, then decanted and dried. (La Mantia and Morreale, 2011).
 - vi. Permanganate treatment: the fibres are immersed in a solution of KMnO_4 in acetone (typical concentrations may range between 0.005 and 0.205%) for 1 minute, then decanted and dried (La Mantia and Morreale, 2011).
 - vii. Silane treatment: the fibres are immersed in a 3:2 alcohol–water solution, containing a silane- based adhesion promoter for 2 hours at $\text{pH} \approx 4$, rinsed in water and oven dried (La Mantia and Morreale, 2011).
 - viii. Latex modification: fibres are given a latex coating by dipping them into a natural rubber latex having 10% dry rubber content after pre-treatment with NaOH (Sreekala *et. al.*, 2002).
 - ix. Acrylonitrile grafting: fibres are bleached with 2% alkali for 30 minutes and then oxidised with 0.02ml KMnO_4 (liquor ratio 1:150) for 10 minute. Then, it is washed with water and put into 1% H_2SO_4 -containing acrylonitrile in the ratio 30:1. The sample is

then placed in a thermostatic water bath at 50°C for 2 hours without any disturbance. The sample is then washed with water thoroughly and dried. The grafted fibre is isolated from any homopolymer formed by Soxhlet extraction using dimethyl formamide (Sreekala *et al.*, 2002).

Effect of alkali treatment on different ligno-cellulosic fibres has been investigated by researchers. Fibres such as hemp, sisal, jute and kapok (Mwaikambo and Ansell, 1999); kenaf (Ansell and Aziz, 2004); banana (Zuluaga *et al.*, 2009); coir (Gu, 2009); date palm (Abdal-hay *et al.*, 2012) have been investigated. Ansell and Aziz (2004) reported that the optimum alkali treatment condition for kenaf fibre is 6 wt.% NaOH, 48 hours soaking time. On the other hand, Abdal-hay *et al.*, (2012) observed that 6 wt.% NaOH, 2 hours soaking time is the optimum treatment condition for jute fibre. In furtherance to these, Mwaikambo and Ansell (1999) treated hemp, jute, sisal and kapok fibres with various concentrations of NaOH and found 6wt.% to be the optimised concentration in terms of cleaning the fibre bundle surfaces yet retaining a high index of crystallinity. They reported that higher concentrations of NaOH are likely to damage the cell wall and reduce the bulk density. Ansell and Aziz (2004) also argued that results obtained for both treated hemp and kenaf fibres indicated that 6 wt.% NaOH treatment did not cause cell wall damage. Based on the variation in optimum treatment condition and soaking time, it can be asserted that optimum NaOH concentration and soaking time might be varying for the different types of natural fibres (Mishra, 2014).

2.7

Retting

Retting is a process employing the action of micro-organisms and moisture on plants to dissolve or rot away much of the cellular tissues and pectins surrounding fibre bundles;

thus, facilitating separation of the fibre from the stalk, and stem or bast. During retting, micro-organisms colonise the stem tissues and partially degrade plant constituents to separate fibres from non-fibre fractions. The two primary methods for retting (microbiological retting) are water and dew (field) retting, traditionally used to extract fibres for commercial applications (Mussig, 2010).

2.7.1 Dew (field) retting

Retting can be done in the field after the growing season is over. The cut plant is allowed to lay in the field and dew collects on it. Naturally-occurring micro-organisms collect on the wet plant and the biological action removes the bark and separates the bast from the core fibres. This process takes longer than the pond/water retting and may take more than a month for complete fibre separation to take place (Pickering, 2011).

2.7.2 Water retting

Water retting depends on colonisation of stalks and fermentation by anaerobic bacteria; for example, *Clostridium felsinium*, to degrade pectins and other substances and free bast fibres. In early times, harvested stalks were bundled and submerged in natural bodies of running or still water (for example, lakes, rivers and dams) for 5–7 days and then dried in the field for 1–2 weeks. Retting pits or tanks were constructed to be flushed with an initial rinse water to remove contaminants, heated to controlled temperatures and even inoculated with specific micro-organisms. Aeration of the tanks has been attempted to modify the microbial species and subsequently the anaerobic metabolism; that is, to reduce acidity and toxins to retting micro-organisms. Different microbial consortia and more complete oxidation of organic materials result from aerated conditions. Water-retted stems are then sun-bleached and dried naturally (Mussig, 2010).

2.7.3 Stand retting

Another method of retting in the field with indigenous fungi for the most part was attempted in the 1960s to 1970s to overcome limitations of dew retting in Northern Ireland. In these trials, glyphosate (N-phosphonomethyl glycine) was used as a pre-harvest desiccant to facilitate retting. Stand-retted fibre pretreated with glyphosate retained more strength than dew-retted fibre, although fungal colonisation and retting were slower. However, recent reports indicate a continuing interest in glyphosate treatment and stand retting (Mussig, 2010).

2.7.4 Chemical retting

Considerable research has been undertaken to find a replacement for water and dew retting. Chemical retting has been evaluated using a variety of methods, including ethylenediaminetetra-acetic acid (EDTA) or other chemical chelators at high pH, detergents, strong alkali and steam explosion. Sharma (1988) patented a chemical retting method using chelating agents that removed divalent cations such as Ca^{2+} , and helped to separate fibre from non-fibre components. Autoclaving flax straw with the chelators, EDTA and oxalate was reported to be an effective method for breeding programmes in increasing the extractability of unretted flax fibres to the level of warm-water-retted flax (Mussig, 2010).

2.7.5 Enzyme retting

Enzyme retting has long been considered as a potential replacement for the dew and water retting. Such potential for enzyme retting has prompted in-depth research to develop effective processes. While the presence of diverse, multiple hemicellulases reportedly contributes to effective retting, early work with water- and dew-retting micro-organisms

showed conclusively that pectinases were a primary requirement for effective retting (Mussig, 2010).

A modification of dew and water retting is enzymatic treatment, called bioscouring, where degrading enzymes are directly added to tank water or in a bioreactor. Despite the complexity of plant stem, the retting process was successfully carried out using purified enzymes. Researchers developed an enzyme-retting process tested in small pilot plant for the treatment of flax in 2001. The main enzymatic activity necessary to free the fibres from other tissues are pectinases, and the addition of chelators with pectinase-rich enzyme mixtures is used to improve retting (Speri, 2011).

2.7.6 Other retting processes

Other methods, sometimes chemical combined with physical methods, have been reported to separate fibres from non-fibre components successfully. A mechanical process to produce fibre strip, followed by a chemical/heating process under pressure, has been patented to separate fibres. Tank retting, by contrast, employs vats usually made of concrete, requires about four to six days, and is feasible in any season. In the first six to eight hours, called the leaching period, much of the dirt and colouring matter are removed by the water, which is usually changed to assure clean fibre. Waste retting water, which requires treatment to reduce harmful toxic elements before its release, is rich in chemicals and can be used as liquid fertilizer. Ultrasonic treatment, following decortication and opening of green flax or hemp stalks, has been used to obtain fibres from diverse sources without the use of chemicals. Flash hydrolysis or steam explosion treatment, with or without impregnation before steam treatment, has been used to remove pectins and hemicelluloses from decorticated flax for small bundles and ultimate fibres. Chemical separation has resulted in

successful laboratory results but at times, fibre properties are less satisfactory than those from other methods (Mussig, 2010).

After retting, the retted stalks called straws are dried in the open air or by mechanical means and are frequently stored for short periods to allow curing to occur, facilitating fibre removal. Final separation of the fibre is accomplished by a breaking process in which the brittle woody portion of the straw is broken, either by hand or by passing through rollers; followed by the scutching operation, which removes the broken woody pieces (shives) by beating or scraping. Some machines combine breaking and scutching operations. Waste material from the first scutching, consisting of shives and short fibres, is usually treated a second time. The short fibre (tow), thus, obtained is frequently used in paper manufacture, and the shives may serve as fuel to heat the retting water or may be made into wallboard(Retting, 2016).

2.8 *Borassus aethiopum* Mart.Arecaceae

Borassus aethiopum (Plate I) with local names: English ron palm, borassus palm, African fan palm, fan palm, deleb palm; Hausa *giginya*; Igbo *ubiri* and Yoruba *agbon-eye* has the following features (Orwa, *et al.*, 2009):

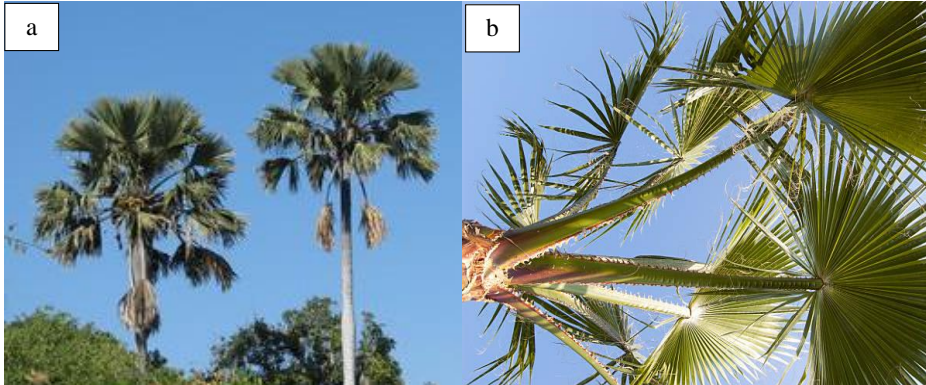


Plate I: *Borassus aethiopum* M. (a) trees; (b) leafstalks

- i. it is an unbranched palm growing up to 20m tall, characterised by a crown up to 8 m wide.
- ii. young palms are covered with dry leaf stalks showing gradually fading leaf scars
- iii. trees over 25 years old have a swelling of the trunk at 12-15m above the ground (at 2/3 of the height).
- iv. bark is pale grey in older palms and is more or less smooth. Leaves are very large, fan shaped, bluish-green, 15-30 in number, up to 3.5m long, including petiole marked with sharp, black thorns.
- v. leaflets symmetric at the base. Flowers dioecious, yellowish; male flowers clustered in a branched spadix, 0.8-1.8 m long. Female flowers with unbranched and shorter spadix, 1.3-2.6m long. The fruit is a large drupe, with diameter of about 15cm, ovoid, orange to brown when ripe.
- vi. fibrous pulp contains three woody kernels with an albumen that becomes hard when ripe.

B. aethiopum is abundant and characteristic in all types of Savannah of the region, occurring at low altitudes along rivers and in coastal woodlands. It is mainly distributed in the Guineo-Congolian and Sudanian Savannas. It can tolerate high temperatures and will grow in areas with rainfall less than 500 mm/year, if the groundwater table is high. It is often in dense stands. The palm can serve as an excellent fire-break, especially in the arid regions of West Africa, which are prone to wildfires. Documented species distribution is native to Benin, Burkina Faso, Congo, Cote d'Ivoire, Democratic Republic of Congo, Ethiopia, Gambia, Ghana, Guinea, Guinea-Bissau, Kenya, Liberia, Mali, Mozambique, Nigeria, Senegal, Sierra Leone, South Africa, Sudan, Tanzania, Togo, Uganda, Zambia, and Zimbabwe (Orwa *et al.*, 2009).

The stem of *B. aethiopum* delivers a hard wood that is resistant to decay and the damaging effect of sea water. The durable wood is rarely attacked by termites and fungi and it has excellent working properties, when used as timber for construction purposes and furniture (Sambou *et al.*, 1992). In Mozambique, the wood is used to make dugout canoe, fibres extracted from the leaf stalk have valuable qualities of resistance to chemicals and termites. Young leaves, before unfolding, can be split into strips and woven into thin mats, baskets and other household objects. The leaf mid-ribs are used to make brooms, fish traps and nets. The leaf stalk can be soaked in water to provide fibres that are used as sponges or filters (Orwa *et al.*, 2009).

2.9 Mechanical Properties of Natural Fibre Reinforced Polymer Composites

There is a large amount of literature detailing the mechanical properties of natural fibre reinforced polymer composites. Mohanty *et al.* (2000) studied the effect of jute fibre content on the tensile behaviour of jute fabrics/polyester amide composites. Jute content

was varied from 20 to 53 wt.% to determine its effect on the tensile properties. It was reported that the tensile strength of composite increased from 20 wt. % to 32 wt. % fibre loading and afterward with further increase of jute content properties tend towards lower values.

Investigation on the mechanical properties of rice husks fibre reinforced polyester composites by Surata *et al.*, (2014) showed that tensile strength and modulus as well as flexural strength and modulus increased with increase in fibre weight fraction. The mechanical properties further improved also with alkalisation of the fibres due to better interfacial bonding between the fibre and the matrix.

Impact strength signifies the ability of the material to absorb impact energy. Lima *et al.* (2011) evaluated the impact strength of recycled polyethylene (PE) composites reinforced with used or new jute fabric. The incorporation of both new and used jute fabric considerably increased the impact energy of composites, with greater values related with the new jute fabric. Furthermore, Părpărită *et al.*, (2014), found that impact strength decreased in composites relative to the matrix polypropylene because the ligno-cellulosic components probably reduced the mobility of the polymer chains and by extension, the ability to absorb energy during fracture propagation. The hardness values of the composites were found to be higher than that of the pure matrix probably due to the formation of a more rigid surface of the composites relative to the pure matrix.

2.10 Water Absorption Properties of Natural Fibre Reinforced Polymer Composites

Water absorption is one of the main disadvantages of natural fibre reinforced composites. It has been shown by researchers to increase with increased fibre content and temperature as

well as being influenced by fibre treatment/coupling agent and fibre arrangement. It is commonly associated with swelling of composites and reduced mechanical properties (Pickering *et al.*, 2016).

Huner (2015) studied the effect of water absorption on mechanical properties of flax fibre reinforced epoxy composites. The study revealed that tensile and flexural properties of reinforced epoxy specimens to decrease with increase in percentage moisture uptake. The moisture also induced degradation of composite samples was significant at elevated temperature. Sreekala and Thomas (2003) investigated the effect of surface modification on water absorption behaviour of oil palm fibres. The investigation was carried out at different temperatures of 30, 50, 70 and 90 °C. They observed at the end of their study that the diffusion mechanism for all the systems were non-Fickian and treatments reduced the water uptake at all temperatures. They also reported that mechanical performance of the fibres decreased on water absorption.

2.11 Thermal Properties of Natural Fibre Reinforced Polymer Composites

Thermal analysis of polymers is an important study used for understanding the structure–property relation and mastering the technology for industrial production of different polymeric materials, especially fibre-reinforced composites. Moreover, it is a useful technique to determine the thermal stability of the materials, especially with the processing conditions in mind (Shehu, 2015).

2.11.1 Dynamic mechanical analysis (DMA)

Dynamic Mechanical Analysis (DMA) is a characterisation technique used to study the viscoelastic properties of polymers and also measure the glass transition temperature (T_g) of

the polymer. It also gives the storage modulus, loss modulus and damping (energy dissipation) behaviour of polymers (Hassan *et al.*, 2013).

Heitor *et al.* (2010) evaluated the performance of glass/sisal hybrid composites focusing on mechanical (flexural and impact) and dynamic mechanical thermal analysis (DMTA). Hybrid composites with different fibre loading and volume ratios between glass and sisal were studied. The results obtained in DMTA revealed an increase in the storage and loss moduli, as well as a shift to higher values for higher glass loading and overall fibre volume.

Hassan *et al.* (2013) studied the dynamic mechanical properties and thermal stability of poly lactic acid (PLA) and poly butylene succinate (PBS) blends composites. The results obtained revealed lowering of storage moduli of all blended PLA which indicated increase molecular mobility by adding PBS due to lower T_g .

2.12 Degradation of Natural Fibre Reinforced Polymer Composites

Shah *et al.* (2008) in their review stated that any physical or chemical change in polymer as a result of environmental factors, such as light, heat, moisture, chemical conditions or biological activities, processes inducing changes in polymer properties (deterioration of functionality) due to chemical, physical or biological reactions resulting in bond scission and subsequent chemical transformation have been categorized as polymer degradation.

Yussuf *et al.* (2010) compared poly lactic acid/kenaf (PLA/K) and poly lactic acid/rice husk (PLA/RH) composites, looking at the influence of the natural fibre on the mechanical, thermal and biodegradability properties. Weight loss results after 90 days of burial, showed biodegradability of the composites to be 1.2 and 0.8% for PLA/K and PLA/RH, respectively.

Kijchavengkul *et al.* (2010) investigated the atmospheric and soil degradation of aliphatic-aromatic polyester films under conditions of solar exposure and soil burial in tropical area for 40 weeks. They concluded that the reduction of molecular weight of soil burial samples was much slower than that of non-cross-linked solar exposed samples, with the solar exposed samples following first order reaction, whereas the soil burial samples showed surface erosion biodegradation behavior.

Sapuan *et al.* (2013) studied the mechanical properties of soil buried kenaf reinforced thermoplastic polyurethane (TPU) composites after 80 days of burial. They found that soil burial test has significant effect on tensile strength of composites after 80 days. They also observed that there was no significant change in flexural properties of soil buried composites. They concluded that TPU as matrix needed more than 80 days to degrade, and that weather, temperature, humidity and degradation process significantly influenced the reduction in tensile strength of the composites.

2.13 Review of Previous Works

In their review on microbial deterioration and degradation of polymeric materials Mohan and Srivastava (2010) believe that all polymers are more or less biodegradable to some extent due to the organic nature of their principal elements like resin and hardener. Polymeric material complexity, structures and compositions are among the important aspects that govern polymer biodegradation. In their conclusion, they stressed the need for microbial attack susceptibility studies for all types of synthetic polymeric materials before application.

Bharath and Rajesh (2010) developed different weight fraction of randomly distributed areca fibre and maize powder reinforced urea formaldehyde and investigated the moisture absorption characteristics of the composites. The results obtained showed that moisture absorption decrease with decrease in fibre to maize powder ratio and moisture content increase with increasing time duration and attained saturation. It was concluded that the composite is a promising substitute for conventional wood based plywood or particle board.

Imoisili *et al.* (2012) investigated the effect of silane coupling agent on the tensile properties of rice husk flour (RHF) polyester composite. The test results showed that tensile strengths, moduli, maximum load and micro hardness of the composites exhibited appreciable improvement with the use of coupling agent on the filler. They concluded that silane and sodium hydroxide treatment in rice husk filler in polyester composite leads to a higher tensile strength compared to the untreated filler composite.

Rassiah *et al.* (2012) studied the effect of sodium hydroxide on the water absorption and biodegradability of Low Density Polyethylene (LDPE)/sugarcane bagasse (SCB) composites. At the end of their study, they suggested that sugarcane bagasse could be used as a biodegradable filler in polymeric matrices so as to minimize environmental pollution.

Fardausy *et al.* (2012) reported that the TG, DTG and DTA curves of jute/PVC composites are the average of fibre and of PVC. The degradation of jute/PVC composites occurred in two stages. The thermal stability of PVC-jute fibre composites was observed to be higher than that of jute fibre but lower than that of PVC film.

Inamdar *et al.* (2013) studied the tensile properties of natural fibre polymer matrix composites. Different natural fibre based polymer matrix composites were prepared by

using coir, saw-dust and rice husk and their hybrid in unsaturated polyester (UP). To have good fibre-matrix compatibility, chemical modification of the fillers was carried out. It was found that the hybrid composite showed better tensile strength than individual fibre composites. It was concluded that the strengths of all composites increased as percentage of reinforcement increased.

Suresha *et al.* (2013) studied the three-body abrasive wear behaviour of organo-modified montmorillonite-filled epoxy nanocomposites with different filler loading. It was reported that 5 wt.% organo-modified montmorillonite-filled epoxy nanocomposite showed enhanced abrasive wear resistance as compared to that of the other organo-modified montmorillonite-filled epoxy nanocomposites and neat epoxy.

Essabir *et al.* (2013) studied the mechanical and thermal properties of polypropylene/Almond shell particles bio-composites, from the thermo gravimetric analysis (TGA) results, three degradation peaks for almond shell (AS) particles were observed. The peaks were observed at 285-320°C, 320-430°C and 432°C representing thermal decomposition of hemicellulose, cellulose and lignin respectively. They concluded that the addition of particles to polymer matrices reduce the overall thermal stability of resulting composites.

Zhu *et al.* (2013) studied the effect of fibre treatments on water absorption and tensile properties of flax/tannin composites. Five fibre pretreatments were employed and expected to enhance the performances of flax/tannin composites. The modifications resulted in rougher fibre surface. Water sensitivity of composites were not reduced by the treatments as expected. However, the NaOH and NaOH-butanetetracarboxylic acid composites

maintained the tensile properties at dry status and show a better resistance to water aging than untreated composites. Moreover, NaOH-aminopropyltriethoxysilane, laccase and laccasse – Doga treatments result in more consistency in the tensile properties of the composites.

Govindaraju *et al.* (2014) worked on the optimization of process parameters for the preparation of wood based polypropylene composites with respect to mechanical properties. Compression moulding based fabrication technique was considered for this study.

The literature review presented and Table 2.1 revealed the following knowledge gap in the research reported so far:

- i. the *B. aethiopum* fibre based products has many end applications such as in housing, packing and furniture, but its potential use in composite applications is yet to be harnessed.
- ii. although works had been done to characterise *B. aethiopum* fibre chemical and mechanical properties, characterisation of composites reinforced by the fibre are rare.
- iii. though a number of research efforts had been devoted to development and characterisation of composites from fibres extracted from *B. flabellifer* (commonly known as the palmyra tree, which is of the same genus, but a different species with *B. aethiopum*), composites reinforced with fibres extracted from *B. aethiopum* has limited report in the literature.

Based on these findings, the present research therefore was aimed at developing and characterising epoxy/BPLSF reinforced composite.

Table 2.1a: Review of Previous Works and Findings

S/N	Author (s)/Year	Title	Findings
1.	Sarki (2012)	Development and characterisation of epoxy/coconut shell particulate composite for engineering applications	He concluded that composites of light weight can be produced using carbonized coconut shell reinforcement because their densities decrease as percentages of coconut ash increases in epoxy matrix.
2.	Nayak and Mishra (2013)	Development and mechanical characterisation of palmyra fruit Fibre reinforced epoxy composites	It was concluded that the composite is suitable for structural applications due to high strength, but could only find application in dry environment due to water absorption that is higher than specified as per standard.
3.	Shanmugam and Thiruchitrambalam (2013)	Static and dynamic mechanical properties of alkali treated unidirectional continuous palmyra palm leaf stalk fibre/jute fibre reinforced hybrid polyester composites.	It was concluded that alkali treatment of fibre enhanced tensile and flexural properties of the composites; and that the new hybrid composites can be a potential replacement in place of synthetic fibres such as glass fibres and some popular natural fibres like sisal, hemp, coir, banana, and bamboo. Hence, new possibilities of the use of this hybrid system may be explored.
4.	Kalia <i>et al.</i> (2013)	Surface modification of plant fibres using environment-friendly methods for their application in polymer composites, textile industry and antimicrobial activities: A review	It was concluded that plasma treatment results into the surface etching and cleaning or activation of the plant fibres.

Table 2.1b: Review of Previous Works and Findings continued

S/N	Author (s)/Year	Title	Findings
5.	Acheampong (2014)	Physico-chemical properties and natural durability within two varieties of <i>Borassus aethiopum</i>	He concluded that generally the two varieties of <i>B. aethiopum</i> recorded greater values from the base to the crown for density (at green and dry states), total extractives, lignin, alpha - cellulose as well as holocellulose. However, less moisture content (at green and dry states), dimensional stability, ash content, hemi-cellulose as well as mass loss and visual durability rating was observed at the peripheries than the cores.
6.	Ngargueudedjim <i>et al.</i> , (2015a)	Mechanical characteristics of fibre Palmyra	They found out that Young's modulus and the failure strains of heartwood and sapwood fibres are of the same order of magnitude; the tensile strength of the fibre of heartwood is higher than that of the fibre of sapwood; the mechanical properties are dependent on the cross-sectional area (diameter) of the fibre.
7.	Ngargueudedjim <i>et al.</i> (2015b)	Mechanical characteristics of tall-palm (<i>Borassus Aethiopum Mart., Arecaceae</i>)of Chad/Central Africa.	They concluded that various mechanical tests show that the palm wood has mechanical properties above average of known mechanical properties of certain species, for the heartwood part. This part of the palm wood has an appreciable resistance, which can be explained in part by the value of its average density, and, thus, the density of the fibres that constitute it.
8.	Shehu (2015)	Development and characterisation of poly (lactic acid) (PLA)/guinea corn (sorghum bicolor) husks particulate (GHP) composites	It was concluded that the composite could be best used indoors, for applications in which high strength is not a requirement or in applications requiring single use before disposal.

CHAPTER THREE
MATERIALS AND METHODS

3.1 Materials

The materials used in this research included borassus palm leaf stalk obtained from borassus palm tree in Dorayi, Giwa Local Government, Zaria, Kaduna State. Distilled water, release agent and lubricant, syringes, sodium hydroxide (NaOH) pellets produced by Fishers Scientific Company, U.S.A., glacial acetic acid 99%, ACS, ISO and natural plant latex, all obtained in Zaria. Epoxy resin of grade 3554A and curing hardener of grade 3554B were supplied by Rodco Nigeria Limited, Ojota, Lagos, Nigeria.

3.2

Equipment

Equipment used in this research are listed in Tables 3.1a and 3.1b

Table 3.1a: List of Equipment used in the study

S/No.	Equipment	Model	Source
1.	Oven	Cole and Palmer, U.S.A. Model: 60628	Department of Metallurgical and Materials Engineering, A.B.U., Zaria.
2.	Digital weighing scale (0.0001g)	Mettler, AE 160 Model: 8606, Zurich	NLNG Multiuser Laboratory, A.B.U., Zaria
3.	Fibre Material Testing Machine	Zwick/Roell BDO – FBO-2005/s	Department of Textile and Polymer Science, A.B.U., Zaria
4.	Instron Universal Testing Machine	Model 3369	Obafemi Awolowo University, Ile-Ife, Osun State
5.	Charpy Impact Testing Machine	Model: Cat. Nr. 412	Department of Metallurgical and Materials Engineering, A.B.U., Zaria
6.	Micro Vickers Hardness Testing Machine	NA	Shell Professorial Office, A.B.U., Zaria
7.	Scanning Electron Microscope	Phenom Pro X, Model: Q150R ES	Department of Chemical Engineering, A.B.U., Zaria

NA = Not available

Table 3.1b: List of Equipment used in the study continued

S/No.	Equipment	Model	Source
8.	Dynamic Mechanical Analyser 242	NETZSCH – Geratabau, Model: DMA 242 E	Department of Mechanical Engineering, A.B.U., Zaria
9.	ATR Fourier Transform Infrared Spectrometer	NA	Multiuser Laboratory, Department of Chemistry, A.B.U., Zaria
10.	Optical Microscope	NJF – 120A	Department of Metallurgical and Materials Engineering, A.B.U., Zaria.

NA = Not available

3.3

Methodology

3.3.1 Water retting

Water retting was used to extract BPLSFs from the leaf stalks. This was achieved by submerging bundles of leaf stalks in water for four weeks. The water, penetrating to the central stalk portion, swelled the inner cells, bursting the outermost layer, thus increasing absorption of both moisture and decay producing microbes. The retted stalk after four weeks was dried in open air and stored for a week. This process facilitated fibre removal. Final separation of the fibre was done by breaking process in which the brittle woody portion of the retted stalk was broken by beating and scraping.

3.3.2 Surface treatment

- i. Treatment with sodium hydroxide (NaOH): Surface treatment with NaOH was carried out to modify the fibre surface. The fibre was immersed in 6 wt.% solution of NaOH for an hour at room temperature (25 - 26°C), after which it was washed with distilled water and the effect of NaOH was neutralized in 2% glacial acetic acid solution according to Ansell and Aziz (2004); Thomas *et al.* (2004). The fibre was oven-dried for 3 hours at 110°C.
- ii. Latex treatment with natural plant latex: The NaOH treated fibre was then given latex coating by dipping into a natural plant latex solution having 10% dry rubber content, dissolved in water according to Sreekala *et al.* (2002). Thereafter, the fibre was oven-dried for 3 hours at 110°C.

3.3.3 Production of epoxy/BPLSF reinforced composite

Wooden moulds (Plate II) were used in the production of the epoxy/BPLSF reinforced composite. The dimensions and shapes of cavities were made according to the size and shape of the samples as per ASTM standards. The composites were formed using the hand lay-up method, with six different fibre loading (0 wt.%, 5 wt.%, 10 wt.%, 15 wt.%, 20 wt.% and 25 wt.%). The resin was mixed with the hardener in the ratio 2:1, the weighed fibres already cut to sample length were carefully laid in the mould cavity and there after impregnated by the mixed resin. Three samples were produced for every weight percentage fibre loading.

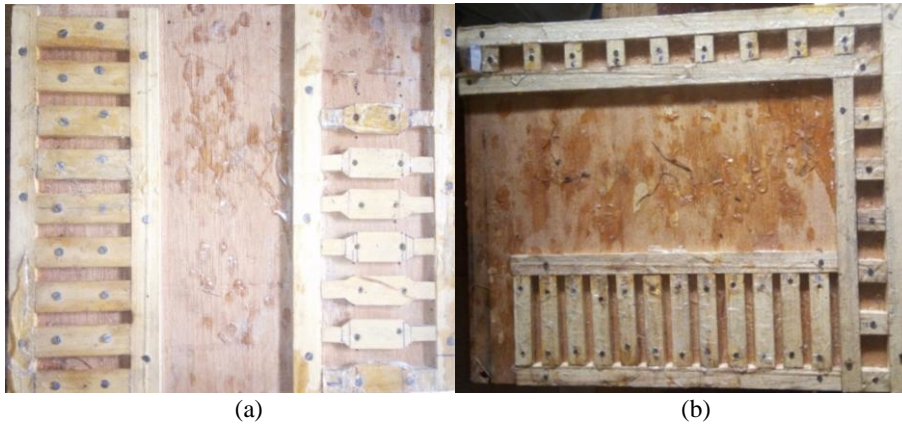


Plate II: Wooden moulds (a) for DMA and tensile test samples; (b) for hardness and flexural test samples

3.3.4 Curing condition

When the composite had hardened, it was removed from the mould and placed in the oven for 6 hours at 50°C for curing according to Sarki (2012).

3.4

Physical Properties

3.4.1 Determination of density

The basic method of determining the density of composite was by measuring the mass and the volume of the sample and estimating the density based on Archimedean principle. Samples were weighed using a digital balance and then suspended in water in a calibrated measuring cylinder. The volume of the samples were determined by displacement method. The densities of samples were then calculated using the expression in Equation (3.1).

$$\text{Density} = \frac{\text{mass}}{\text{volume}} \quad (3.1)$$

3.4.2 Water absorption test

Samples were sectioned, oven dried and then weighed (and the weight measured taken as initial weight, w_i) and immersed in distilled and river water for 30 days. The samples were removed from the water after every 24 hours, cleaned and dried by wiping with a clean cloth, and weighed (final weight, w_f), then immersed in water again over this period according to the procedure recommended by ASTM D570 for water absorption of plastics. The percentage moisture content was evaluated using the Equation:

$$\% \text{ moisture content} = \frac{w_f - w_i}{w_i} \times 100 \quad (3.2)$$

3.4.3 Degradability test

Degradability test of composite was carried out by assessing the influence of soil burial and exposure to natural weather on the samples.

(a) Soil burial test: The samples for soil burial test were prepared according to the requirement of hardness test, the specimens were oven dried at 50°C for 6 hours and

then buried at a depth of 20cm for 90 days. The samples were removed after 90 days, cleaned with detergent and oven dried at 50°C for 6 hours, reweighed and then followed by simple weight loss calculations using Equation (3.3). The test was conducted during the harmattan season from mid-October, 2016 to mid- January, 2017 and mid-May, 2017 to mid-August, 2017 rainy season.

(b) Exposure to natural weather: Samples were produced as per hardness test standard, after which the oven dried samples were exposed to natural weather using the customized weather –rackets method (samples clipped to safety pins were suspended on a copper wire line) for 90 days. The procedure was followed by simple weight loss calculations using Equation (3.3). The test was conducted during the harmattan season from mid-October, 2016 to mid- January, 2017 and mid-May, 2017 to mid-August, 2017 rainy season.

$$\text{Percentage weight loss} = \frac{w_i - w_f}{w_i} \times 100 \quad (3.3)$$

Where:

w_i = initial weight

w_f = final weight

3.5 Mechanical Tests

The study of mechanical properties such as tensile, flexural, impact and hardness behavior of epoxy/BPLSF reinforced composite were conducted according to ASTM standards.

3.5.1 Single fibre tensile test

Single fibres were manually separated from the fibre bundles and cut to the length of 100mm. The strength of the fibres were determined according to ASTM D – 3379 – 75. Gauge length of 50mm and cross head speed of 1mm/min were employed. The machine is equipped with 500N standard load cell. Specimens were gripped at both ends on a Zwick/Roell fabric testing machine and pulled until failure, the fibre strength is then read on a screen. Five readings were taken for each specimen and the average value recorded.

3.5.2 Tensile test

Tensile test was carried out using Instron 3369 Universal Testing Machine with load cell capacity of 50KN. Dumbbell standard samples according to ASTM (D638) were prepared (Plate III), specimen measuring $100.0 \times 8.0 \times 4.0$ mm were positioned in the grips of the testing machine. The grips were tightened firmly to prevent any slippage with gauge length kept at 60mm. The test was conducted at constant cross head speed of 5mm/min. As the tensile test starts, the specimen elongates; the resistance of the specimen increases, and is detected by a load cell. These load values were recorded on a computerised screen until a rupture of the specimen occurred. Threesamples of each composite were used for the test and the average results recorded.

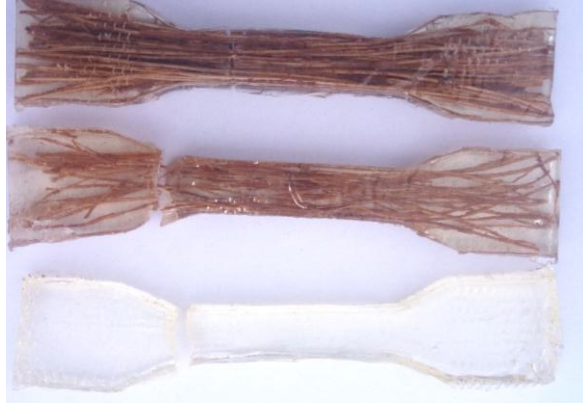


Plate III: Fractured tensile test samples

3.5.3 Flexural test

Flexural test was performed on specimens measuring $100.0 \times 10.0 \times 8.0\text{mm}$ (Plate IV) according to ASTM D790 using computerized Instron 3369 Universal Testing Machine with load cell capacity of 50KN. Three-point bend flexural test method was used with cross-head speed of 5mm/min and span length of 65mm. Samples were positioned on the support span and the load was applied to the centre by the loading nose producing three point bending. The test was stopped at 5% deflection.



Plate IV: Ruptured flexural test samples

3.5.4 Impact test

Charpy impact test was conducted on all the composite samples using impact testing machine using 15J hammer. The test samples were produced according to ASTM 2000 with dimension of $100.0 \times 10.0 \times 8.0$ mm (Plate V). Before the test, sample was mounted on the machine, the pendulum was released to calibrate the machine. The test samples were then gripped horizontally in a vice and the freely swinging pendulum provided the force required to break the bar. The value of the angle through which the pendulum swung before the test sample was broken corresponded with the value of the energy absorbed in breaking the sample, and this was read from the calibrated scale of the machine.



Plate V: Impact test samples

3.5.5 Hardness test

Micro Vickers hardness test was conducted on the specimens produced according to ASTM C1327 with dimension of $30.0 \times 30.0 \times 10.0$ (Plate VI). Indentation technique using Vickers diamond pyramid indenter on the micro hardness tester was used. The measurement was done on the surface by applying 0.3kg load for 15 seconds. Three Vickers hardness readings were taken for each sample and the average recorded.



Plate VI: Hardness test samples

3.6 Single Fibre Diameter Measurement

Optical microscope in conjunction with a computer software “Digimizer” was used to measure fibre diameter. Prior to the measurement, calibration was done using a laboratory slide. Each sample of fibre consists of five specimens on which three adjacent readings were taken and averages recorded to the nearest 10 μm .

3.7 Dynamic Mechanical Analysis (DMA)

NETZSCH – Geratabau DMA 242E was used to determine the thermo-mechanical properties of the composite produced. Samples were produced with dimensions 60.0 mm \times 10.0 mm \times 4.0 mm (Plate VII). The Test was performed at frequencies of 2 Hz, 5 Hz and 10 Hz. The specimens were heated from 30°C to 140°C at heating rate of 3°C/min and the

experiment was carried out in the 3 point deformation mode with preloading force of 1.1N and amplitude of 60 μm .



Plate VII: Dynamic mechanical analysis samples

3.8 Scanning Electron Microscopy (SEM)

SEM was carried out on the surfaces of impact fractured specimens and water absorbed samples. The test was carried out using Phenom Pro – X Scanning Electron Microscope. The samples were sputter coated with a thin layer of gold (5nm) prior to being exposed to electron beam to make it conductive for proper imaging. The image was captured on an attached computer monitor.

3.9 Fourier Transform Infrared (FTIR) Spectroscopy

The FTIR spectra of untreated and treated fibres were obtained using the Agilent Technologies attenuated total reflection spectrophotometer. This was done by clamping the sample tightly on the spectrophotometer and transmitting the IR through the probe. Spectra that correspond to functional groups stretching or vibration were captured on an attached computer and printed out for further analysis.

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Fibre Characterisation

4.1.1 Fibre diameter measurement

From the result obtained by Digimizer software on photomicrographs of the fibre, average fibre diameter was 350 μm for untreated fibre, and 140 μm for the treated fibres to the nearest 10 μm .

4.1.2 Single fibre strength

Figure 4.1 presents the results of single fibre tensile strength and average fibre diameters of untreated, alkali treated and latex treated fibres.

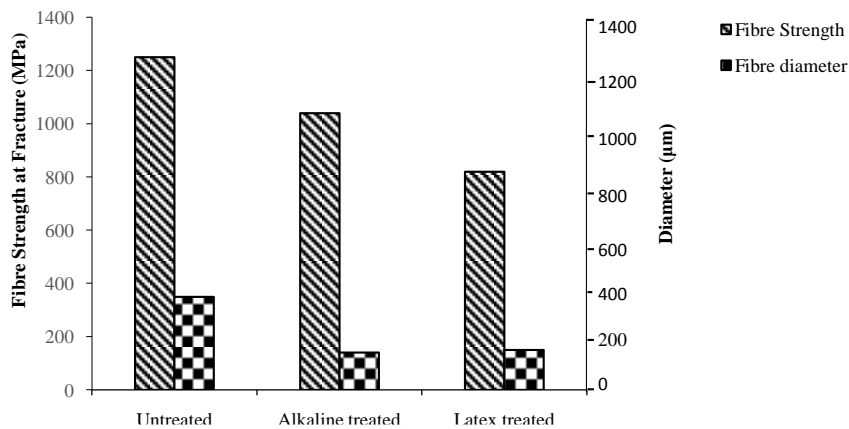


Figure 4.1: Average fibre strength at fracture and fibre diameter

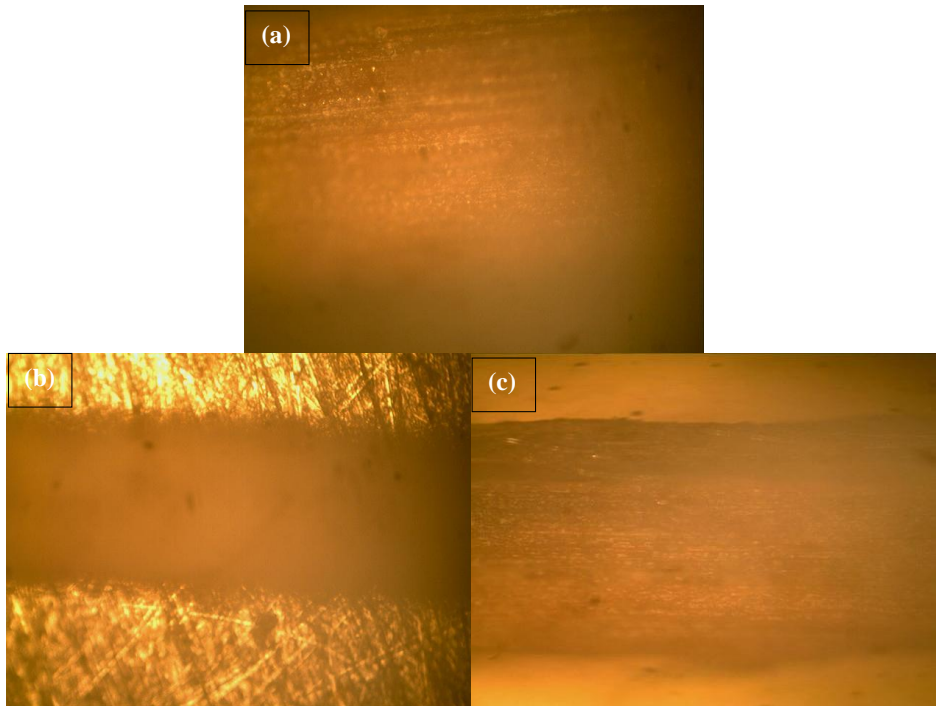


Plate VIII: Micrographs showing fibre diameter X 100 (a) Untreated; (b) alkali treated and (c) latex treated

A comparison of the untreated and the treated fibres indicated that untreated fibre has the highest average fibre strength (σ_f) of 1250MPa and average diameter of 350 μm ; followed by alkaline treated fibre with average strength at fracture of 1040 MPa and average diameter of 140 μm , and the latex treated fibre exhibited the least average strength at fracture of 820 MPa and average diameter of 150 μm .

Reduction in strength of alkali treated fibre compared to the untreated fibre may be attributed to loss of intracellular hemicellulose and cell-wall lignin between microfibrils during NaOH surface treatment, thereby destroying the packing in cellulose chain and causing disorder in crystalline pattern (Zhu *et al.*, 2013). Reduction in average diameter in alkali treatment fibre compared to the untreated fibre is as a result of removal of alkali

soluble amorphous matters such as impurities, waxes, oil, hemicellulose and lignin (Kabir *et al.*, 2012).

Further reduction in fibre strength with latex treatment may be attributed to more disruption in packing of the cellulose chains which cause disorder in the crystalline pattern. This agrees with the findings of Singha and Rana (2012) who reported that crystallinity of fibre increased after mercerization but decreased after silane treatment.

4.1.3 Fourier transform infrared spectroscopy (FTIR) characterisation

Figures 4.1 to 4.3 and Tables 4.1 to 4.3 present the results obtained from attenuated total reflection (ATR) FTIR analysis.

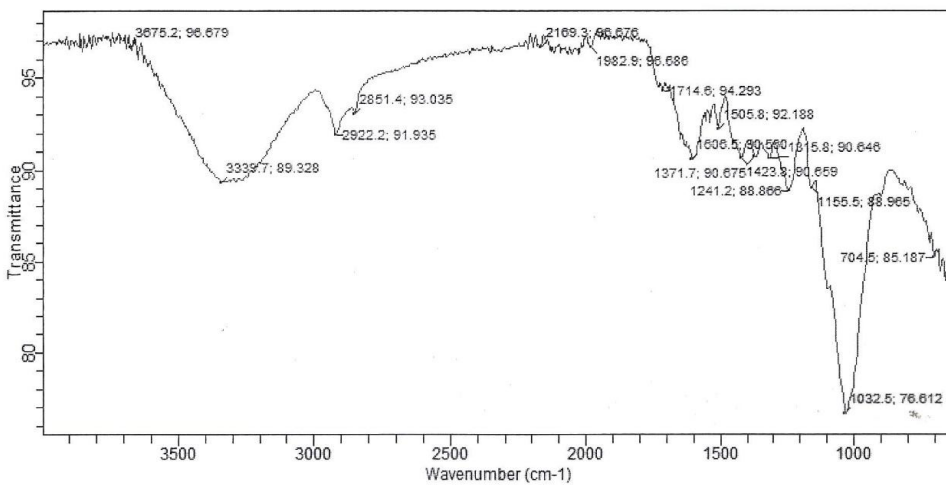


Figure 4.2: FTIR spectrum for untreated fibre

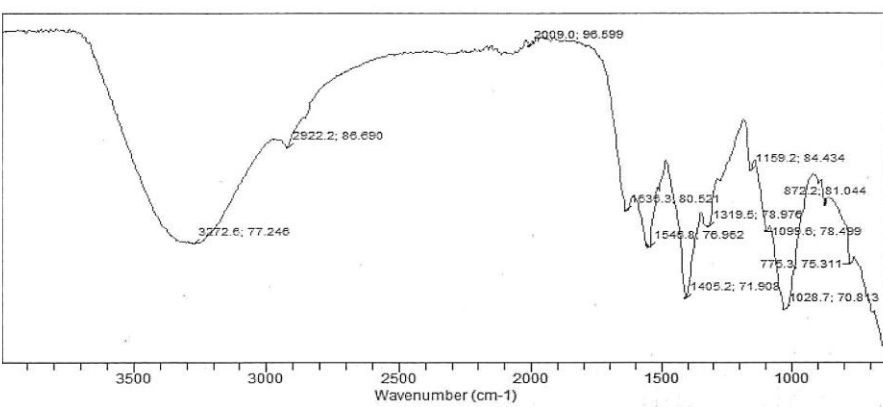


Figure 4.3: FTIR spectrum for alkaline treated fibre

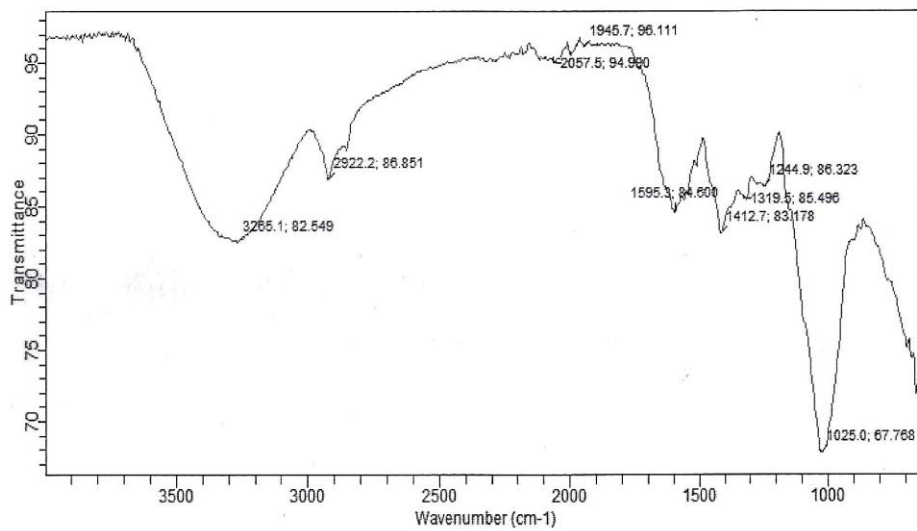


Figure 4.4: FTIR spectrum for latex treated fibre

Table 4.1: Untreated Fibre Functional Groups, Nature and Types of Vibration

Wave Number (cm ⁻¹)	Functional Groups, Nature and Types of Vibration
3675.2	Weakly bonded O – H stretch
3339.7	Hydrogen-bonded O – H stretch
2922.2	C – H Stretch
2851.4	C – H stretch off – C = O
2169.3	C ≡ C stretch HC ≡ C – CH ₃
1714.6	C = O stretch (R – C (O) – OH)
1606.5	C = C stretch
1505.8	N – H bend
1371.7	N – O stretch
1315.8	N = O bend

Table 4.2: Alkaline Treated Fibre Functional Groups, Nature and Types of Vibration

Wave Number (cm ⁻¹)	Functional Groups, Nature and Types of Vibration
3272.6	≡ C – H stretch HC ≡ C – CH ₃
2922.2	C – H Stretch
2009.0	C ≡ C Alkynes (R – C ≡ C – H)
1636.2	C = O stretch (R – C (O) – NR'R'')
1546.0	N – H bend
1405.2	C – H bend
1319.5	N = O stretch
1159.2	C – O stretch 3°
1099.6	C – O stretch 2°

Table 4.3: Latex treated fibre functional groups, nature and types of vibration

Wave Number (cm ⁻¹)	Functional Groups, Nature and Types of Vibration
3265.1	≡ C – H stretch (HC ≡ C – CH ₃)propyne
2922.2	C – H Stretch
2057.5	C ≡ C(R – C ≡ C – H)
1595.3	C – C = C symmetric stretch
1412.7	Aromatic ring OR N – H bend
1319.5	C – N Amines
1244.9	= C – O – C asymmetric stretch
1025.0	= C – O – C symmetric stretch
1099.6	N = O stretch

From Tables 4.1 to 4.3, it was observed that C – H stretch at 2922.2 cm⁻¹ was present in all fibres. The carbonyl peak at 1714.6 cm⁻¹ in the untreated fibre was seen to have been reconfigured in the alkaline treated fibre. The removal of hemicellulose from the fibre surface caused this peak to disappear. The lignin peak at 1505.8 cm⁻¹ in the untreated fibre was altered by alkaline treatment with further alteration in the latex treated fibre. Latex treatment introduced amine and asymmetric and symmetric stretch = C – O – C, which could improve the reactivity of the fibre with the matrix. Sgriccia *et al.* (2008) reported that changes in peaks in the FTIR spectrum at 1730, 1625 and 1239 cm⁻¹ indicated that the alkali treatment removes hemicellulose and lignin from natural fibre surfaces.

4.1.4 Fibre morphology

Plate VIII a and b is SEM of fibre morphology

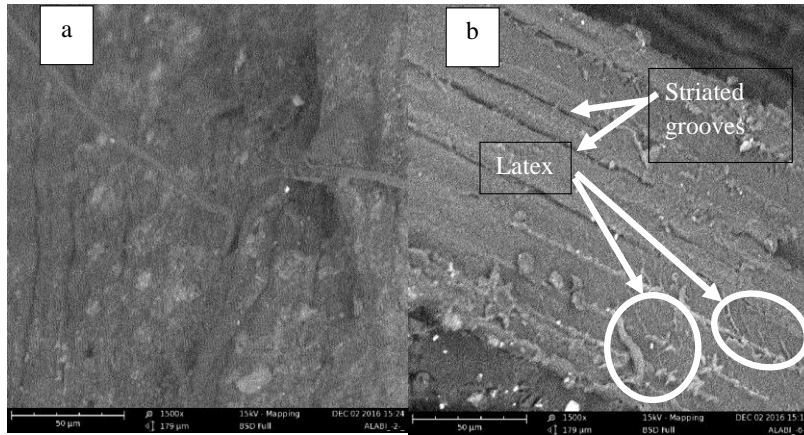


Plate IX: SEM of fibre surfaces morphology (a) untreated (b) Latex treated

From plate VIII, it can be clearly seen that interfibrillar material, hemicellulose and lignin present in (a) are etched away in (b) by the NaOH treatment. This is in accordance with Sgriccia *et al.*, (2008) who found that kenaf and hemp fibre morphology changed after NaOH treatment with SEM revealing evidence of removal of pectin, waxy substances and natural oils covering the surface of fibre cell wall. They further stated that this makes the fibre surface rough by revealing the fibrils. Haque *et al.*, (2015) reported that fibrillation was observed between fibre cells in alkali treated sisal fibre and that rougher surface was observed in SEM of the treated sisal. Ansell and Aziz (2004) reported similar result. They observed that SEM of hemp fibres show a very clean surface after 6% NaOH treatment, and stated that wax, oil and surface impurities were observed on the SEM micrographs of the untreated fibre.

4.2 Composite Characterisation

4.2.1 Physical properties

a. Density determination

The results for the density values of the matrix (epoxy) and the composites produced with the untreated and treated fibre are shown in Figure 4.4, and Table A1 in Appendix A.

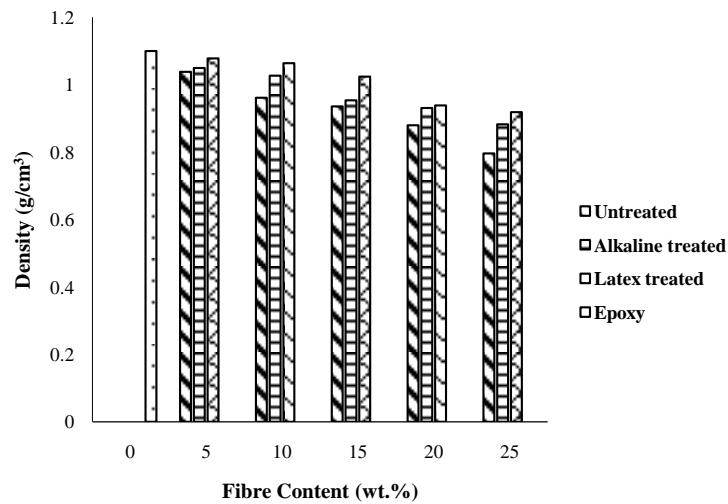


Figure 4.5: Effect of fibre content (wt%) on the density of treated and untreated fibre reinforced composite

From Figure 4.5, it was observed that densities of both untreated and treated fibre reinforced composites were lower than that of the matrix. It was also seen that for all composites produced there was a general decrease in density with increasing fibre content. The untreated fibre reinforced composite had the least density of 0.7967 g/cm^3 at 25 wt. % fibre content. Latex treated fibre reinforced composite exhibited the highest density of 1.0786

g/cm³ at 5wt.% fibre content. The alkaline treated fibre reinforced composites exhibited intermediate density values.

The densities of the untreated fibre reinforced composites lied between 0.7967 and 1.0381 g/cm³, indicating lower density values than that of the alkaline treated fibre reinforced composites which lied between 0.8827 and 1.0498 g/cm³. The latex treated fibre reinforced composites showed the highest density values, ranging from 0.9192 to 1.0786 g/cm³. Density value for epoxy was 1.100g/cm³.

The reported epoxy density values from other studies were 1.16 g/cm³ (Chanap, 2012) and 1.15 g/cm³ (Mishra, 2014). Decrease in density values of all composites with increase in fibre content may be attributed to lower density value of the fibre compared to that of the matrix (Ngargueudedjim *et. al.*, 2015b). Alkaline treated fibre reinforced composites exhibited higher density values than the untreated fibre reinforced composites due to a better interfacial bonding that lead to improved compatibility between the fibre and matrix, thereby reducing the amount of voids in the composites (Shehu, 2015). Latex treated fibre reinforced composites exhibited the highest density values which may be as a result of better compatibility, reduction in the amount of microvoids and increase in average fibre thickness/weight as a result of latex coating layer on fibre surface.

b. Water absorption

Figures 4.6 to 4.9 and Figures C1 to C6 in Appendix C give the water absorption test result profiles of matrix and composites after 30 days of exposure to distilled and river waters.

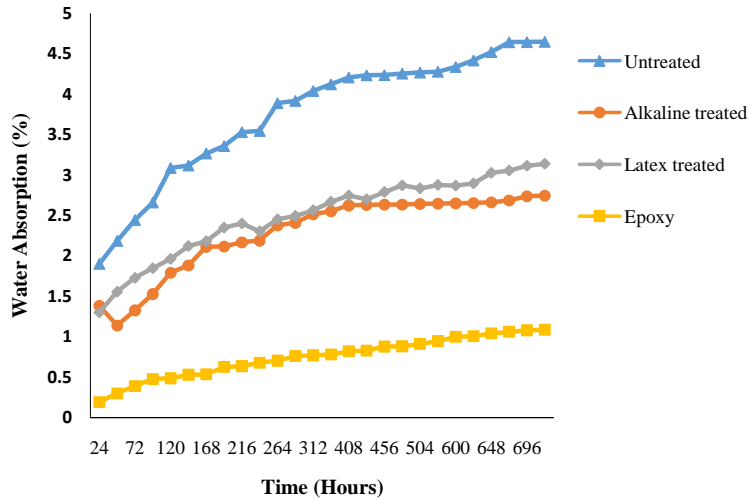


Figure 4.6: Effect of distilled water exposure duration (hrs) on water absorption of 5 wt% treated and untreated fibre reinforced composite

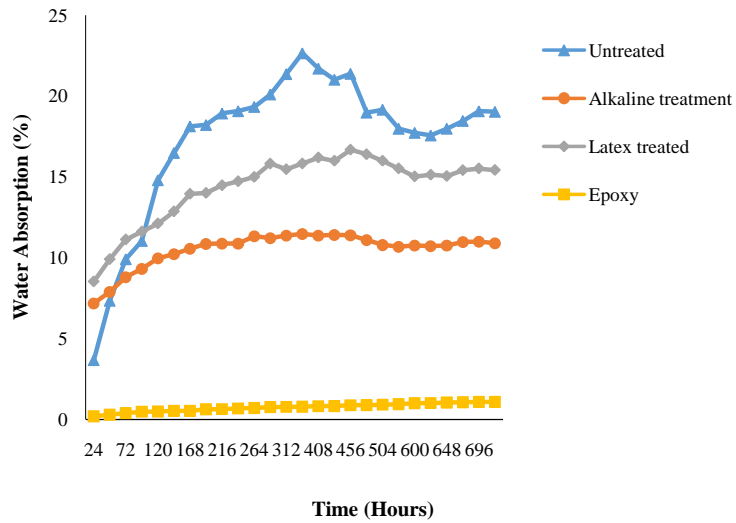


Figure 4.7: Effect of distilled water exposure duration (hrs) on water absorption of 25 wt% treated and untreated fibre reinforced composite

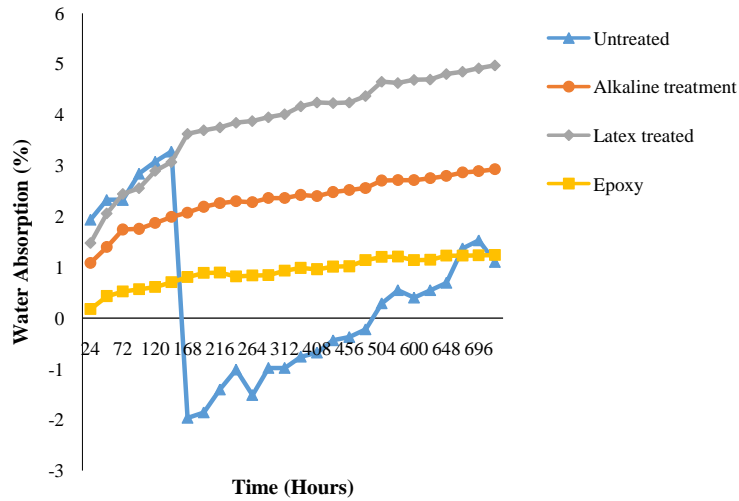


Figure 4.8: Effect of river water exposure duration (hrs) on water absorption of 5 wt% treated and untreated fibre reinforced composite

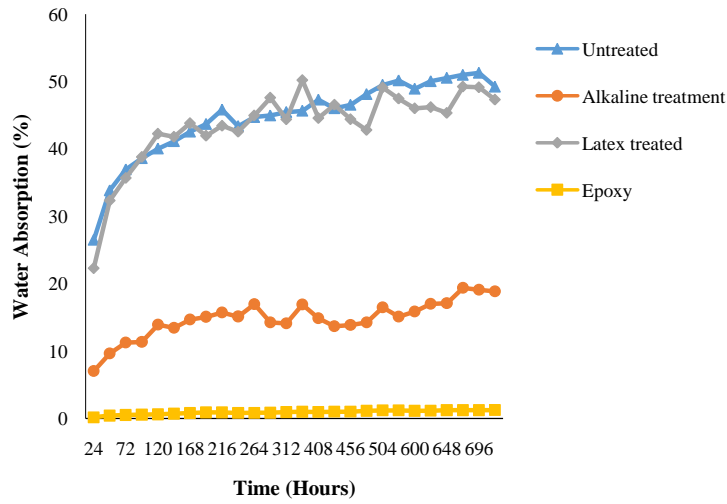


Figure 4.9: Effect of river water exposure duration (hrs) on water absorption of 25 wt% treated and untreated fibre reinforced composite

The Figures above and Figures C1 to C6 in Appendix C present the water absorption of the matrix and all composite formulations as percentage of dry weight per day after continuous immersion in water for 30 days. It was observed that water sorption for the matrix was lower than that of the composites in both distilled and river waters. It was also observed that water absorption increased for all composites with soaking time, but the rate of absorption decreased over time. The matrix absorbed about 1.0832% in distilled water and 1.2428% after 720 hours of immersion in river water. The results also indicated general increase in water absorption with increasing fibre content. Untreated fibre reinforced composites exhibited the highest water absorption in all respects, with 25 wt.% fibre reinforced composite absorbing up to 22.6432% and 51.3041% in distilled and river water after 720 hours, respectively. Alkaline treated fibre reinforced composites showed the lowest intake in both media with 5 wt. % fibre reinforced composite, absorbing 2.7437% and 2.9288% in distilled and river water after 720 hours of immersion, respectively. Latex coated fibre reinforced composites indicated intermediate water absorption between the alkaline treated and untreated fibre reinforced composites.

The lower water intake of matrix compared to the composites demonstrated its stable hydrophobic nature (Sreekala *et al.*, 2002; Shehu, 2015). Increased water absorption of all composites with increasing fibre content wt.% and soaking time agree with the findings of Tawakkal *et al.* (2012) and Abdelmouleh *et al.* (2007). Other factors that water absorption depends on are temperature, orientation of fibre, fibre permeability, area of exposed fibre surface, fibre-matrix interface, and diffusivity, among others (Sreekala *et al.*, 2002). Alkaline treated fibre reinforced composite exhibited least water absorption, and this may be due to reduction in free hydroxyl group which became chemically bonded in the alkaline

treated fibre thereby reducing the hydrophilic nature of the fibre, in tandem with the finding of Sreekala *et al.* (2002) and Zhu *et al.* (2013). Increase in water absorption of latex coated fibre reinforced composite compared to alkaline fibre treated composite may be as a result of the water affinity of the latex due to its polar nature (Sreekala and Thomas, 2003; Sreekumaret *al.*, 2009). FTIR results also proved the introduction of amine and asymmetric and symmetric stretch = C – O – C in latex treated fibres which could improve the reactivity of its composites.

Comparison of water absorption in distilled and river waters showed that epoxy exhibited negligible water absorption in both media (about 1%) over exposure period of 30 days. However, the matrix resin showed an incremental 14.82% water absorption in river water as compared to distilled water. All composites indicated a marked increase in water sorption in river water compared to the distilled water, except for 5 wt. % fibre content untreated composite, where negative values were recorded for river water.

The increment in water absorption of all composites in river water compared to distilled water may be as a result of the differential physico-chemical and microbial characteristics of the two media in terms of pH value, dissolved oxygen, biological oxygen demand, chemical oxygen demand, total dissolved solid, and microbial count, which gave rise to varied microbial and chemical attack on composites in the two media.

c. Water absorption surface morphology

Plate IX a – d are surfaces of composites before and after water absorption

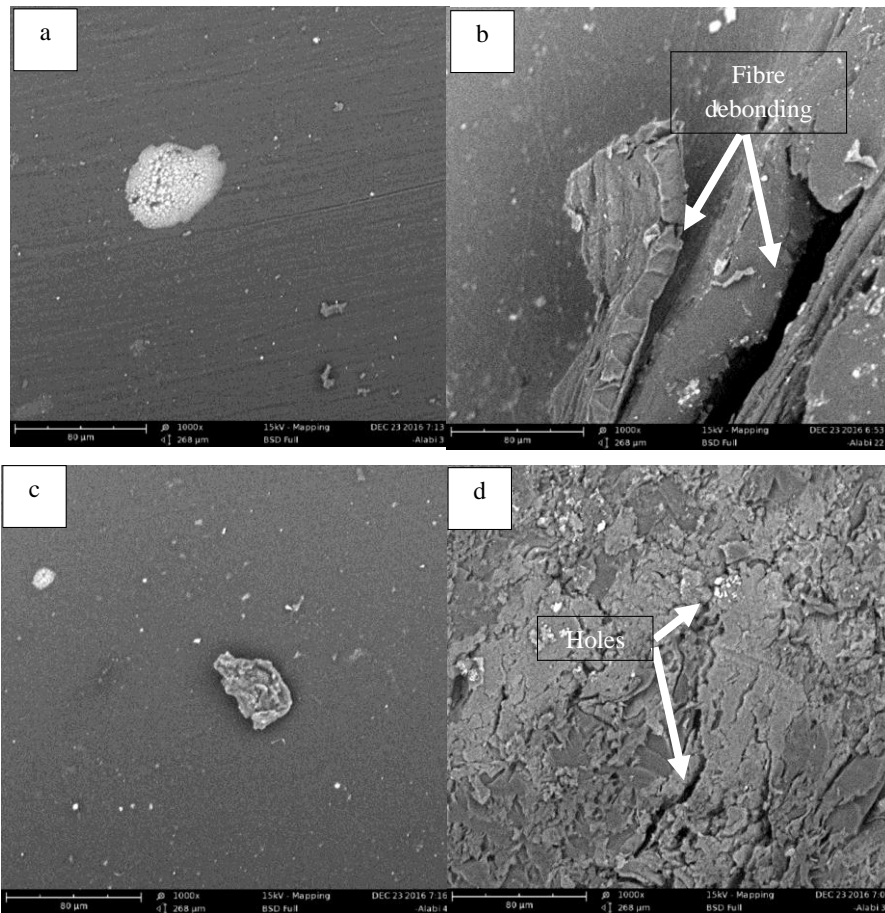


Plate X: SEM of water absorption samples: (a) untreated 25 wt.% fibre content before the test (b) untreated 25 wt.% fibre content after the test (c) latex treated 25 wt.% fibre content reinforced composite before the test (d) latex treated 25 wt.% fibre content reinforced composite after the test

The SEM micrographs in Plate IX show the states of composite surfaces before and after water absorption test. It was observed that continuous water immersion resulted in appearance of some holes and cracks on the surface of the composite. These pores are the channels through which water molecules penetrate into the composite. It could be clearly seen from the micrographs that water absorption of the composite resulted in fibre debonding and loss of surface integrity. Tserki *et al.* (2006a) reported that water absorption could lead to rapid debonding, delamination and a loss of structural integrity, which leads to deterioration of the material's mechanical properties.

4.2.2 Mechanical properties

a. Tensile strength

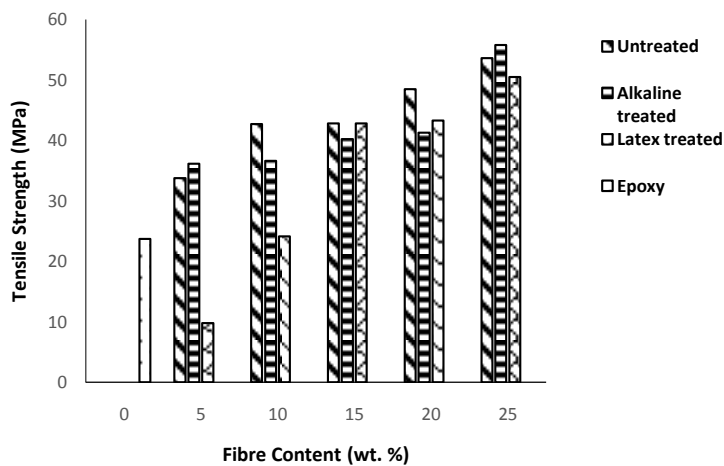


Figure 4.10 presents the tensile strength results of epoxy and all composites formulations.

Figure 4.10: Effect of fibre content (wt.%) on tensile strength of treated and untreated fibre reinforced composite

It can be observed that increasing wt.% fibre content in all composite formulations resulted in increase in tensile strengths of the composite. This may be attributed to good interfacial

bonding between epoxy and *BPLSF*, which agrees with Ku *et al.*, 2011; Mishra and Srivastava, 2014; Venkatesh *et al.*, 2016. They all reported considerable increase in tensile strength with increase in fibre loading, due to the good interaction between matrix and reinforcement.

Tensile strengths of the alkaline treated fibre reinforced composites ranging from 36.12MPa to 41.29MPa at 5wt.% to 20 wt.% were seen to decrease slightly compared to the untreated fibre reinforced composites with tensile strengths ranging from 33.77MPa to 48.44MPa at the same fibre contents, respectively. This may be attributed to the loss of intra-matrix hemicellulose, and cell wall lignin between micro fibrils during NaOH surface treatment, thereby destroying the packing in cellulose chains and causing disorder in crystalline pattern (Singha and Rana, 2012; Zhu *et al.*, 2013).

Latex treated fibre reinforced composite exhibited improvement in tensile strength over that of the alkaline treated composite generally. This may be attributed to the presence of terpenes (which are derived biosynthetically from units of isoprene) in latex coating which makes the fibre more reactive thereby attaching and forming chemical links with active sites on the matrix.

b. Tensile modulus

The results of tensile modulus of the matrix and composites are shown in Figure 4.11.

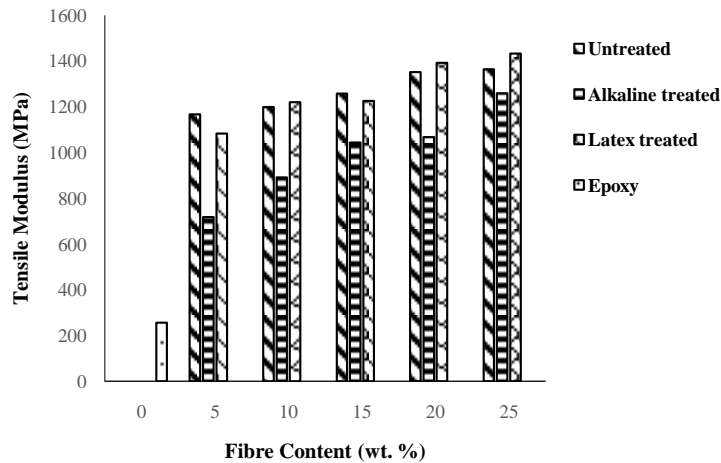


Figure 4.11: Effect of fibre content (wt.%) on tensile modulus of treated and untreated fibre reinforced composite

From Figure 4.11, it was observed that the tensile moduli increased generally with increasing wt.% fibre content. This can be attributed to good fibre-matrix adhesion that results in high degree of stress transfer from matrix to fibre during loading (Heitoret *al.*, 2010). Tensile moduli values of 1166.68MPa and 1256.81MPa for the untreated fibre reinforced composites at 5 wt.% and 15 wt.% were higher than those for the modified fibre reinforced composites with alkaline treated fibre exhibiting the least tensile moduli of 717.37MPa and 1043.26MPa at the same fibre compositions, respectively. Latex treated fibre reinforced composites exhibited the highest tensile moduli values of 1220.01MPa, 1391.41MPa and 1431.72MPa at 10, 20 and 25 wt. % fibre contents, respectively.

The general decrease in tensile moduli of the alkaline treated fibre reinforced composites compared to the untreated fibre reinforced composites may be due to either the loss of

intra-matrix hemicellulose, and cell wall lignin between micro-fibrils during NaOH surface treatment, or insufficient soaking time that may hinder the re-packing in cellulose chains causing disorder in crystalline pattern.

c. Flexural strength

Figure 4.12 presents the flexural strengths of epoxy and all composites formulations.

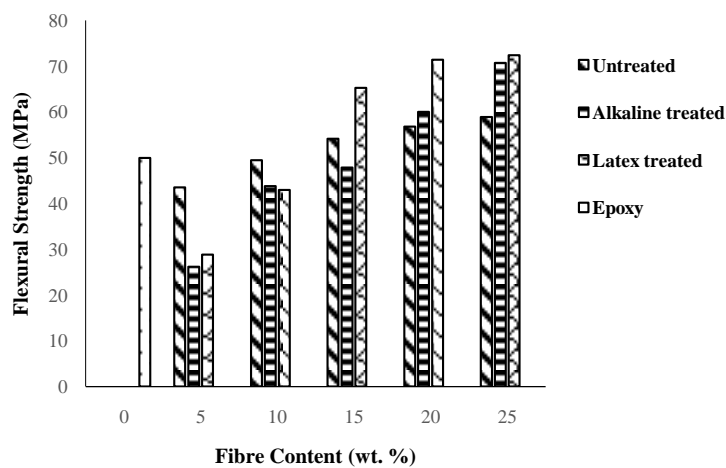


Figure 4.12: Effect of fibre content (wt.%) on flexural strength of treated and untreated fibre reinforced composite

Results for flexural strengths of the composites are shown in Figure4.12. It can be seen that increase in wt.% fibre content of all composites resulted in increased flexural strengths. This findings agreed with the result of Khalid *et al.* (2008) of steady increase in flexural strength with increase filler content, as a result of increasing degree of molecular orientation.

It was observed that alkaline treated fibre reinforced composites exhibited the least values of flexural strengths ranging from 26.17MPa at 5 wt% and 70.72MPa at 25wt.% compared

to the latex treated fibre reinforced composites with flexural strengths ranging from 28.82MPa at 5wt.% to 72.35MPa at 25wt% fibre contents. The result implies that alkali treatment did not have positive influence on the flexural strengths, whereas latex treatment had a considerable positive influence on the flexural strength of the composites.

d. Flexural modulus

The flexural moduli of the matrix and composites are shown in Figure 4.13.

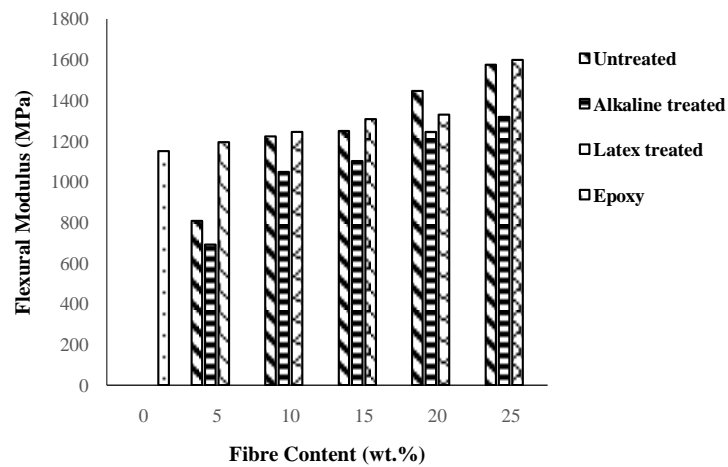


Figure 4.13: Effect of fibre content (wt.%) on flexural modulus of treated and untreated fibre reinforced composite

From Figure 4.13, it was observed that flexural moduli increase generally with increasing wt.% fibre content. Latex treated fibre reinforced composites with flexural moduli ranging from 1195.03MPa at 5wt.% to 1598.04MPa at 25wt.% exhibited the highest flexural strength. This was followed by the untreated fibre reinforced composites with flexural moduli ranging from 807.40MPa at 5wt.% to 1574.81MPa at 25wt.%. The alkaline treated fibre reinforced composites exhibited the least flexural moduli at all fibre content.

e. Impact strength

Figure 4.14 shows the results of impact strengths for epoxy and all the formulated composites.

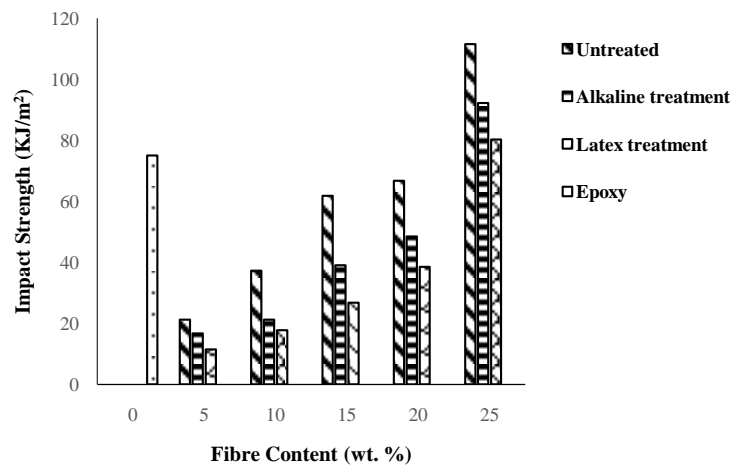


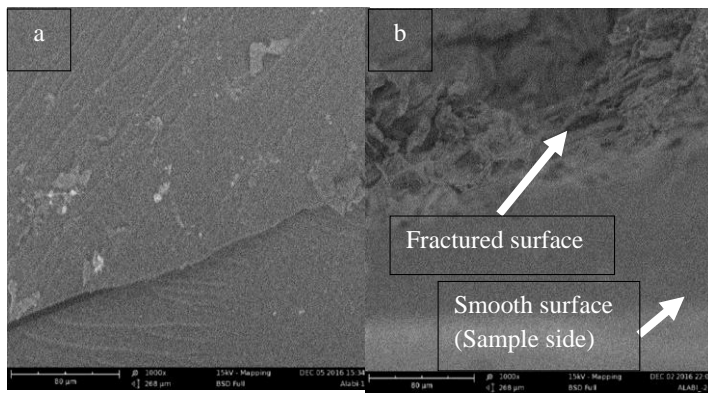
Figure 4.14: Effect of fibre content (wt.%) on impact strength of treated and untreated fibre reinforced composite

Impact strength increased with increase in wt. % fibre content. Comparing the impact strength of the composite with that of the resin, there was a decrease up to 20wt% fibre addition, then an increase at 25wt% addition. This agreed with the report of Pickering *et al.* (2016) that impact strength of epoxy resin reduced with addition of fibre up to 25wt% fibre, but then increased to give overall improvement in impact strength.

For the reinforced composites, the untreated samples exhibited higher impact strength than the treated samples. This may be attributed to reduction in fibre toughness based on the effect of surface modification on fibre make-up.

Morphological studies

SEM of impactfractured surfaces are represented in Plates Xa – f



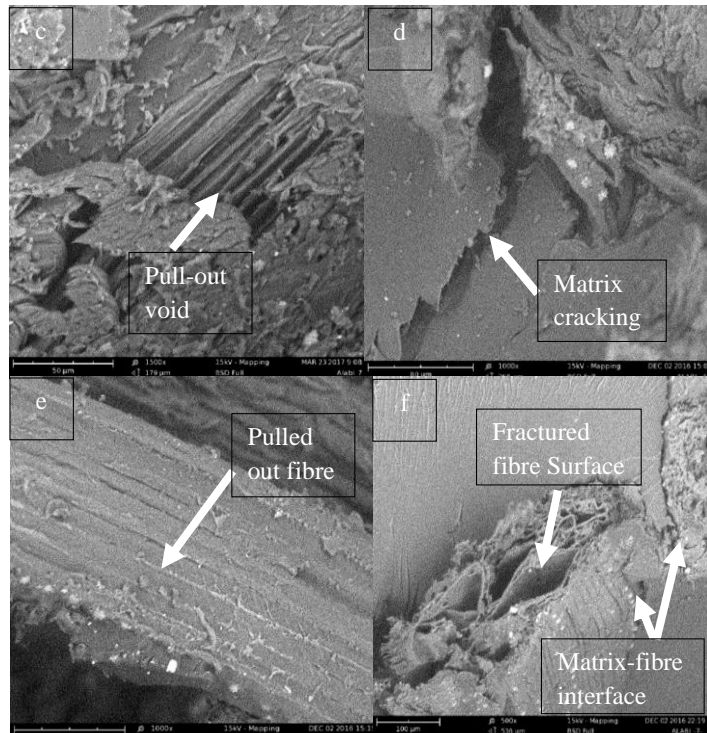


Plate XI: SEM of impact fractured surfaces of composite: (a) unreinforced matrix,(b) 5 wt.% untreated, (c) 25 wt.% untreated, (d) 5 wt.% alkaline treated, (e) 5 wt.% latex treated (f) 25 wt.% latex treated.

Examination of impact fractured surfaces by SEM revealed that epoxy matrix is characterised by smooth fractured surface indicating brittle failure. However, the composites indicated rougher fractured surfaces with voids, matrix cracking and tearing, fibre pull-outs and fibre failures. It could be argued that, though composites showed a more ductile failure as compared to the unreinforced matrix, composites fracture mode was by matrix cracking and tearing, fibre pull-outs and fibre failures. The findings were in accordance with the results of Lee and Wang,(2005) and Thakur and Thakur (2014) that fractured fibre and matrix cracking are observed on fractured surfaces of composites.

f. Hardness test

Figure 4.15 presents the results of hardness values for epoxy and all composites

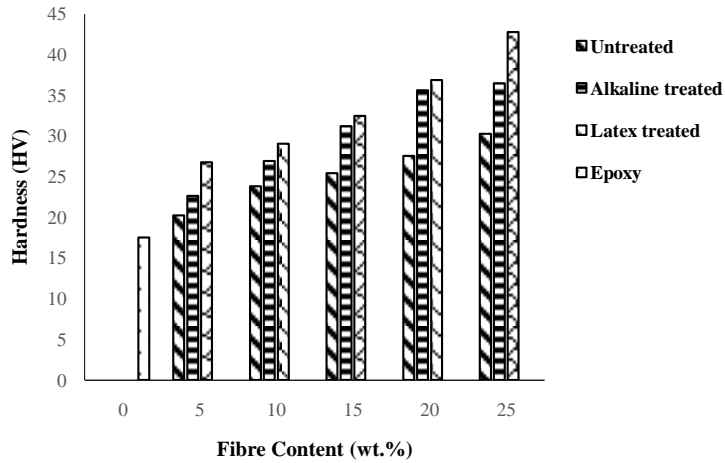


Figure 4.15: Effect of fibre content (wt.%) on hardness value of treated and untreated fibre reinforced composite

Hardness values of epoxy and the composites are shown in Figure. 4.15. Epoxy with 17.5 Micro Vickers hardness (MVH) value exhibited the least hardness value. Micro Vickers hardness values of the composites rose progressively with increase in wt.% fibre content ranging from 20.23 to 30.27MVH for untreated composite at 5 wt.% to 25wt.%, 22.63 - 36.47MVH for alkali treated at 5wt.% to 25wt.% and 26.77 to 42.77MVH at 5 wt.% to 25wt.%MVH for latex treated composites, respectively. This result agree with that of Shehu (2015), who reported a general increase in hardness values with percentage increase in filler content. This finding could be attributed to a more compact or rigid structure on the surface of the composite that led to generation of greater resistance to penetration and, consequently, higher hardness values were obtained for the composites, with increasing

fibre loading (Shehu, 2015). It was also observed that fibre modification improved hardness values in the order: untreated < alkaline treated < latex treated. Increase in hardness with surface modification could be argued in terms of a more rigid and compact fibre structure due to removal of alkaline soluble fractions; such as waxy layers, hemicellulose and lignin.

4.2.3 Degradability test

a. Soil burial test

Figure 4.16 shows the results of soil burial test for epoxy and all composites.

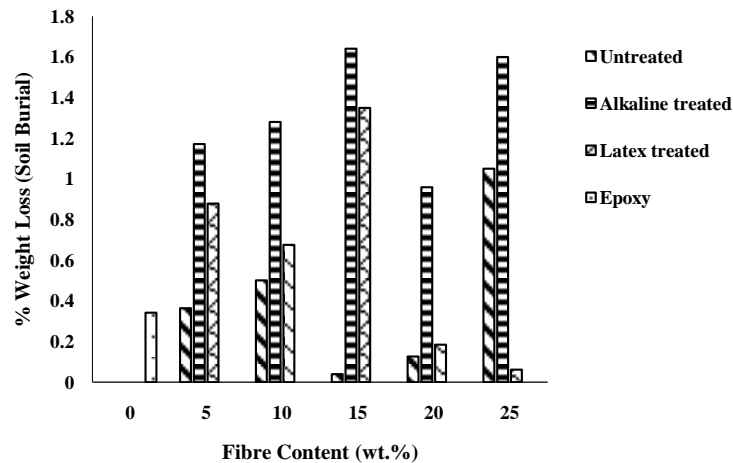


Figure 4.16: Effect of fibre content (wt.%) on weight loss of treated and untreated fibre reinforced composites buried for 90 days in harmatan season

Epoxy degraded only by 0.3% after 90 days of soil burial (Figure 4.16). As expected the thermoset polymer was not biodegradable due to its resistance to microbial attack. Although weight loss increased with wt.% fibre content for all the composites, majority of the composite samples recorded weight loss less than 1% in 90 days with the highest weight loss recorded as 1.6% for 15wt.% alkali treated composite. The results are comparable to (Dixit, 2014) findings on epoxy/coir composite that recorded up to 0.38% weight loss over the same period of time for plain epoxy and 1.98% weight loss on addition of 23.56 wt.% coconut coir.

Tserki *et al.*(2006b) also reported that as the filler content increased, weight loss of composite increased due to the growth of microorganisms which are more in higher filler contents. Yussuf *et al.*(2010) also found weight loss to be two-fold for poly(lactic acid)/rice husk and three-fold for poly (lactic acid)/kenaf composites as compared to the neat poly lactic acid polymer after 90 days of soil burial.

Figure 4.17 shows the results of soil burial test for epoxy and all composites.

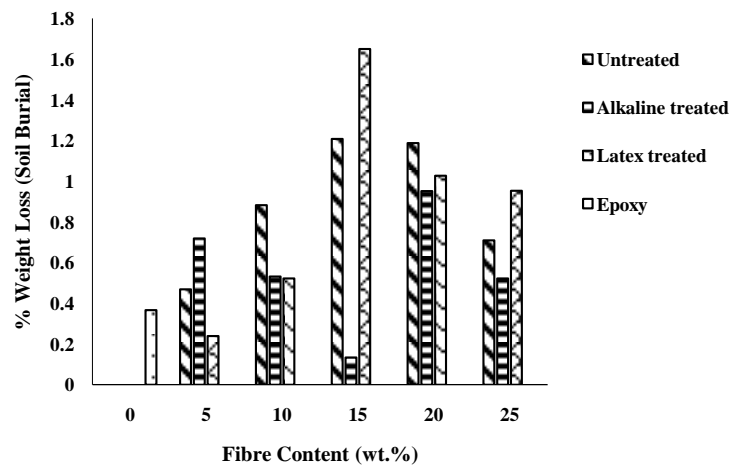


Figure 4.17: Effect of fibre content (wt.%) on weight loss of treated and untreated fibre reinforced composite buried for 90 days in rainy season

From Figure 4.17, the matrix material degraded by 0.36% after 90 days of burial in rainy season. This indicated an incremental degradation of about 0.06% compared to the same material buried over the same period of time in harmatan season. Even though 15wt.% composition exhibited the highest weight loss of 1.65% for latex treated composite in rainy season, as the case was in harmatan season, which recorded 1.64% for alkali treated composite at the same 15wt.% composition. It is difficult to draw conclusion due to

inconsistencies in data generated from this test for both dry cold-harmatan and rainy seasons. Although generally matrix material degraded less than the composites, weight loss increased with increasing wt.% fibre content with 15wt% exhibiting the highest weight loss in both harmatan and rainy seasons.

Kim *et al.* (2006) reported that degradation increased with filler loading because the filler could easily be attacked by microorganism. The degree of biodegradation according to Patel and Sen (2011) depends on temperature, available oxygen and matrix stability. Most microorganisms need water, oxygen, light and favourable temperature in the biodegradation process (Patel and Sen, 2011).

b. Natural Weather Exposure

The result of weight loss in epoxy and composites exposed to natural weather is shown in Figure 4.18 below.

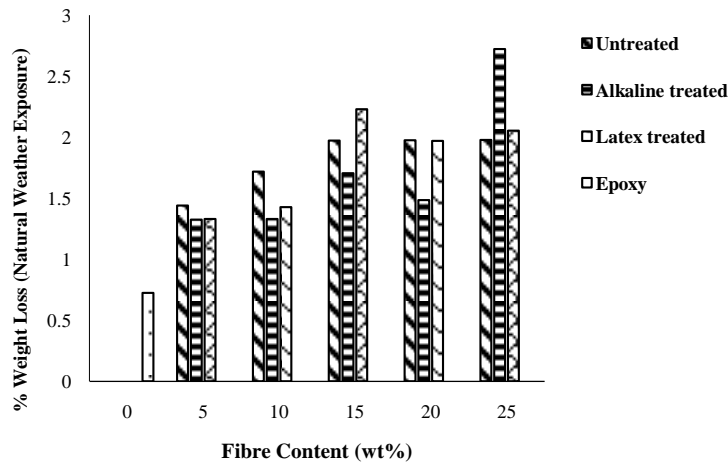


Figure 4.18: Effect of fibre content (wt.%) on weight loss of treated and untreated fibre reinforced composite exposed to natural weather for 90 days in harmatan season

Figure 4.18 shows the weight loss of all samples exposed to natural weather for 90 days. The results obtained for all composites were compared to that of the matrix (epoxy). Epoxy exhibited least weight loss than the composites. Weight loss of composites rose with wt.% fibre content. Alkaline treated fibre reinforced composites showed slight decrease in weight loss for all composite samples except 25 wt.% fibre content, indicating the significance of alkaline treatment on the fibre. Latex treated composite indicated less increase in weight loss compared to alkaline treated fibre reinforced composite. Visual examination of all samples indicated a colour shift from crystal clear to light yellow. Weight loss and colour change in all samples were due to exposure to ultraviolet radiation that led to changes in chemical configuration of samples as a result of covalent chain scission, photo-oxidation or chain cross-linking in the polymer. The findings of the current study agree with the results of previous works of Hill and Abdulkhalil(1999) and Azwa *et al.*(2013) that natural fibre composites exposed outdoor to direct sunlight are subjected to UV radiation, which breaks the covalent bonds in polymers causing yellowing, colour fading, weight loss, surface roughening, mechanical property deterioration and embrittlement with more reduction in wetter conditions. Only yellowing was observed during the course of this work, which is because of the high resistance of epoxy to UV degradation. The yellowing could be eliminated or reduced to the barest minimum through addition of additives such as stabilisers, blockers or colourants.

Figure 4.19 presents weight gain results of natural weather exposed epoxy and composites

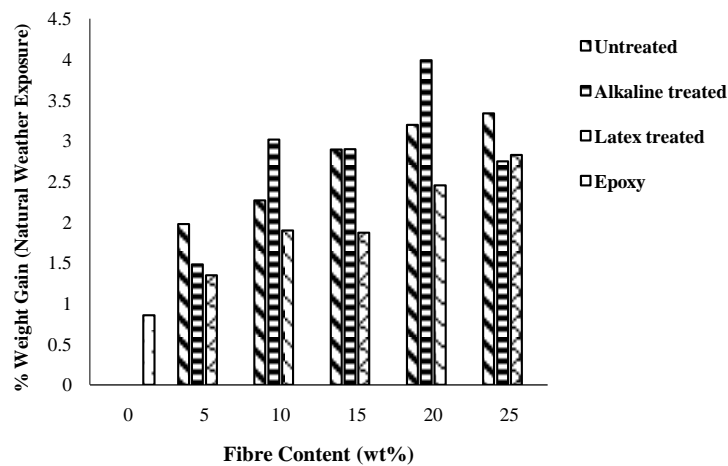


Figure 4.19: Effect of fibre content (wt.%) on weight gain of treated and untreated fibre reinforced composite exposed to natural weather for 90 days in rainy season

From Figure 4.19, it can be seen that both matrix and composites gained weight on exposure to natural weather for 90 days in rainy season. The results obtained for matrix and all composites in the rainy season was opposed to that obtained in the harmatan season. Weight gain was observed in this case while weight loss was the case in harmatan season. Weight gain was higher in composites than in matrix and increased with wt.% fibre content. Visual examination of samples in this case also indicated colour change from crystal clear to light yellow as in the case of harmatan season samples. Weight gain in all samples could be attributed to water absorption of samples from rain water.

4.2.4 Thermo-mechanical Analysis

a. Dynamic mechanical analysis (DMA)

Figures 4.20 to 4.25 show the thermo-mechanical analysis results of epoxy and composites

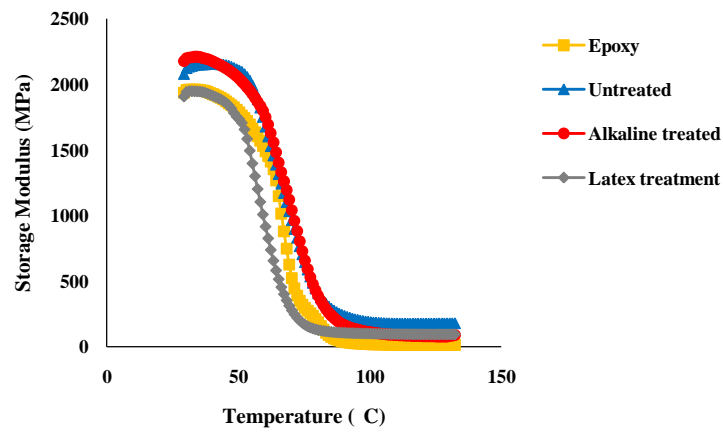


Figure 4.20: Storage modulus against temperature of epoxy and 5 wt.% composites

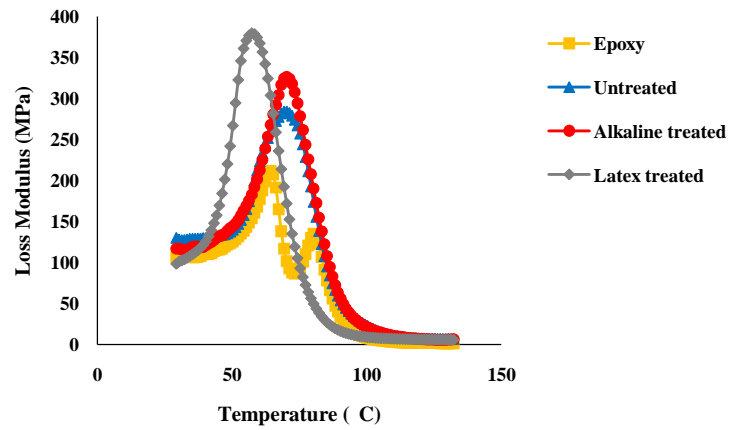


Figure 4.21: Loss modulus against temperature of epoxy and 5 wt.% composites

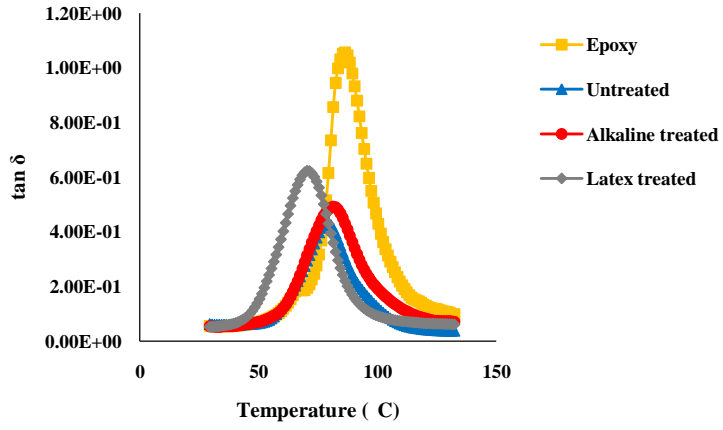


Figure 4.22: Tan δ against temperature of epoxy and 5 wt.% composites

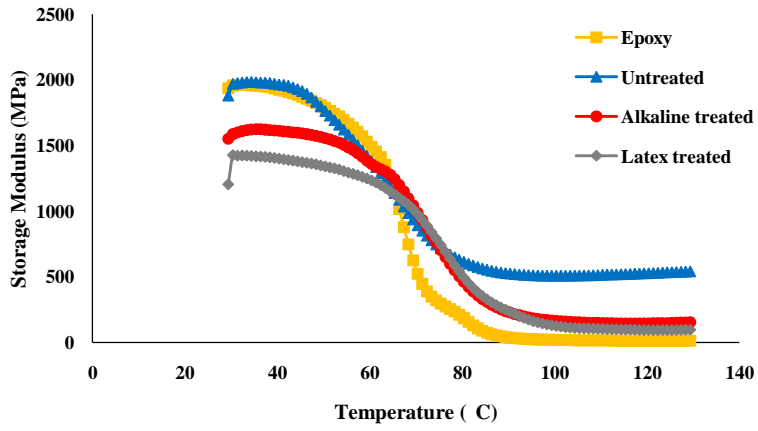


Figure 4.23: Storage modulus against temperature of epoxy and 25 wt.% composites

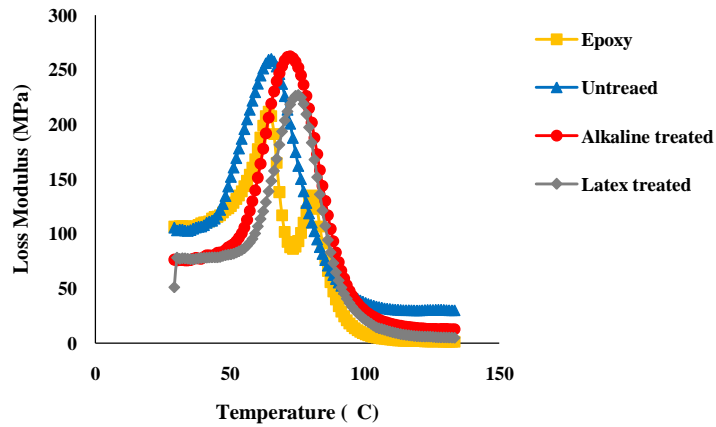


Figure 4.24: Loss modulus against temperature of epoxy and 25 wt.% composites

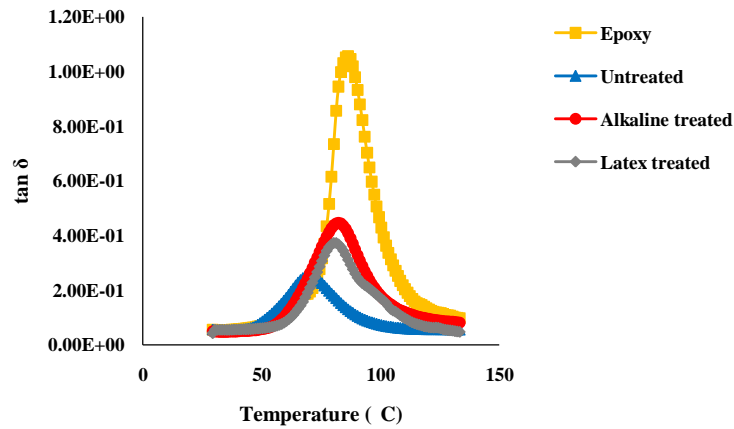


Figure 4.25: Tan δ against temperature of epoxy and 25 wt.% composites

Figures 4.20 – 4.25 show the dynamic storage modulus, loss modulus and tan δ as functions of temperature for epoxy and composites with 5 wt.% and 25 wt.% fibre content, respectively. As the temperature increased, it was observed that storage modulus decreased for both epoxy and all composite samples. This finding may be attributed to an increase in

molecular mobility of covalently bonded chains in the polymer. The value of storage modulus is higher in alkaline treated and untreated fibre-reinforced composites at 5 wt.% fibre content, followed by latex treated fibre reinforced composite and unreinforced epoxy, which have very close values. This result agree with the findings of Huda *et al.*(2005) who showed that at 25 wt.% fibre content however, untreated fibre reinforced composite sample indicated the highest storage modulus. This modulus was slightly higher than that of unreinforced epoxy, the storage moduli for alkaline treated fibre reinforced composite and that of latex treated fibre reinforced composite samples indicating lower storage moduli than that of the matrix.

It was observed that values of loss modulus increased and then decreased with temperature for both epoxy and all composites. Loss modulus for both 5 wt.% and 25wt.% fibre content composites indicate higher peaks than that of the epoxy, the double peaks in the case of the epoxy signifies incomplete crosslinking during curing of the resin. Peak broadening were also observed in the loss modulus curves of the composites, indicating the inhibition of relaxation process as a result of multiple of chain segments hinged on fibre addition. Temperatures at which epoxy and all composites attained the maximum values of loss moduli may be considered as the glass-transition temperature (T_g). This result was in agreement with that of Shanmugam and Thiruchitrabalam (2013) who demonstrated that the temperature at which the specimen attains maximum value of E'' (loss modulus) can also be considered as T_g for the composites.

Tan δ , also called mechanical loss factor, is the ratio of loss modulus to storage modulus. From Figures 4.22 and 4.25, it was clearly observed that peak of tan δ curves reduced for all composites compared to that of the matrix, and this was due to fibre incorporation in the

composites. Decrease in $\tan \delta$ also signifies good adhesion (Shanmugam and Thiruchitraambalam, 2013). The temperature corresponding to $\tan \delta$ peaks is also referred to as the T_g . It could, therefore, be observed that alkaline treated fibre reinforced composite gave the highest T_g values at both 5 wt.% and 25 wt.% fibre content. The result agree with the finding that the position and height of $\tan \delta$ peaks are indicative of the structure and properties of a particular composite material (Heitor *et al.*, 2010).

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

This work was aimed at developing and characterising epoxy/BPLSF reinforced composite by hand lay-up method. At the end of the study, the following conclusions were made.

- i. Characterisation of untreated and treated fibre confirmed the etching away of surface and interfibrillar materials by NaOH treatment, and further revealed latex layers on latex treated fibres that served as reactive sites with epoxy matrix. Single fibre strength showed a progressive decrease in fibre strength with surface treatment. The average strength of 1250 MPa recorded for untreated, 1040 MPa for NaOH treated and 820 MPa for latex treated fibres, respectively.
- ii. BPLSF was successfully used to produce natural fibre reinforced composite using hand lay-up method.
- iii. Density of composite decreased with increase in percentage fibre content, from 1.100 g/cm³ for epoxy to 1.0381g/cm³ for 5wt.% reinforced and 0.7967g/cm³ for 25wt.% reinforced with untreated fibre. For 25wt.%, alkali treatment reduced water up-take of composite from 51.3% for untreated to 19.4%. Composite also absorbed less indistilled water with uptake of 22.6% for untreated, 11.4% for alkali treated and 16.7% for latex treated composite at the same fibre content of 25wt. %. The tensile, flexural and impact strengths increase generally with percentage increase in fibre content. Tensile strength of epoxy (10.45608MPa) increased about five fold (55.74935MPa) on addition of 25wt. % alkali treated fibre. Flexural strength increased from 49.93321MPa for epoxy to 72.34649MPa for latex treatment at

25wt% fibre content. Impact strength also increased from 75.00KJ/m² for epoxy matrix to 111.50 KJ/m² at 25wt. % untreated fibre content addition. Hardness value increased progressively with percentage fibre content to more than double at 25wt.% latex treatment with hardness value of 42.77 compared to 17.50 for epoxy. Alkaline treated fibre reinforced composite gave better storage and loss moduli, untreated fibre have the least tan δ value. Morphological studies of impact fractured sample surfaces revealed that failure of composites is by matrix cracking, tearing, fibre failure and pull-out. In the case of water absorption, untreated fibre-reinforced composite revealed fibre debonding.

- iv. Degradability of the composite increased with percentage fibre content in both soil burial (1.6%) and natural weather exposure (2.7%).

5.2

Recommendation

From the results obtained the following are recommended:

- i. Study of composite produced through other means such as resin transfer moulding (RTM) and resin injection molding (RIM) could be carried out.
- ii. Effect of other treatments other than alkaline and latex treatment could be investigated on the fibre.
- iii. Factors that increase water absorption in river water should be studied.
- iv. The effect of stabilisers, blockers and colourants addition on composite degradability should be looked into.

REFERENCES

- Abdal-hay, A., Suardana, N. P. G., Jung, D. Y., Choi, K.-S. and Lim, J. K. (2012). Effect of diameters and alkali treatment on the tensile properties of date palm fibre reinforced epoxy composites. *International Journal of Precision Engineering and Manufacturing*, 13(7): 1199 – 1206.
- Abdelmouleh, M., Boufi, S., Belgacem, M.N. and Dufresne, A. (2007). Short natural-fibre reinforced polyethylene and natural rubber composites: Effect of silane coupling agents and fibres loading. *Composites Science and Technology*, 67: 1627–1639.
- Acheampong, J.B. (2014). Physico-chemical properties and natural durability within two varieties of *Borassus aethiopum*. A Thesis Submitted to the Department of Wood Science and Technology, Kwame Nkrumah University of Science and Technology, Kumasi, Ghana. 8 – 29.
- Amirou, S., Zerizer, A., Haddadou, I. and Merlin, A. (2013). Effect of corona discharge treatment on the mechanical properties of biocomposites from polylactic acid and Algerian date palm fibres. *Scientific Research and Essays*, 8(21): 946 – 952.
- Ansell, M.P. and Aziz, S.H. (2004). The effect of alkalization and fibre alignment on the mechanical and thermal properties of kenaf and hemp bast fibre. *Composites: Part 1 – Polyester resin matrix, Composite Science and Technology*, 64: 1219 – 1230.
- Araoye, B.O. (2015). A Study of Development, characterisation and degradability of polyester/nano-locust beans pods (*Pakia biglobosa*) ash composite. An Unpublished M.Sc. Thesis, Ahmadu Bello University, Zaria. p 2.
- Azwa, Z.N., Yousif, B.F., Manalo, A.C. and Karunasena, W. (2013). A Review on the degradability of polymeric composites based on natural fibres. *Materials and Design*, 47: 424–442.
- Bhattacharyya, D., Yuan, X. and Krishnan, J. (2004). Effect of plasma treatment in enhancing the performance of woodfibre/polypropylene composites. *Composites Part A* (35): 1363 – 1374.
- Bharath, K.N. and Rajesh, A.M. (2010). Moisture Absorption Characteristics of Areca/Maize reinforced hybrid polymer composites. *International Journal of Advanced Engineering and Application*, 1: 207 – 211.
- Begum, K. and Islam, M.A., (2013). Natural fibre as a substitute to synthetic fibre in polymer composites: A review. *Research Journal of Engineering Sciences*, 2: 46 – 53.
- Bledzki, A.K. and Gassan, J. (1999). Composites reinforced with cellulose based fibre. *Progress in Polymer Science*, 24: 221 – 274.
- Bongarde, U.S. and Shinde, V.D. (2014). Review of natural fibre reinforcement polymer composites. *International Journal of Engineering Science and Innovative Technology*, 3: 431 – 436.

- Chanap, R. (2012). Study of Mechanical and Flexural Properties of Coconut Shell Ash reinforced Epoxy Composites. A Project Submitted to Department of Mechanical Engineering, National Institute of Technology, Rourkela, India. P 8.
- Dixit, S. (2014). Degradation analysis of lignocellulosic fillers infused coir epoxy composites in different environmental conditions. *International Journal of Lignocellulosic Products*, 1(2): 160 – 179.
- Ebewele, R.O. (2000). *Polymer Science and Technology*, CRC Press LLC, Boca Raton, Florida. U.S.A. p 3.
- Emmanuel, B.E. (2010). Fishing crafts characteristics and preservation techniques in Lekki Lagoon, Nigeria. *Journal of American Science*, 6(2):18 – 23.
- Essabir, H., Nekhlaoui, S., Malha, M., Bensalah, M.O., Arrakhiz, F.Z., Qaiss, A. and Bouhfid, R. (2013). Bio-composites based on polypropylene reinforced with almond shells particles: mechanical and thermal properties. *Materials and Design*, 51: 225 – 230.
- Fakhrul, T. and Islam, M.A. (2013). Degradation behavior of natural fibre reinforced polymer matrix composites. *Procedia Engineering*, 56: 795 – 800.
- Fardausy, A., Kabir, M. A., Kabir, H., Rahman, M. M., Begam, K., Ahmed, F., Hossain, M. A. and Gafur, M. A. (2012). Study of physical, mechanical and thermal properties of unidirectional jute fibre reinforced PVC film composites, *International Journal of Advanced Research in Engineering and Technology*, 3: 267 – 274.
- Faruk, O., Bledzki, A.K., Fink, H. and Sain, M. (2012). Biocomposite reinforced with natural fibres: 2000 – 2010. *Progress in Polymer Science*, 37: 1552 – 1596.
- Gu, H. (2009). Tensile behaviours of the coir fibre and related composites after NaOH treatment. *Materials and Design*, 30: 3931–3934.
- Govindaraju, R., Jagannathan, S., Chinnasamy, M. and Kandhavadi, P. (2014). Optimization of process parameters for fabrication of wool fibre-reinforced polypropylene composites with respect to mechanical properties, *Journal of Engineered Fibres and Fabrics*, 9:126 – 133.
- Han, S. O., Cho, D., Park, W. H. and Drzal, L. T. (2006). Henequen/poly(butylene succinate) biocomposites: electron beam irradiation effects on henequen fibre and the interfacial properties of biocomposites. *Composite Interfaces*, 13(2-3): 231 – 247.
- Haque, R., Saxena, M., Shit, S.C. and Asokan, P. (2015). Fibre-matrix adhesion and properties evaluation of sisal polymer composite. *Fibres and Polymer*, 16(1):146 – 152.
- Hassan, E., Wei, Y., Jiao, H. and Muhuo, Y. (2013). Dynamic mechanical properties and thermal stability of poly(lactic acid) and poly(butylene succinate) blends composites. *Journal of Fibre Bioengineering and Informatics*, 6(1):85 – 94.

- Heitor, L.O., Alexandre, S.B., Rudinei, F., Ademir, J.Z. and Sandro, C.A. (2010). Mechanical and dynamic mechanical analysis of hybrid composites molded by resin transfer molding. *Journal of Applied Polymer Science*, 118:887 – 896.
- Hill, C.A.S. and Abdul Khalil, H.P.S., (1999). The Effect of environmental exposure upon the mechanical properties of coir and oil palm fibre reinforced composites. *Journal of Applied Polymer Science*, 77:1322–1330.
- Huda, M.S., Drzal, L.T., Mohanty, A.K. and Misra, M. (2005). Wood fibre reinforced poly(lactic acid) composites. A Paper Presented at 5th Annual SPE Automotive Composites Conference, Troy, Michigan, U.S.A. 1 – 12.
- Imoisili, P.E., Olunlade, B.A. and Tomori, W.B. (2012). Effect of silane coupling agent on the tensile properties of rice husk flour (RHF) polyester composite. *The Pacific Journal of Science and Technology*, 13(1): 457 – 462.
- Inamdar, P.S., Shivanand, H.K. and Kumar, S.S. (2013). Studies on tensile properties of natural fibre polymer matrix composites. *International Journal of Engineering Research and Technology*, 2(3): 1 – 3.
- Huner, U. (2015). Effect of water absorption on the mechanical properties of flax fibre reinforced epoxy composites. *Advances in Science and Technology Research Journal*, 9(26):1 – 6.
- Kabir, M.M., Wang, H., Lau, K.T. and Cardona, F. (2012). Chemical treatment on plant-based natural fibre reinforced polymer composites: An Overview. *Composites, Part B*, (43):2883 – 2892.
- Kalia, S., Thakur, K., Celli, A., Kiechel, M.A. and Schauer, C.L. (2013). Surface modification of plant fibre using environment friendly methods for their application in polymer composites, textile industry and antimicrobial activities: A review. *Journal of Environmental Chemical Engineering*, (1): 97 – 112.
- Khalid, M., Ratnam, C.T., Chuah, T.G., Ali, S. and Choong, T.S.Y. (2008). Comparative study of polypropylene composites reinforced with oil palm empty fruit bunch fibre and oil palm derived cellulose. *Materials and Design*, 29:173 – 178.
- Kijchavengkul, T., Auras, R., Rubino, M., Alvarado, E., Montero, J.R.C. and Rosales, J.M. (2010). Atmospheric and soil degradation of aliphatic – aromatic polyester films. *Polymer Degradation and Stability*, 95: 99 – 107.
- Kim, H.S., Kim, H.J., Lee, J.W. and Choi, I.G. (2006). Biodegradability of bio-flour filled biodegradable poly(butylene succinate) bio-composites in natural and compost soil. *Polymer Degradation and Stability*, 91:1117 – 1127.
- Ku, H., Wang, H., Pattarachaiyakop, N. and Trada, M. (2011). A Review on the tensile properties of natural fibre reinforced polymer composites. *Composites Part B*, 42:856–873.

- La Mantia, F.P. and Morreale, M. (2011). Green composite: A brief review. *Composites Part A*, 42:579 – 588.
- Lee S.H. and Wang, S. (2005). Biodegradable polymers/bamboo fibre biocomposite with bio-based coupling agent. *Composites Part A*, 37:80 – 91.
- Lima, A.C., Monteiro, S. N. and Satyanarayana, K. G. (2011). Recycled polyethylene composites reinforced with jute fabric from sackcloth: Part II-Impact strength evaluation. *Journal of Polymers and the Environment*, 19:957 – 965.
- Mallick, P.K. (2007). *Fibre-reinforced Composites: Materials, Manufacturing, and Design*. (3rd Ed.). Taylor and Francis Group, LLC. University of Michigan, Dearborn, Michigan, U.S.A. 1– 19.
- Mishra, V. (2014). Physical, mechanical and abrasive wear behaviour of jute fibre reinforced polymer composites. A Doctoral Dissertation submitted to Department of Mechanical Engineering, National Institute of Technology, Rourkela, India. 11 – 51.
- Mishra, V. and Srivastava, A. (2014). Epoxy/wood apple shell particulate composite with improved mechanical properties. *International Journal of Engineering Research and Applications*, 8(1):142 – 145.
- Mohan, S.K. and Srivastava, T. (2010). Microbial deterioration and degradation of polymeric materials. *Journal of Biochemical Technology*, 2(4), 210 – 215.
- Mohanty, A.K., Khan, M. A. and Hinrichsen, G. (2000). Influence of chemical surface modification on the properties of biodegradable jute fabrics/polyester amide composites. *Composites: Part A*, 31:143–150.
- Mortensen, A. (2007). *Concise encyclopaedia of Composite Materials*. 2nd Ed. Elsevier Ltd., Kidlington, Oxford, UK. 1 – 2.
- Mussig, J. (2010). *Industrial Applications of Natural fibres: Structure, Properties and Technical Applications*. John Wiley and Sons, Ltd. West Sussex. United Kingdom. 94 – 102.
- Mwaikambo, L.Y. and Ansell, M.P. (1999). The effect of chemical treatment on the properties of hemp, sisal, jute and kapok fibres for composite reinforcement. 2nd *International wood and Natural Fibre Composite Symposium*, June 28 – 29, 1999. Kassel, Germany. 81 – 83.
- Nayak, N.C. and Mishra, A. (2013). Development and mechanical characterisation of palmyra fruit Fibre reinforced epoxy composites. *Journal of Production Engineering*, 16 (2):69 – 72.
- Ngarguededjim, K., Allarabeye, N., Charlet, J.F., Destrebecq, R., Moutou P. and Robert, J.L. (2015a). Mechanical characteristics of fibre palmyra. *Global Journal of Researches in Engineering*, 15:1 – 9.

- Ngargueuedjim K., Bassa B., Nadjitonon N., Allarabeye N., Annouar D. M., Abdel-Rahim M., ... Jerome D. (2015b). Mechanical characteristics of tall-palm (*Borassus aethiopum* Mart., *arecaceae*) of Chad/Central Africa. *International Journal of Engineering and Technical Research*, 3:2454 – 2498.
- Orwa, C., Mutua, A., Kindt, R., Jamnadass R. and Anthony, S. (2009). *Agro forestree Database: A Tree Reference and Selection Guide Version 4.0*. retrieved on 15th March, 2016 from: <http://www.worldagroforestry.org/sites/treedbs/treedatabases.asp>
- Părpărită, E., Darie, R.N., Popescu, C-M., Uddin, Md. A. and Vasile, C. (2014). Structure–morphology–mechanical properties relationship of some polypropylene/lignocellulosic composites. *Materials and Design*, 56, pp. 763 – 772.
- Pascault J. and Williams, R.J.J. (2010). *Epoxy polymers: New Materials and Innovations*. Wiley Weinheim, Germany. p.11.
- Patel, R. G. and Sen, D. J. (2011). Biodegradable Polymers: an Eco friendly Approach in Newer Millennium. *Internationale Pharmaceutica Scientia*, 1(3): 29 – 44.
- Pickering, K.L. (2011). *Properties and Performance of Natural-fibre Composites*. Woodhead Publishing Limited. Cambridge, England. p. 39.
- Pickering, K.L., Aruan Efendy, M.G. and Le, T.M. (2016). A review of recent developments in natural fibre composites and their mechanical performance. *Composites, Part A*, (83):98 – 112.
- Qian, S., Mao, H., Sheng, K., Lu, J., Luo, Y. and Hou, C. (2013). Effect of low-concentration alkali solution pretreatment on the properties of bamboo particles reinforced poly lactic acid composites. *Journal of Applied Polymer Science*, 130(3):1667 – 1674.
- Ramesh, M., Palanikumar, K., Reddy, K.H. (2013). Comparative evaluation of properties of hybrid glass fibre sisal/jute reinforced epoxy composites. *Procedia Engineering*, 51, 745 – 750.
- Rassiah, K., Nagapan, S. and Mat-Jidin, R. (2012). The effect of sodium hydroxide (naoh) on water absorption and biodegradability of low density polyethylene (LDPE)/sugarcane bagasse (SCB) composites. *Canadian Journal on Mechanical Sciences and Engineering*, 3(1): 19 – 24.
- Ratna, D. (2009). *Handbook of Thermoset Resins*. Ismithers, United Kingdom. 81 – 83.
- Retting. (2016). In *Encyclopædia Britannica*. Retrieved on April 20, 2016, from *Encyclopædia Britannica*
Online: <http://www.britannica.com/EBchecked/topic/500159/retting>

- Salit, M.S. (2014). *Tropical Natural Fibres and Their Properties*. Tropical Natural Fibre Composites, Engineering Materials. Springer Science Business Media, Singapore. p 15.
- Sambou, B., Lawesson, J.E. and Barfod, A.S. (1992). Borassus aethiopicum a threatened multiple purpose palm in Senegal. *Principes*, 36(3):146 – 155.
- Sapuan, S.M., Pua, F., El-Shekeil, Y.A. and Al-Oqla, F.M. (2013). Mechanical properties of soil buried kenaf fibre reinforced thermoplastic polyurethane composites. *Materials and Design*, 50: 467 – 470.
- Sarki, J. (2012). Development and characterisation of epoxy/coconut shell particulate composite for engineering applications, Unpublished M.Sc. Thesis, Ahmadu Bello University, Zaria, p60.
- Selvaraju, S. and Ilaiyavel, S. (2011). Application of composites in marine industry. *Journal of Engineering Research and Studies*, 2:89 – 91.
- Sgriccia, N., Hawley, M.C. and Mistra, M. (2008). Characterisation of natural fibre surfaces and natural fibre surfaces and natural composites. *Composites, Part A*, 39:1632 – 637.
- Shah, A.A., Hassan, F., Hameed, A. and Ahmed, S. (2008). Biological degradation of plastics: A comprehensive review. *Biotechnology Advances*, 26:246–265.
- Shanmugam, D. and Thiruchitrabalam, M. (2013). Static and dynamic mechanical properties of alkali treated unidirectional continuous palmyra palm leaf stalk fibre/jute fibre reinforced hybrid polyester composites. *Materials and Design*, 50:533 – 542.
- Sharma, H.S.S. (1988). Chemical retting of flax using chelating compounds. *Annals of Applied Biology*, 113:159–165.
- Shehu, U. (2015). Development and characterisation of poly (lactic acid) (PLA)/guinea corn (sorghum bicolor) husks particulate (GHP) composites, Unpublished Doctoral Thesis, Ahmadu Bello University, Zaria. 14 – 33.
- Shukor, F., Hassan, A., Islam, M.S., Mokhtar, M. and Hassan, M. (2013). Effect of ammonium polyphosphate on flame retardancy, thermal stability and mechanical properties of alkali treated kenaf fibre fill poly lactic acid (PLA) bio-composites. *Materials and Design*, 52: 425 – 429.
- Shupe, T., Lebow, S. and Ring, D. (2016). *Causes and Control of Wood Decay, Degradation and Stain*. Retrieved on April 11, 2016, from: www.lsuagcenter.com
- Singh, A., Singh, S. and Kumar, A. (2013). Study of mechanical properties and absorption behavior of coconut shell powder-epoxy composites. *International Journal of Material Science and Applications*. 2(5):157 – 161.

- Singha, A.S. and Rana, A.K. (2012). Effect of aminopropyltriethoxysilane (APS) treatment on properties of mercerized lignocellulosic *Grewia optiva* fibre. *Journal of Polymer and the Environment*, 21(1):1 – 10.
- Speri, M. (2011). Insights on microbial and biochemical aspects of retting for bast fibre plant processing in a bioreactor. A Doctoral dissertation. University of Advanced Studies, Verona, Italy, p 21.
- Sreekala, M.S., Kumaran, M.G. and Thomas, S. (2002). Water sorption in oil palm reinforced phenol formaldehyde composites. *Composites, Part A*, (33):763 – 777.
- Sreekala, M.S. and Thomas, S. (2003). Effect of fibre surface modification on water-sorption characteristics of oil palm fibres. *Composite Science and Technology*, 63:861 – 869.
- Sreekumar, P.A., Thomas, S.P., Saiter, J.M., Joseph, K., Unnikrishnan, G. and Thomas, S. (2009). Effect of fibre surface modification on the mechanical and water absorption characteristics of sisal/polyester composites fabricated by resin transfer molding. *Composites: Part A*, 40: 1777–1784.
- Surata, W., Suriadi, G.A.K. and Arnis, K. (2014). Mechanical properties of rice husk fibre reinforced polyester composites. *International Journal of Materials, Mechanics and Manufacturing*, 2(2):165 – 168.
- Suresha, B., Ravishankar, B. L. and Sukanya, L. (2013). Dynamic mechanical analysis and three-body abrasive wear behavior of epoxy nanocomposites. *Journal of Reinforced Plastics and Composites*, 32:61–71.
- Tawakkal, M.A., Talib, R.A., Abdan, K. and Ling, C.N. (2012). Mechanical and physical properties of kenaf derived cellulose (KDC)-filled polylactic acid (PLA) composites. *Bioresources*. Universiti Putra, Selangor, Malaysia. 1648 – 1655.
- Thakur, V.K. and Thakur, M.K. (2014). Processing and characterisation of natural cellulose fibre/thermoset polymer composites. *Carbohydrate Polymers*, 109:102 – 117.
- Thomas, S., Jacob, M. and Varughese, K.T. (2004). Mechanical properties of sisal/oil palm hybrid fibre reinforced natural rubber composites. *Composite Science and Technology*, 64:955 – 965.
- Tong, J.Y., Royan, N.R.R., Ng, Y.C., Ab Ghani, M.H. and Ahmad, S. (2014). Study of the mechanical and morphological properties of recycled HDPE composite using rice husk filler. *Advances in Materials Science and Engineering*, 1:1 – 6.
- Tserki, V., Matzinos, P. and Panayiotou, C. (2006a). Novel biodegradable composites based on treated lignocellulosic waste flour as filler Part II. Development of biodegradable composites using treated and compatibilized waste flour. *Composites, Part A*, 37:1231–1238.

- Tserki, V., Matzinos, P., Zafeiropoulos, N. E. and Panayiotou, C. (2006b). Development of Biodegradable Composites with Treated and Compatibilized Lignocellulosic Fibres. *Journal of Applied Polymer Science*, 100:4703–4710.
- Venkatesh, R.P., Ramanathan, K. and Raman, V.S. (2016). Tensile, flexural, impact and water absorption properties of natural fibre reinforced polyester hybrid composites. *Fibres and Textile in Eastern Europe*, 24, 1(115):90 – 94.
- Verma, D., Gope, P.C., Shandilya, A., Gupta, A. and Maheshwari, M.K. (2013). Coir fibre reinforcement and application in polymer composites: A review, *Journal of Materials and Environmental Science*, 4:263 – 276.
- Wambua, P., Ivens, J. and Verpoest, I. (2003). Natural fibres: can they replace glass in fibre reinforced plastics? *Composites Science and Technology*, 63: 1259–1264.
- Yussuf, A.A., Massoumi, I. and Hassan, A. (2010). Comparison of polylactic acid/kenaf and polylactic acid/rice husk composites: The influence of the natural fibres on the mechanical, thermal and biodegradability properties. *Journal of Polymer Environment*, 18:422 – 429.
- Zafeiropoulos, N.E. (2011). *Interface Engineering of Natural Fibre Composites for Maximum Performance*. Woodhead Publishing Limited, Cambridge, UK. p13.
- Zhu, J., Zhu, H., Abhyankar, H. and Njuguna, J. (2013). Effect of fibre treatments on water absorption and tensile properties of flax/tannin composites. A Conference Paper Presentment at the 19th International Conference on Composite Materials, Montreal, Canada.
- Zuluaga, R., Putaux, J. L., Cruz, J. Vélez, J., Mondragon, I. and Ganan, P. (2009). Cellulose microfibrils from banana rachis: Effect of alkaline treatments on structural and morphological features. *Carbohydrate Polymers*, 76:51 – 59.

Appendix A: Single Fibre Strength and Diameter

Table A1: Average Single Fibre Strength at Fracture and Average Fibre Diameter

Untreated Fibre Strength, σ_f (GPa)	Average Diameter of Untreated Fibre (μm)	Alkaline treated Fibre Strength, σ_f (GPa)	Average Diameter of Alkaline Treated Fibre (μm)	Latex treated Fibre Strength, σ_f (GPa)	Average Diameter of Latex Treated Fibre (μm)
1.25	350	1.04	140	0.82	150

Appendix B: Density Values of the Composite Formulations

Table B1: Density of Composites

Fibre Content (wt. %)	Densities (g/cm^3)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	1.1000			
5		1.0381	1.0498	1.0786
10		0.9621	1.0268	1.0642
15		0.9363	0.9542	1.0241
20		0.8800	0.9311	0.9388
25		0.7967	0.8827	0.9192

AppendixC: Water Absorption Test after 30 Days

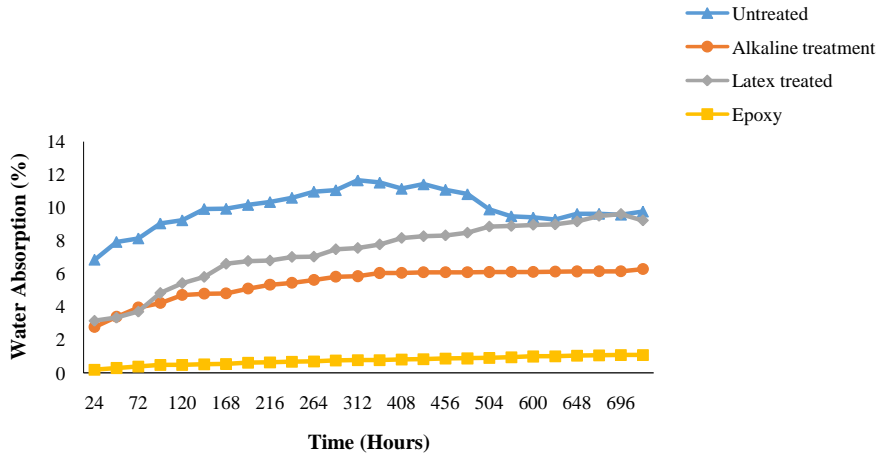


Figure C1: Water absorption of unreinforced matrix and 10wt.% reinforced composites in distilled water

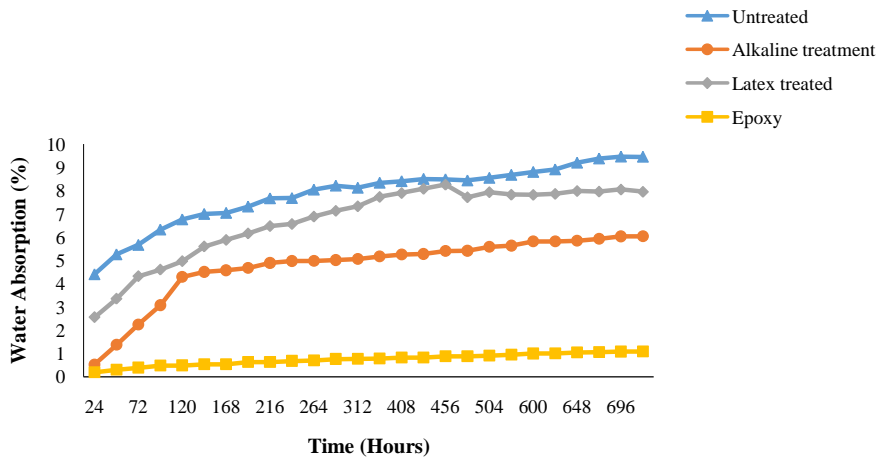


Figure C2: Water absorption of unreinforced matrix and 15wt.% reinforced composites in distilled water

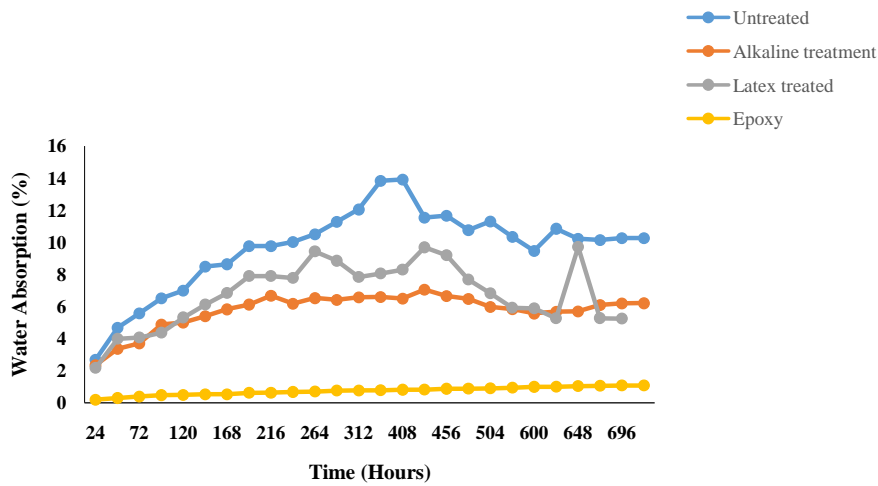
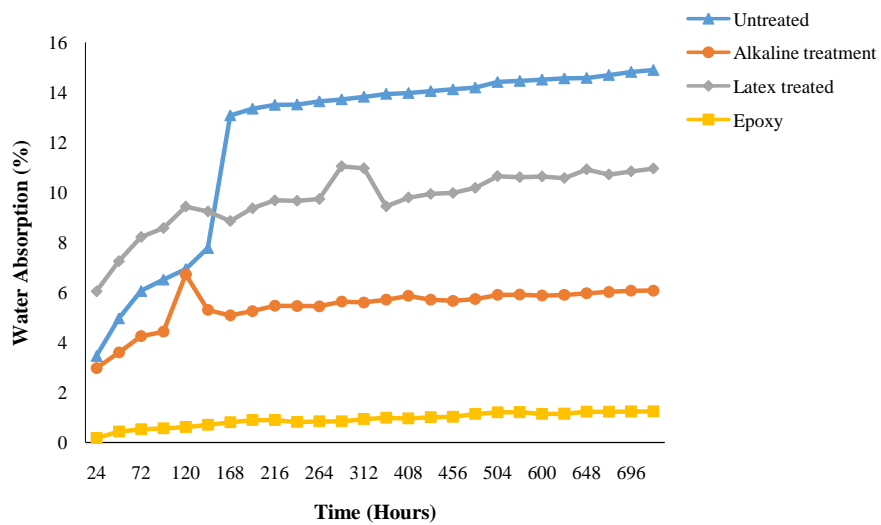


Figure C3: Water absorption of unreinforced matrix and 20wt.% reinforced composites in



distilled water

Figure C4: Water absorption of unreinforced matrix and 10wt.% reinforced composites in

river water

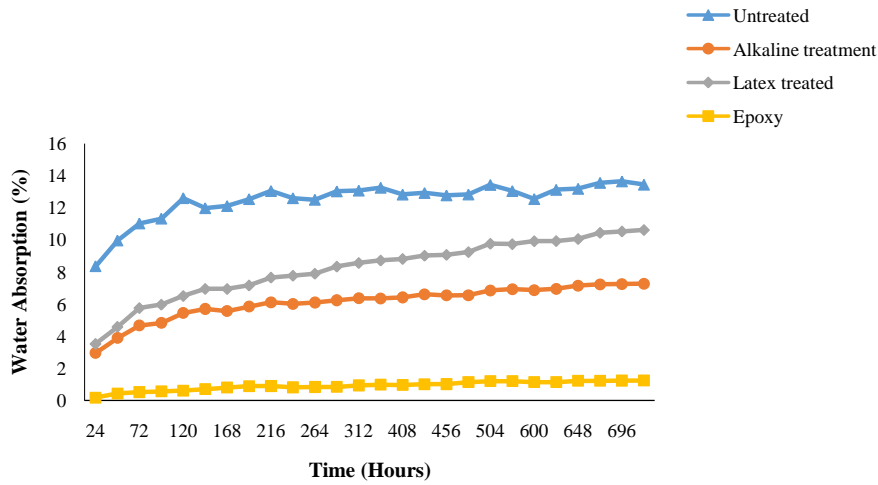


Figure C5: Water absorption of unreinforced matrix and 15wt.% reinforced composites in river water

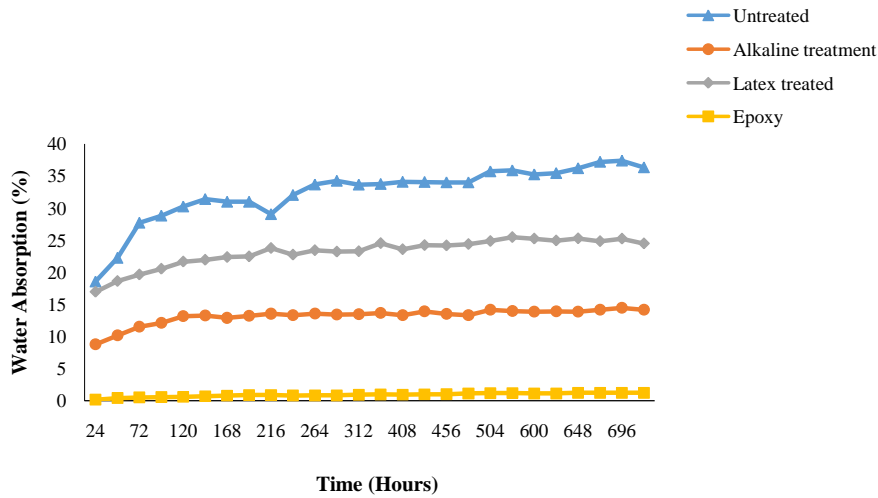


Figure C6: Water absorption of unreinforced matrix and 20wt.% reinforced composites in river water

Appendix D: Mechanical Properties of Composites

Table D1: Tensile Strength of Composites

Fibre Content (wt. %)	Tensile Strength (MPa)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	23.68567	-	-	-
5	-	33.77198	36.11696	9.79359
10	-	42.70707	36.60406	24.12278
15	-	42.80335	40.21187	42.80335
20	-	48.44133	41.28731	43.28921
25	-	53.58842	55.74935	50.47670

Table D2: Tensile Modulus of Composites

Fibre Content (wt. %)	Tensile Modulus (MPa)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	256.11143	-	-	-
5	-	1166.68425	717.36965	1082.37908
10	-	1199.19930	891.14189	1220.01057
15	-	1256.81391	1043.26315	1224.79210
20	-	1351.68524	1066.95356	1391.41075
25	-	1363.79175	1258.58173	1431.71673

Table D3: Flexural Strength of Composites

Fibre Content (wt %)	Flexural Strength (MPa)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	49.93321	-	-	-
5	-	43.51299	26.16727	28.82321
10	-	49.42906	43.75288	42.92349
15	-	54.15352	47.80418	65.22737
20	-	56.77944	59.97584	71.43063
25	-	58.91179	70.71840	72.34649

Table D4: Flexural Modulus of Composites

Fibre Content (wt %)	Flexural Modulus (MPa)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	1149.57514	-	-	-
5	-	807.39861	691.52250	1195.02525
10	-	1222.75977	1047.27192	1243.89257
15	-	1249.55072	1101.33133	1308.10139
20	-	1445.73584	1244.62337	1328.89442
25	-	1574.80755	1317.83314	1598.03696

Table D5: Impact Strength of Composites

Fibre Content (wt %)	Impact Strength (KJ/m ²)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	75.00	-	-	-
5	-	21.25	16.75	11.50
10	-	37.25	21.25	17.75
15	-	61.75	39.00	26.75
20	-	66.75	48.5	38.50
25	-	111.50	92.25	80.25

Table D6: Hardness of Composites

Fibre Content (wt %)	Hardness (HV)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	17.50	-	-	-
5	-	20.23	22.63	26.77
10	-	23.80	26.90	29.03
15	-	25.40	31.2	32.47
20	-	27.53	35.6	36.87
25	-	30.27	36.47	42.77

Appendix E: Degradability Test Data

Table E1: Percentage Weight Loss of Composites buried for 90 Days in Dry Season

Fibre Content (wt. %)	% Weight Loss (Soil Burial)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	0.3419	-	-	-
5	-	0.3639	1.1706	0.8774
10	-	0.5009	1.2786	0.6748
15	-	0.0400	1.6401	1.3488
20	-	0.1268	0.9577	0.1852
25	-	1.0495	1.5985	0.0613

Table E2: Percentage Weight Loss of Composites Exposed to Natural Weather for 90 Days

Fibre Content (wt. %)	% Weight Loss (Natural Weather Exposure)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	0.7232	-	-	-
5	-	1.4409	1.3254	1.3299
10	-	1.7168	1.3287	1.4267
15	-	1.9733	1.7040	2.2299
20	-	1.9753	1.4838	1.9697
25	-	1.9774	2.7215	2.0547

Table E3: Percentage Weight Loss of Composites Buried for 90 Days in Rainy Season

Fibre Content (wt. %)	% Weight Loss (Soil Burial)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	0.3666	-	-	-
5	-	0.4692	0.7185	0.2393
10	-	0.8830	0.5327	0.5228
15	-	1.2083	0.1336	1.6511
20	-	1.1877	0.9522	1.0270
25	-	0.7091	0.5228	0.9530

Table E4: Percentage Weight Gain of Composites Exposed to Natural Weather for 90 Days in Rainy Season

Fibre Content (wt. %)	% Weight Gain (Natural Weather Exposure)			
	Epoxy	Untreated	Alkaline treated	Latex treated
0	0.8562	-	-	-
5	-	1.9770	1.4801	1.3442
10	-	2.2672	3.0127	1.8972
15	-	2.8912	2.8963	1.8676
20	-	3.1949	3.9885	2.4507
25	-	3.3355	2.7467	2.8261