STUDY OF THE EFFECTS OF ALKALINE TREATED OKRO BAST FIBRES AS FILLERS IN UNSATURATED POLYESTER RESIN COMPOSITES

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JANUARY, 2017

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DEPARTMENT OF POLYMER AND TEXTILE SCIENCE,

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AHMADU BELLO UNIVERSITY, ZARIA NIGERIA.

JANUARY, 2017

DECLARATION

I declare that the work in this dissertation entitled **"Study Of The Effects Of Alkaline Treated Okro Bast Fibres As Fillers In Unsaturated Polyester Resin Composites"** has been carried out by me in the Department of Polymer and Textile Science. The information derived from the literature has been duly acknowledged in the text and a list of references provided. To the best of my knowledge, no part of this dissertation was previously presented for another degree at this or any other Institution.

Idris Hindatu Umar

Name of Student Signature

Date

CERTIFICATION

This dissertation entitled **"Study Of The Effects Of Alkaline Treated Okro Bast Fibres As Fillers In Unsaturated Polyester Resin Composites ,** by Idris Hindatu Umar with registration number p13ScTX8005 meets the regulations governing the award of the degree of Master of Science (M.Sc) in Polymer and Textile Science of the Ahmadu Bello University, Zaria,Nigeria

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DEDICATION

This project work is dedicated to my beloved Mum, Hajia Rukaiyya Umar and Dad Alhaji Idris Umar.

ABSTRACT

The study deals with evaluation of properties as well as the morphological analysis of short Okro bast fibre /unsaturated polyester (UPR) composites. The Okro bast fibres were modified using sodium hydroxide at varying concentrations (5%, 10% and 15% NaOH); the fibres were neutralized with 1% acetic acid and finally rinsed with distilled water. The composites were also prepared by hand lay technique adding different weight proportions of Okro fibres (5%, %10%and 15%). The prepared composites where tested for properties in accordance with ASTM methods. The treated Okro bast fibre /unsaturated polyester resin showed moderate improvement in the mechanical properties over the unmodified composites such as tensile strength 22.21 Mpa ,tensile modulus of 287.36 Mpa and percentage elongation (17%) were obtained for 15% alkaline treated okro fibre /unsaturated polyester resin composites at 15% fibre loading, however the control sample has the observed values of 20.83Mpa, 259Mpa and 7.5% respectively. The highest impact strength and hardness were also observed at 15% NaOH treatment 0.65J/m and hardness value of 68.4HRF. The composites shows increase in water absorption with increasing filler loading, and increase in sodium hydroxide concentration with increasing number of days until it attain a saturation period where no water uptake was observed. The result shows that the highest water absorption was observed at 15% alkali treatment and 15% filler loading of Okro bast fibre/unsaturated polyester resin Composites .It was also observed that the density of the composites decreases with increase in filler loading .Scanning electron microscope revealed better adhesion between the treated okro bast fibre and the matrix. However, pullout micro cracks and voids were observed for untreated okro bast fibre/unsaturated polyester resin composites. This composite can add value to Okro bast fibre in composites field and can be used for manufacturing of structural materials for indoor use such as ceiling boards, particle board, partition board and Packaging.

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ABBREVIATIONS

CMC	Ceramic Matrix Composite
SSMA	Maleic Acid
MEKP	Methyl Ethyl Ketone Peroxide
MMC	Metal Metrix Complex
Mpa	Mega Pascal
NFC	Natural Fibre Composite
NaOH	Sodium Hydroxide
OBF	Okro Bast Fibre
PG	Polyethylene Glycol
TOBF	Treated Okro Bast Fibre
UOBF	Untreated Okro Bast Fiber
UPR	Unsaturated Polyester Resin
FL	Filler Loading

CHAPTER ONE

1.0 INTRODUCTION

Natural fibre reinforced polymer composites (NFRPC or simply NFC), have become valuable alternative material for a wide range of applications. Fibres such as jute, hemp, sisal, palm, kenaf, and flax are utilized as fillers or reinforcing material for polymer-based matrices. Such utilization of natural fibres can transform waste materials to wealth (Kalia *et al.*, 2011). The materials are attractive from environmental point of view where they can be used as an alternative to the traditional glass/carbon polymer composites (Faruk *et al.*, 2012). They can also be used in different applications such as packaging, disposable accessories, furniture, building, insulation, and automotive industries (Al-Oqla and Sapuan 2014). Moreover, these NFC have several advantages over the traditional types of materials like the low costs and density as well as acceptable specific strength and modulus which can lead to low weight products.

Furthermore, natural fibre composites are acceptable from environmental points of view because they can participate in producing recyclable and biodegradable products. NFC is much cheaper, and possess better as thermal as well as acoustic insulating properties that can widen their industrial applications (Alves *et al*, 2010).

Natural fibres have several other advantages over the traditional glass fibres such as: availability, CO_2 sequestration enhanced energy recovery, reduced tool wear in machining, dermal and respiratory irritation usage. (Al-Oqla and Sapuan, 2014; Kalia *et al.*, 2011).

The production of natural fibres continues to be a major agricultural activity world-wide. The textile and paper industries are the primary converters of fibres into the

numerous products needed in our modern society (Raymond and Herman, 1988). Fibres and fibrous products should possess some primary properties for acceptance as suitable raw materials. These properties include length to diameter ratio (aspect ratio), tenacity (strength) and flexibility. Acceptable extensibility for processing, cohesion and uniformity of properties is also important (Modibbo, *et al*, 2009). Fibres used for making undergarments and garments are generally known as comfort fibres (Sharma, 2006). Also, fibres used for making carpets, seat covers, curtains are known as safety fibres. Industrial fibres are used as reinforcing agents and as such they possess high modulus strength, thermal stability, toughness and durability. They are used for making rigid and flexible tubes, pipes, tyre cords and composites, construction of boats, cars and planes (Bhatnagar, 2004).

It has become necessary to consider affordable, sustainable alternative local raw materials of plant bast fibres for industrial growth. This is because; recent studies have reported that, natural fibres from plants, animals and minerals, account for more than half (about 65%) the fibres produced annually in the world (Khurmi and Sedha, 2005). Out of this percentage, a larger portion (46%) is cotton. The other 35% of annually produced fibre is from animal fibres (wool and silk) 5%, and artificial fibres (cellulose and synthetic fibres) provide 30% of the world's production (Alexander *et al.*, 2002). Cotton, the most widely used natural fibre, is processed into numerous apparel, home furnishings and industrial products. Other types of natural fibres include: flax, a strong, silky fibre from the stems of flax plants, used in making clothing, napkins and other linen products: Hemp, jute and sisal are coarse fibres used in cords, ropes and rough fabrics (Khusk, 1993).

Animal fibres include fur and hair, wool; the hair Sheared from sheep and certain other animals are popular in clothing and home furnishings. Rough surfaces on wool fibres give bulk and warms to wool clothing and blankets. Silk is the strongest natural fibre and manufacturers unwind silk filaments from silkworm cocoons and make silk yarn for clothing and household fabrics (Saha and Sarkar, 1990).

Natural fibre being eco-friendly, light weight strong and low cost can be embedded in the polymeric matrices to reinforce and achieve desired properties such as impact strength, stiffness, low density sound damping and texture together with eco-friendly characteristics in composites. Most bast fibres are characterized by fines and flexibility, and they are quite strong and are widely used in the manufacture of ropes and twines bagging materials and heavy-duty industrial fibres. (Ajayi, *et al.,* 2000).

Present work focuses on the preparing composites that are made of natural bast fibres due their characteristics such as light weight, strength and less expensive than that of synthetic fibre. This bast fibre yield better mechanical property when added to either thermoset or thermoplastic resin

1.2 Statement of the Research Problem.

Most composites materials are mostly based on synthetic fibres which are expensive and heavy in nature when compared to natural fibre. More so, they are not replenish-able and ecofriendly. In quest to produce composites which are ecofriendly, light weight and less expensive, this research prepare and characterize composites of okro bast fibre /unsaturated polyester resin. It is intended to produce environmental friendly composites, which will add value to okro fibre that has little popularity in composites world despites its abundance and mostly end up as waste.

1.3 Aim and Objectives

Aim

To produce and characterize composites using okro bast fibres /unsaturated polyester resin at varying NaOH concentrations and filler loadings. However this work specifically intend to:

1 Extract fibres from the bark of Okro plant and prepare the bast fibres by retting processes.

2 Treat fibres with different concentration of sodium hydroxide (NaOH) (5, 10 and 15 %).

3 Prepare composites using treated and untreated Okro bast fibres at varying filler loading of 5%, 10%, and 15%.

4 Determine morphological, physical, and mechanical properties of the composites

1.4 Justification

The research serves as a means of obtaining lightweight materials with improved mechanical properties and optimal performance. It will also encourage the development of more ecofriendly materials to address environmental challenges posed by synthetic fibre composites and the need to contribute to existing data on properties of okro bast fibre unsaturated polyester resin composites. It will also generate more income to the farmers

1.5 Scope of the Study

The scopes of the study are stated below:

I. The Okro fibre extraction was done using water retting method;

II. Fibres were modified with sodium hydroxide at various concentrations.

III. Unsaturated polyester resin (UPR) was used as a polymer matrix for the preparation the composites

CHAPTER TWO

2.0 LITRATURE REVIEW

2.1 BAST FIBRES

Plant fibres are made up of mainly cellulose, they are categorized as “natural cellulosic fibres”, which may consist of one plant cell or an aggregate of cells cemented together by non-cellulose materials. Thus, a cellulosic fibre can be either unicellular or multi cellular which is covered externally by a delicate skin or cuticle, which is sometimes called 'bast'. The upper most layer of a plant body is called bark, which is composed of a series of layers. The structures of the bark layers are composed of tough, elongated cells and vessels, called fibre or bast (Royle, 1983).

Bast fibres are obtained from the stalks of dicotyledonous plants. The fibres occur in that portion of the fibro vascular area, generally termed the phloem, Located around the woody, central portion and just under the outer bark or cuticle of the stalk. The so called "true" bast fibres occur in bundles i.e. aggregates of sclerenchymatous (thick-walled) cells, with the ends overlapping so as to produce continuous filaments throughout the length of the stalk. The fibres are usually very long and are relatively strong. For this reason, the bast fibre is considered to be the most important fraction of any plant like wood, jute, hemp, flax, and kenaf (Paridah *et al.*, 2009).

Jute, for example, has 5 to 15 cells, which may be reduced upon storing or processing. Because of this characteristic, fibres that are separated from bast plants are often referred to as “crude fibre” (aggregates of single fibres bound together), which are usually much coarser and much longer, whilst those reported in many studies are defined based on scanning electron micrographs of micro fibrils or single strand fibre.

Hence the reported of average fibre lengths and widths reported are much smaller respectively, for example 2.5 mm and 18 μm for jute, and 2.3 mm and 16.1 μm for kenaf (Paridah *et al.*, 2009).

In some plants the fibres are interlaced through the length of the stalk. The bast bundles of fibres are held together by the cellular tissue of the phloem and also by the gummy and waxy substances inside the fibre. The function of the bast bundles is to impart strength to the stalk of the plant. Bast fibres are also termed as 'soft fibres' the principal fibres in this group are jute, flax, ramie, hemp. Some bast fibres however are brittle, harsh and stiff. With a few exceptions, almost all important bast fibres have been utilized commercially in the form of full-length bast bundles. The most important exceptions are flax and ramie fibres which are separated during spinning process for the fine yarns (Montgomary, 1954).

Schneider and Karmaker (1996), developed composites using jute and kenaf bast fibres and polypropylene resins. They reported that jute fibre provides better mechanical properties than kenaf fibre. Sreekala *et al, 1997* performed one of the pioneering studies on the mechanical performance of treated oil palm fibre-reinforced composites; they also studied the tensile stress-stain behaviour of composites having 40% by weight fibre loading. Isocyanine, silane, acrylated, latex coated and peroxide-treated composite withstood tensile stress to higher strain level. Isocyanate treated, silane treated, acrylated, acetylated and latex coated composites showed yielding and high extensibility. Tensile modulus of the composites at 2% elongation showed slight enhancement upon mercerization and permanganate treatment. The elongation at break of the composites with chemically modified fibres was attributed to the changes in the chemical structure and bond ability of the fibre.

Mohanty *et al*, (2001) studied the influence of different surface modifications of jute bast fibre on the performance of the bio composites. More than a 40% improvement in the tensile strength occurred as a result of reinforcement with alkali treated jute. Jute fibre content also affected the bio composite performance and about 30% by weight of jute showed optimum properties of the bio composites. Arrifulzaman *et al.,* (2014), in their study observed that as the filler content of OBF increases, tensile strength, young's modulus and flexural strength of okro bast fibre/phenol formaldehyde resin (OBF/PFR) composites increases. Results suggested that the appropriate percentage of OBF in composite is 29 wt%, but a larger amount of OBF would decrease the tensile strength and flexural strength of OBF/PFR composites.

However, the presence of hydroxyl groups in OBF also increases water absorption and result in poor compatibility between the OBF and the hydrophobic phenol formaldehyde resin (PFR), and found that grafted hydrophobic character of OBF increases compatibility with phenol formaldehyde resin (PFR). Alkali treatment and bleached OBF are well distributed in PFR and gave higher mechanical properties of the prepared composites.

2.1.1 Advantages And Disadvantages Of Bast Fibres

The use of plant fibres has a number of advantages. First, it comes from a renewable resource. Much of the low grade fibre (6 million tonnes and more) is wasted in the present agricultural system and some fertile land might profitably be turned over to the production of high grade fibre (James, 1990). Plant fibres have characteristic properties like agro-renewability, durability, eco-friendliness, biodegradability, appreciable tensile strength, antistatic property, low-thermal conductivity, moderate moisture regain, good insulation and affinity for various classes of cellulosic dyes and compatibility in

blending with other allied natural and man-made fibres (Chakravarty, 1977; Bagchi and Suba, 1999).

Advantages of bast fibre over the traditional reinforcing fibres such as glass and carbon include low cost, low density, high toughness, acceptable specific strength properties, improved energy recovery, carbon dioxide sequestration, and biodegradability. With the increasing consciousness of preserving the environment and the need to recycle, there has been renewed interest in composite sectors using natural fibres as partial replacement for synthetic carbon, glass, or aramid fibres. Long fibres offer greater flexibility for enhancement processes, particularly in the woven and pultrusion composite industries (Pharidah and Khalina 2011).

According to Sur (2005) any textile fibre should be made up of long-chain molecules so as to ensure continuity and strength along the length of the fibre axis. The homogeneity of this long fibre depends very much on the technique of producing the fibre bundles, which is known as the retting or degumming process.

The success in use of bast fibres rest on largely to environmental friendly characteristics. Some of the bast fibres are comparable or superior to synthetic fibres in physical and chemical characteristics. Bast fibres are biodegradable and products made from them are easily disposed off without causing environmental hazard. By rotating with other crops, some bast fibres improve soil fertility and increase the productivity of other crops (Chakravarty, 1977). The use of bast fibre as a geo -textiles will help to solve to certain extent, two biggest environmental problems which we are facing today - deforestation and soil erosion (Paul,*et al.,* 1976).

2.1.2 USES OF BAST FIBRES

Bast fibres are produced and used to manufacture a wide range of traditional and novel products; which include textiles, ropes and nets, carpets and mats, brushes, and mattresses, in addition to paper and board materials. They are used in the form of fine powder as in sawdust, short fibres as in random and non-woven mat, or even long fibres as in woven mat and for making different types of bio composite products. Some composites made from natural fibres have useable structural properties at relatively low cost. (Mohanty *et al*., 2001).

2.1.3 COMMON BAST FIBRES

Common bast fibres include; jute, hemp, kenaf, and flax.

2.1.4 Hemp

Is the early source of bast fibre, it produce a strong and durable fibre (kymalainen 2004) it also prefer a mild climate ,humid atmosphere and rain fall it also require good soil moisture for seed germination and plant to grow. The world hemp market is dominated by china and South Korea.

2.1.5 Jute

The fibres are extracted from the ribbon of the stem. When harvested the plants are cut near the ground with a sickle shaped knife. The short fibres length, 5 mm, are obtained by successively retting in water, beating, stripping the fibre from the core and drying. A single jute fibre is a three dimensional composite composed mainly of cellulose, hemicelluloses, and lignin with minor amounts of protein, extractives and in organics. These fibres were designed, after millions of years of evolution, to perform in nature in a wet environment. Nature is programmed to recycle jute, in a timely way, back to

basic building blocks of carbon dioxide, and water through biological, thermal, aqueous, photochemica1, chemical, and mechanical degradations. (Tara, *et al.,* 2011)

2.1.6 Flax

Flax can be grown and harvested within three month under reasonable moisture and relatively cool temperature. Flax has also been considered as a source of linen, providing high quality fabrics in textile industries. Advantages of flax fibre is its ability to absorb up to 12% of its own weight in water and its strength increases by 20% when wet ,its dries quickly and it is anti static.(Pharida *et al.,* 2011)

2.1.7 Kenaf Fibre

Kenaf (*Hibiscus cannabinus L.*) is a member of the Malvaceae family, which includes cotton and okro. It is a herbaceous annual non-wood fibre plant whose origin was in east-central Africa. The crop, which closely resembles jute, has been considered as a potential substitute for jute in the manufacture of cordage products in the 1940s. Kenaf grows so fast that it can be harvested in 4 months, with high dry matter yield. It can be grown under more wide-ranging conditions than jute (Shah *et al.,* 1980).

Kenaf is used in the manufacture of sacking, cordage, ropes, fishing nets, etc. During the past decades, researchers have been seeking other potential markets for kenaf to take advantage from an environmental point of view. In the pulp and paper industries, kenaf has been expected to serve as an alternate non-wood fibre because it can help in reducing the deforestation worldwide and have a favorable impact on the economies of many developing and developed countries (Kaldsor, 1992). The feasibility of producing medium-density particle board from whole stalk kenaf was researched by Webber *et al.,(*1999). Currently, commercial interest in kenaf fibres among natural non-wood fibres is growing in the fields of textiles and automobiles.

2.2 OKRO PLANT (*ABELMOSCHUS ESCULENTUS* (L.) MOENCH)

Okro (*Abelmoschus esculentus* (L.) Moench), also known as *Hibiscus esculentus* L., is a member of the mallow (*Malvaceae)* family, which includes hibiscus and cotton among other species, it can be found as a tall-growing (2m tall and has leaves 10–20 cm long and broad, with lobes ranging from 5 to 7), warm-season annual or perennial (in India and Africa) that is well suited to a wide range of soil types (Shamsulalam and Arifuzzaman, 2007).

It represents the only vegetable crop in the Malvaceae family, whose products have significant use in the food sector. In several parts of the world it is known as Okro, Quingumbo, and Lady's finger, Gombo, Gumbo, Bamia, Bhendi and Bhindi. The origin of Okro is disputable, but it seems to be native to the Abyssinian centre of origin of cultivated plants, an area that includes Ethiopia, Eritrea and the eastern part of the Anglo-Egyptian Sudan. It is currently grown throughout tropical Asia, Africa, the Caribbean and southern United States (Shamsulalam and Arifuzzaman, 2007).

2.3 Chemical Composition of Okro Plant

The chemical composition as well as the morphological microstructure of vegetable fibres is extremely complex due to the hierarchical organization and the different compounds present at various concentrations. The vegetable fibres are mainly composed of cellulose and non-cellulosic materials, such as hemicelluloses, lignin, pectin, waxes, and some water-soluble compounds. The lignin and pectin act as bonding agent (Muhanti *et al.,* 2005).

Okro plant contains 60–70% cellulose, 15–20% hemicellulose, 5–10% lignin, 3.4% pectin, 3.9% fats and waxes and 2.7% water-soluble compounds (Alan *et al.,* 2007). In

comparison with other bast fibres, okro bast fibres can also be used as reinforcement in natural composite materials.

De Rosa *et al*., (2009) in their study observed that the cross-sectional shape of okro fibre shows a polygonal shape that varies notably from irregular shape to reasonably circular. Their diameter considerably vary in the range of about 40-180μm. Each ultimate cell is roughly polygonal in shape with a central hole, or lumen like other natural plant fibres. The cell wall thickness and lumen diameter vary typically between 1 to 10μm and 0.1 to 20 μm respectively. As a consequence, the considerable difference of the diameter values of the single fibre and lumen, and their rough shape strongly affects the mechanical and dimensional properties of okro fibres. Okro bast, a multicellular was analyzed for chemical composition by (Shamsul *et al.*, 2007).

The composition of okro fibres in terms of cellulosic and non-cellulosic constituents is reported in along with the composition of other bast and leaf fibres which are commonly used as reinforcements in natural fibre composites. Okro fibres are very similar to that of other bast or leaf fibres, in which comparable thermal behavior and stability are expected.

2.4 Fibre Extraction

2.4.1 Retting Of Bast Fibre

Retting is the process of antimicrobial action on the pectin in the plant which bind the fibre to the woody core material. It is sometimes termed as degumming, which is a chemical process for removing non-cellulosic material that are attached to fibres to release individual fibres (Pharidah,*et al.*, 2011). After harvesting, the stems are usually kept either in the field or under water for 2 to 3 weeks, during which the pectinous substances that bind the fibres together with other plant tissues are softened and

degraded by micro-organisms. The quality of fibres is determined by the retting condition and duration. Apparently, there is no single method that can give optimum results in terms of retting period, fibre strength, environmental pollution, and cost. (Pharidah *et al.*, 2011).

2.4.2 Dew Retting

Dew or field retting largely relies on indigenous soil, fungi to colonise the stem and degrade the pectin and hemicellulose by releasing poly galacturunase (PGase) and xylanase (Brown et al. 1986). Farmers monitor the process closely to ensure that the bast fibres separate from the inner core without much deterioration in quality. Moisture is needed for the microbial break-down to occur, but then the weather must be dry enough for the stalks to dry for bailing. Although varying weather conditions affect the quality of fibre, field retting has been used extensively for hemp because it is inexpensive, mechanized, and does not use water. Dew retted fibres are often coarse and are of variable quality. (Tahir *et al.*, 2011)

2.4.3 Water Retting

Water retting is performed in an aqueous environment, and anaerobic, pectinolytic bacteria are responsible for the decomposition of pectin substances and the subsequent release of fibre (Akin *et al*., 2002). This process consistently yields high quality fibres, and does not change the colour of the fibres (Van Sumere, 1992). Water retting has been largely abandoned in countries where labor is expensive or environmental regulations exist. Most bast fibres currently used in textiles is water retted; duration of retting is 7-14 days Vansharma 1992, and Akin *et al.*, 2007). They produce fibres of greater uniformity and higher quality. Extensive stench and pollution arising from

anaerobic bacteria fermentation of plant, high cost and putrid odor, environmental problems and low grade fibres requires higher water treatment maintenance.

2.4.4 Chemical Retting

Chemical and enzyme retting offer substantially more control compared to dew and water retting. Paridah *et al.,* (2009) used 5% sodium hydroxide and 5% sodium benzoate during retting of kenaf bast fibre and found that both methods produced fibres of relatively lower tensile strength than those obtained with water alone. The colours of chemically-treated bast fibres were also darker. Van Sumere, (1992) reported that the bacterial method is relatively better than chemical, because it offers better fibre quality and lower pollution, whilst chemical retting requires high energy and generates costly wastes. It is more efficient and can produce clean and consistent long smooth surface bast fibres within short time. Fibres retted in more than 1% NaOH, has unfavorable color and high processing cost (Kawaha *et al.*, 2005, Mooney *et al.*, 2001).

2.4.5 Enzymatic Retting

This method is mainly achieved by the pectic enzymes produced by bacteria. During retting, the bacteria multiplies and produce extracellular pectinases, which releases the bast fibres from the surrounding cortex by dissolving the pectin. Nowadays, with the advancement of biotechnology tools, such enzymes can be commercially produced, thus making enzymatic retting a more popular choice for the production of long fibres. The duration is 12-24 hours (Vansharma, 1992 and akin *et al.*, 2007). Enzymatic retting is easier refining, particularly for pulping purpose that degrades and provides selective properties for different applications.

2.4.6 Mechanical Retting

Fibres are separated by hammer mill or decorator. It produces massive quantities of short fibres in short time. It has high cost of production and lower fibre quality (Paridah, 2009).

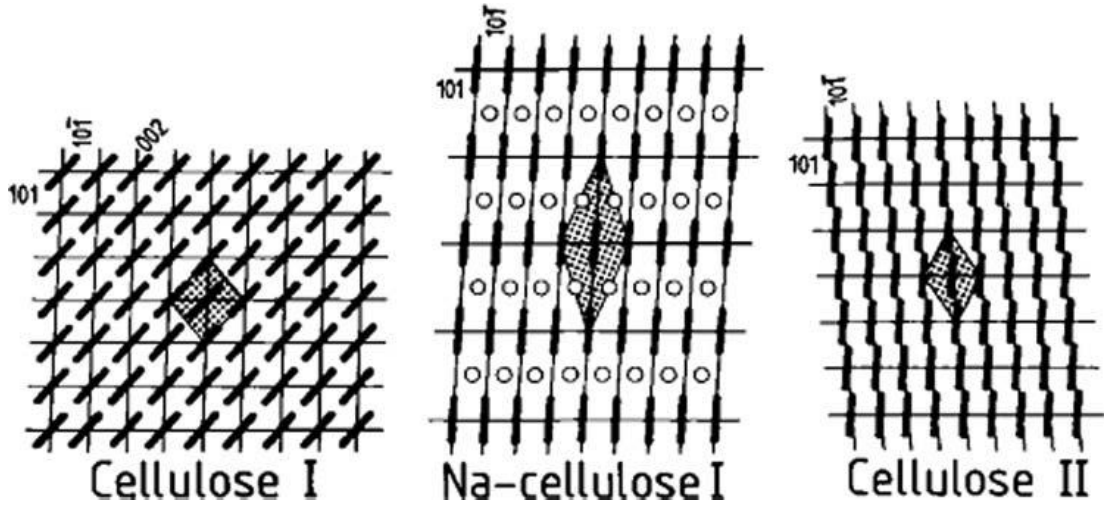
2.5 Chemical Treatments

2.5.1. Alkaline Treatment

Alkaline treatment is a common fibre surface treatment that can also be used as a pre-treatment when combined with other chemical modifications. Originally, these treatments were carried out to improve a fibres dye affinity and luster (Vendeweyanberg., *et al.,* 2006). Researchers subsequently discovered that alkalization also has a positive effect on the mechanical properties of fibre-reinforced composites, yielding significant improvements in the interfacial performance (Gonzalez, 1999).The structure of cellulose in plant fibres exhibits a monoclinic crystalline lattice of Cellulose-I that can be changed into different polymorphous forms through alkaline treatment (Mwaikambo *et al.,* 2002). The effect of alkali on a cellulose fibre is a swelling reaction, during which the natural crystalline structure of the cellulose relaxes. The type of alkali and its concentration will influence the degree of swelling and the degree of lattice transformation into Cellulose-II. Usually, sodium hydroxide (NaOH) is used because Na+ is able to widen the smallest pores in between the lattice planes and penetrate into them. After removing the excess NaOH, the new Na-Cellulose-I lattice is formed. Meanwhile, the OH groups of the cellulose are converted into ONa- groups. Rinsing with water removes the linked Na ions and converts the cellulose to a new crystalline structure: Cellulose-II, whose lattice is more stable than Cellulose-I. The total reaction is shown below.

$$\text{Fiber–OH} + \text{NaOH} \longrightarrow \text{Fiber–O–Na}^{+} + \text{H}_2\text{O}$$

After alkali treatment the surface of the lingo cellulosic fibres reveal a smoothing effect. This is partially due to the removal of the outer surface of the fibre layer through dissolution in chemical solutions during the treatment. Alkaline treatments promote the interfacial adhesion between fibres and matrices, particularly improving the mechanical adhesion**.**



A schematic which represents the crystalline lattices of Cellulose-I, Na-Cellulose-I, and Cellulose-II. (Ven de weyanberg, 2000)

However, moderate or short time alkaline treatments have been reported to significantly improve the mechanical properties, impact fatigue and dynamic behaviour of fibre-reinforced composites (Cao *et al.*, 2006). who used different concentrations of NaOH solutions for the alkaline treatment of bagasse fibres, concluded that the utilization of a 1% NaOH solution for the treatment led to the best results as to the mechanical properties of composites. The improvement was also the result of external fibrillation, which occurred during the alkaline treatment. An increased alkali concentration led to a loss of mechanical strength, due to depolymerisation. Similar results were also obtained

by a large number of authors, who used different types of bast fibres, sodium hydroxide concentrations and polymeric matrices (Mishra *et al.,* 2003 and Jacob *et al.,* 2006).

Gassan and Bledzki (1999) reported that treating the fibre surface by 26 wt% NaOH for 20 minutes at 20°C improves the mechanical properties of unidirectional jute/epoxy composite up to 60% compared to untreated fibre composite, at a fibre content of 40 %. (Prasad *et al.,* 1983) studied the alkali treatment of coir fibre by 5% NaOH for 72-96 hours at 28°C. An improvement of the tensile strength and Young’s modulus of the fibre by 10-15% and 40%, respectively, was observed. The alkali treatment of the coir fibre improved the flexural strength of the polyester resin composites by 40%. Sydenstricker *et al.,* (*2003*) obtained increasing shear strength from 2.6 to 6.9 MPa in the case of fibre surface treatment by 2% NaOH for 1 hour at room temperature of sisal/polyester composites. Ray *et al.*, (*2001*) used a solution of 5% NaOH to treat the jute fibre for 0, 2, 4, 6 and 8 hours at 30°C. For the vinyl ester resin composites reinforced by 35 wt% jute fibre treated for 4 hours, an improvement of 20% for the flexural strength, of 23% for the flexural modulus and of 19% for the laminar shear strength was observed.

2.5.2 Acetylation

Acetylation of cellulose is one of the first discovered routes to obtain cellulosic derivatives. The results of the research on vegetal fibre composite production indicated an increased mechanical strength of the resulting bio composites. Mishra *et al.,* (2003) investigated the possibilities of acetylating vegetal fibres for increasing the mechanical properties of some vegetal fibre-polyester composites, by acetylating treatment with previous mercerization (Mishra *et al.,* 2003).

Vegetal fibre – OH + CH_3 – C (=O) – O – C (=O) – CH_3 Fibre – $OCOCH_3$ + CH_3COOH

The 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 a few minutes, then filtered, washed and dried in ventilated oven. This is an esterification method which should stabilize the cell walls, especially in terms of humidity absorption and consequent dimensional variation (Mishra *et al.*, 2003).

2.5.3. Benzoylation

Benzoylation is another important transformation in organic syndesertation and vegetal fibre treatment (Paul, 2003). Benzoyl chloride is most often used in fibre treatment. Benzoyl chloride includes benzoyl ($C_6H_5C=O$), which is responsible for the reduced hydrophilic nature of the treated fibre and improved interaction with the hydrophobic polymeric matrix. Benzoylation has also been found to contribute to the increase of the mechanical properties of natural fibre-polymer composites. The increase in mechanical strength (tensile and flexural properties) has been reported for natural fibre-reinforced composites with polystyrene matrix, (Paul, 2003) polyester resins, (Joshy *et al.,* 2006) high-density polyethylene (HDPE) and low-density polyethylene (LDPE), either individually or in mixture (Wang, 2007).

2.5.4 Anhydride Treatment

It is usually carried out by utilizing maleic anhydride or maleated polypropylene (or polyethylene) in a toluene or xylene solution, where the fibres are immersed for impregnation and reaction with the hydroxyl groups on the fibre surface. Literature reports significant reduction of water absorption (Elsabbagh *et* a*l*., 2009).

2.5.4.1 Maleic Anhydride

Cis-butenedioic anhydride, toxilic anhydride, dihydro-2, 5-dioxofuran, an organic compound with the formula $C_4H_2O_3$, is, in its pure state, a colourless or white solid with acrid odour. Maleic anhydride might be used as an esterification reagent for the free hydroxyl groups present on the surface of the natural fibres. Maleic anhydride easily hydrolyses to maleic acid, which is also used as a treatment agent. The main purpose of using maleic anhydride is to assure chemical bonding between the fibre and the polymeric matrix.

Maleic acid or maleic anhydride is first reacted with the polymeric matrix, to be chemically linked to it. In a secondary stage of the process, the modified polymeric matrix is reacted with the cellulose contained in the fibre, by formation of ester linkages with the free hydroxyl groups. Positive results on the chemical bonding of polymer matrix to bast fibres by maleic acid or anhydride treatment were obtained by (Elsabbagh *et a*l., 2009) in the effort of manufacturing flax/polypropylene composites. Other results confirmed the possibility of increasing interfacial energy in the bast fibres-polymeric (other than polypropylene) composites; the mechanical properties, as well as their hydrophobic characteristics were also improved (Bessadok *et al.,* 2007).

2.5.5 Silane Treatment:

Using silane coupling agents was found to be effective in modifying the natural fibre-matrix interface Culler *et* al., (1986), Ghatge and Khisti (1989) and George *et al.,* (1998). Gassan and Bledzki (1997) investigated the influence of silane coupling agent on the performance of jute-epoxy composite. A treatment of jute fibre by epoxy functional-γ-lycidoxypropyltrimethoxy-silane (A-187 from Osi-Specialtis GmbH, Germany) solution in alcoholic with the concentration of 2% for 24 hours at 23°C was

found to increase by approximately 30% of the static characteristic values of the composites compared to unmodified composites at standard humidity.

Yan and Lin, (2005) showed the influence of fibre surface treatment on the performance of the sisal textile reinforced vinylester composites. Sisal fibre was treated with 3-aminopropyltriethoxy silane and γ-methacryloxypropyl trimethoxy silane solution in acetone with a concentration of 6% for 24 hours. Only marginal improvement of 3% and 14% of the tensile strength and tensile modulus of the composites, respectively, was obtained in the case of fibre surface treatment. The flexural strength and modulus of the silaned fibre composites increased by 15% and 30% respectively compared to untreated composites. However, the fibre surface treatment by silane did not affect the impact energy. (Mader and Gliesche, 1995) investigated epoxy composites and found out that there was a reduction in the flexural strength and an increase by about 20% in flexural modulus of the flax composites, if an aqueous γ-amino propyltriethox silane solution of 3% was used for treating the fibre surface at 80°C. That did not result, however, to any remarkable increase in flexural strength but resulted to an increase by about 20% for the flexural modulus of the ramie composites.

The fibres were immersed in a 3:2 alcohol– water solution, containing a silane-based adhesion promoter for 2 hour at pH 4, rinsed in water and oven dried. Silane should react with the hydroxyl groups of the fibres and improve their surface quality (Mader and Gliesche, 1995).

2.5.6 Isocyanate Treatment

Isocyanates, especially poly [methylene poly (phenyl isocyanate)] (PMPPIC), are known in wood chemistry as wood binders with successful application in particleboard in Europe. Extensive experimental work on the application of isocyanate as coupling agents for different types of cellulose materials and polymers has been carried out (Koma *et al.,* 1990). Composites were manufactured with cellulosic material, which was either pre-coated with an isocyanate polymer mixture, or the isocyanate was added directly into the mixture of fibre polymer. George *et al., (*1998) reported on the mechanical properties of isocyanate treated fibre-reinforced thermoplastics composites. The poly methylene (polypheny1 isocyanate) (PMPPIC) treatment has significant influence on the properties of composites, i.e., increased thermal stability, reduced water absorption etc (Joseph and Thomas, 1995, George *et al.,* 1998). PMPPIC is chemically linked to the cellulose matrix through strong covalent bonds. The -N = C = O group of PMPPIC is highly reactive with the -OH group of cellulose.

2.5.7 Graft Copolymerization

Graft polymerization is the process of obtaining a polymer containing molecules whose main backbone chain of atoms has, at various points, side chains attached to it, containing atoms or groups different from those in the main chain. The main chain may be a copolymer or it may be derived from a single monomer. Acrylic acid, acrylonitrile or styrene might be used for graft polymerization of cellulosic bast fibres. Acrylic acid treatment might be performed in non-polar solvents, in the presence of benzoyl peroxide (Digabel *et al.,* 2004 , Bessadok *et al.,* 2007). Li, (2008), improved tensile strength and lowered the water absorption capacity of flax fibre-HDPE composites after

acrylic acid treatment of the above mentioned bast fibres. Acrylonitrile is also a good candidate for graft polymerization onto the surface of the vegetal fibre:

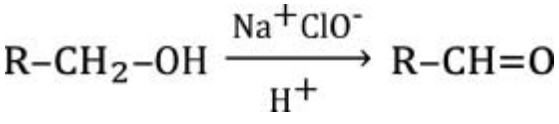
Vegetal fibre – OH + CH_2 = CHCN Vegetal fibre – OCH_2CH_2CN

Grafting is an effective method for the modification of natural fibres. Grafting on the cellulose fibre may take place before or during compounding. In the former case, pre-treatment of fibres by compatibilizing agents generally occurs in solution. The excess of (unreacted) compatibilizing agent is eliminated by washing. In the latter case, treatment during compounding occurs at the mixing temperature of the matrix. Grafting efficiency, grafting proportion and grafting frequency determine the degree of compatibility of cellulose fibres with a polymer matrix. The grafting parameters are influenced by the type and concentration of initiator, the monomer to be grafted and the reaction conditions (Digabel *et al.,* 2004, Bessadok *et al.,* 2007).

2.6. Bleaching

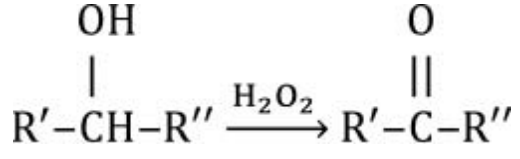
Bleaching treatments are typically used on cotton fibre and synthetic fibres to deduce the colour and increase fibre whiteness. Rosa, *et al*., (2009) the underlying principle of bleaching is the reaction of a bleaching agent with the functional groups of a fibre, which show different colors, changing the structure of the groups to cause whitening. The colour imposed by the inclusion of lignin in a fibre is due to the conjugation between aromatic rings and carbon double bonds. Since lingo cellulosic fibres will contain certain amounts of lignin and pectin, which are difficult to clean by other chemical treatments, bleaching can be used as an effective way to degrade lignin on the surface of fibre flax, as well as hydrolyze pectin's. The two major types of bleaching agents that exist are classified according to their mechanisms of chemical reaction: oxidation and reduction bleaching. Oxidation bleaching is the most commonly used

method, including bleaching using sodium hypochlorite (NaClO), hydrogen peroxide (H_2O_2), or sodium chlorite ($NaClO_2$) (Rosa, *et al.*, 2009) . Sodium hypochlorite can attack the hydroxyl groups in the lignin to form aldehyde groups (CHO), degrading the lignin is shown below.



2.6.1 Oxidation bleaching with sodium hypochlorite

(where R and R' represent the structures of lignin). However, the existing aldehyde groups by this approach can cause the cellulose to degrade, leading to detrimental performance of the fibres (Rosa, *et al.*, 2009). To alleviate this, hydrogen peroxide can be used to react with the hydroxyl groups of the lignin, forming carbonyl group (CO) which deduce the lignin as shown below



2.6.2 Oxidation bleaching with hydrogen peroxide

Where R and R represent the structures of lignin. During this bleaching process, the degradation of lignin is the highest among all oxidation methods yet the cellulose fibres will not break down. However, the cost of hydrogen peroxide is much higher than many other bleaching agents with similar capabilities. Sodium chlorite bleaching mechanisms are still controversial, and there exist several assumptions to explain the reaction. Despite this, sodium chlorite bleaching can reduce lignin and pectin with minimal amounts of cellulose degradation, at a reasonable cost compared to sodium hypochlorite and hydrogen peroxide (Rosa *et., al* 2009).

2.7 Scouring

Scouring is the process that removes all undesirable impurities; these include pectins, waxes, gums fats and oils. During this treatment, natural impurities and adventitious dirt were removed by saponification. The scoured sample appeared soft, smooth, slightly thicker and brighter. It has been established (Sadov and Korchangin, 1978) that during alkali treatment, fatty acids are converted to soap which helps to emulsify other wax like substances. Caustic soda hydrolyses protein and the molecules are broken along peptide links with the formation of alkali soluble amino acids. Similarly, pectins are hydrolyzed, gradually destroyed or decomposed to form methyl alcohol and glacturonic acid (Sadov and Korchangin, 1978). Pentoses are hydrolysed to pentosans, which in turn contain aldehyde groups. This together with the size starch decomposition impacts a reducing property to the boiling liquor.

2.8 Composites

Composites can be defined as materials that consist of two or more chemically and physically different phases separated by a distinct interface. The different systems are combined judiciously to achieve a system with more useful structural or functional properties non attainable by any of the constituent alone. Composites, materials are becoming an essential part of today's materials due to the advantages such as low weight, corrosion resistance, high fatigue strength, and faster assembly. They are extensively used as materials in making aircraft structures, electronic packaging to medical equipment, and vehicle to home building (Josmin *et al*., 2012) .The most important polymeric composites are found in nature and these are known as natural composites. Therefore connective tissues in mammals belong to the most advanced polymer composites known to mankind where the fibrous protein, collagen is the

reinforcement. It functions both as soft and hard connective tissue.(Josmin *et al.*, 2012).

Composites are combinations of materials differing in composition, where the individual constituents retain their separate identities, these separate constituents act together to give the necessary mechanical strength or stiffness to the composite part. Composite material is composed of two or more distinct phases called matrix phase and dispersed phase and having bulk properties different from those of any of the constituents. The matrix phase is the primary phase having a continuous character which is usually more ductile and less hard phase, its holds the dispersed phase and shares a load with it. Dispersed or secondary phase (reinforcing) phase is embedded in the matrix in a discontinuous form is usually stronger than the matrix, it is sometimes called reinforcing phase (Josmin *et al.*,2012).

Ironically, despite the growing familiarity with composite materials and ever-increasing range of applications, the term defines a clear definition. Loose terms like "materials composed of two or more distinctly identifiable constituents" are used to describe natural composites like timber, organic materials, like tissue surrounding the skeletal system, soil aggregates, minerals and rock.

Composites that forms heterogeneous structures which meet the requirements of specific design and function, imbued with desired properties which limit the scope for classification. However, this lapse is made up for, by the fact new types of composites are being innovated all the time, each with their own specific purpose like the filled, flake, particulate and laminar composites.

Fibres or particles embedded in matrix of another material would be the best example of modern-day composite materials, which are mostly structural. Laminates are composite material where different layers of materials give them the specific character of a composite material having a specific function to perform. Fabrics have no matrix to fall back on, but in them, fibres of different compositions combine to give them a specific character. Reinforcing materials generally withstand maximum load and serve the desirable properties. Though composite types are often distinguishable from one another, no clear determination can be really made. To facilitate definition, the accent is often shifted to the levels at which differentiation take place viz., microscopic or macroscopic. In matrix-based structural composites, the matrix serves two paramount purposes viz., binding the reinforcement phases in place and deforming to distribute the stresses among the constituent reinforcement materials under an applied force (Josmin,*et al.*,2012).

The demands on matrices are many, they may need to temperature variations, be conductors or resistors of electricity, have moisture sensitivity etc. This may offer weight advantages, ease of handling and other merits which may also become applicable depending on the purpose for which matrices are chosen. Solids that accommodate stress to incorporate other constituents provide strong bonds for the reinforcing phase are potential matrix materials. A few inorganic materials, polymers and metals have found applications as matrix materials in the designing of structural composites, with commendable success. These materials remain elastic till failure occurs and show decreased failure strain, when loaded in tension and compression. The interface is the area of contact between the reinforcement and the matrix materials. In some cases, the region is a distinct added phase. Whenever there is inter-phase, there

has to be two inter-phases between each side of the inter- phase and its adjoin constituent. Some composites provide inter-phases when surfaces dissimilar constituents interact with each other. Choice of fabrication method depends on matrix properties and the effect of matrix on properties of reinforcements. One of the prime considerations in the selection and fabrication of composites is that the constituents should be chemically inert non-reactive (Josmin *et al.*,2012).

2.8.1 Classification Of Composites

On the basis of matrix phase, composites can be classified as follows:

I. metal matrix composites (MMCs)

II. ceramic matrix composites (CMCs)

III. polymer matrix composites (PMCs) (Sundipt and Ananda,2008)

The classifications according to types of reinforcement are

I. particulate composites (composed of particles)

II. Fibrous composites(composed of fibres)

III. Laminate composites (composed of laminates). (Sundipt and Ananda,2008)

Fibrous composites can be further subdivided on the basis of natural/bio-fibre or synthetic fibre. Bio-fibre encompassing composites are referred to as bio-fibre composites. They can be again divided on the basis of matrix, that is, non-biodegradable matrix and biodegradable matrix . Bio-based composites made from natural/bio fibre and biodegradable polymers are referred to as green composites. These can be further subdivided as hybrid composites and textile composites. Hybrid composites comprise of a combination of two or more types of fibres (Josmin *et al.*, 2012).

2.8.2 Matrix Materials

Although it is undoubtedly true that the high strength of composites is largely due to the fibre reinforcement, the importance of matrix material cannot be underestimated as

it provides support for the fibres and assists the fibres in carrying the loads. It also provides stability to the composite material. Resin matrix system acts as a binding agent in a structural component in which the fibres are embedded. When too much resin is used, the part is classified as resin rich. On the other hand if there is too little resin, the part is called resin starved. A resin rich part is more susceptible to cracking due to lack of fibre support, whereas a resin starved part is weaker because of void areas and the fact that fibres are not held together and they are not well supported (Pandey, 2004).

2.8.3 Functions of a Matrix

I. In a composite material, the matrix material serves the following functions:

II. Holds the fibres together.

III. Protects the fibres from environment.

IV. Distributes the loads evenly between fibres so that all fibres are subjected to the same amount of strain.

V. Enhances transverse properties of a laminate.

VI. Improves impact and fracture resistance of a component.

VII. Helps to avoid propagation of crack growth through the fibres by providing alternate failure path along the interface between the fibres and the matrix. Carry inter laminar shear. (Pandey, 2004)

2.8.4 Properties of a Matrix

The properties of the matrix which are important for a composite structure are as follows:

i. Reduced moisture absorption.

ii. Low shrinkage.

iii. Low coefficient of thermal expansion.

iv. Good flow characteristics so that it penetrates the fibre bundles completely and eliminates voids during the compacting/curing process.

v. Reasonable strength, modulus and elongation (elongation should be greater than fibre).

vi. Must be elastic to transfer load to fibres.

vii. Strength at elevated temperature (depending on application).

viii. Low temperature capability (depending on application).

ix. Excellent chemical resistance (depending on application).

x. Should be easily process able into the final composite shape.

Dimensional stability (maintains its shape). (Pandey, 2004

The matrix causes the stress to be distributed more evenly between all fibres by causing the fibres to suffer the same strain, this stress is transmitted by shear process, which requires good bonding between fibre and matrix and also high shear strength and modulus for the matrix itself. One of the important properties of cured matrix system is its glass transition temperature (Tg) at which the matrix begins to soften and exhibits a decrease in mechanical properties. The glass transition temperature is not only an important parameter for dimensional stability of a composite part under influence of heat, but it also has effect on most of the physical properties of the matrix system at ambient temperature (Pandey 2004).

2.8.5 Physical Properties that Influence the Behaviour Of Matrix in Composites are as follows:

i. Shrinkage during cure,

ii. Modulus of elasticity,

iii. Ultimate elongation,

iv. Strength (tensile, compressive and shear), and

v. Fracture toughness. (Pandey, 2004)

2.8.6 Types of Matrix Thermoset Resin

Polyesters, epoxy and other resins in liquid form contain monomers (consisting of simple molecules), which is converted into polymers (complex cross-linked molecules) when the resin is cured. The resulting solid .is called thermosets, which is tough, hard, insoluble and infusible. The property of infusibility distinguishes thermosets from the thermoplastics. Cure and polymerization refer to the chemical reactions that solidify the resin. Curing is accomplished by heat, pressure and by addition of curing agents at room temperature.

Thermosetting materials can be further divided into two groups depending on how they react to form their network structure. For example, epoxies and polyesters react to form a network structure without formation of a volatile by-product. Phenolics react to form a volatile by-product i.e., water. The fact that some thermosets form volatile by-products means that high pressure laminating techniques must be used to prevent the formation of voids or other defects. Epoxies and polyesters can be cured at atmospheric pressures and also at ambient temperatures (Pandey, 2004). Polyester matrices have been in use for the longest period in the widest range of structures.

Polyesters cure with the addition of a catalyst (usually a peroxide) resulting in an exothermic reaction, which can be initiated at room temperature. The most widely used matrices for advanced composites have been the epoxy resins. These resins cost more than polyesters and do not have the high temperature capability of the Bismaleimides or Polyimides (Pandey ,2004).

2.8.7 Thermoplastic Resin

Thermoplastics can be repeatedly softened by heating, and hardened by cooling. Thermoplastics possess several advantages over the thermosets, one of the most important being that they do not need storing under refrigeration. They also possess improved damage tolerance, environmental resistance, fire resistance, recyclability and potential for fast processing. Primary reason for the use of thermoplastics is their cost effective processing. Increased use for thermoplastics is also due to three different reasons, viz.

i. First - Processing can be faster than that of thermoset composites since no curing reaction is required. Thermoplastic composites require only heating, shaping and cooling.
ii. Second - The properties are attractive, in particular, high delamination resistance and damage tolerance, low moisture absorption and the excellent chemical resistance of semi-crystalline polymers.
iii. Third- In the light of environmental concerns, thermoplastic composites offer other advantages also. They have low toxity since they do not contain reactive chemicals (therefore storage life is infinite).

iv. Because it is possible to re -melt and dissolve thermoplastics, their composites are also easily recycled or combined with other recycled materials in the market for molding compounds. Since the release of gases during processing and inherent brittleness is serious disadvantage of some thermoset resins, thermoplastic composites are of great interest. Thermoplastics usually require high temperature and pressure during processing and generally lack good solvent resistance. Process conditions for high performance thermoplastics are temperature in the range of 300 to 400°C (570 to 750°F) and pressure between atmospheric pressure for thermofolding process to 20 times the atmospheric pressure for high performance press forming. Due to their high strains to failure, thermoplastics are the only matrices currently available that are suited to thermo-forming and other forms of rapid manufacture.

Thermo-loading is the most straight forward thermoplastic forming technique where a straight line is heated and folded. The process is used in volume applications like aircraft floor boards. Thermofolding operations can be carried out on solid laminate materials as well as on sandwich panels (Pandey, 2004).

2.8.8. Polyester Resins

Generally polyester resins can be made by a dibasic organic acid and a dihydric alcohol. They can be classified as saturated polyester, such as polyethylene terephthalate, and unsaturated polyester. To form the network of the composite matrix, the unsaturated group or double bond needs to exist in a portion of 8 the dibasic acid. By varying the acid and alcohol, a range of polyester resins can be made. Orthophthalic polyesters are made by phthalic anhydride with either maleic anhydride

or fumaric acid. Isophthalic polyesters, however, are made from isophthalic acid or terephthalic acid.

The polyester resin is usually dissolved in monomer (styrene is the most widely used), which will copolymerize with it and contribute to the final properties of the cured resin. The addition of catalyst will cause the resin to cure (Bagherpou , 2012). The most frequently used catalyst is methyl ethyl ketone peroxide (MEKP) or benzoyl peroxide (BPO) and the amount varies from 1-2%. The catalyst will decompose in the presence of the polyester resin to form free radicals, which will attack the unsaturated groups (like C=C) to initiate the polymerization. The processing temperature and the amount of the catalyst can control the rate of polymerization, the higher temperature or the more the catalyst, the faster the reaction. After the resin turned from liquid to brittle solid, post cure at higher temperature may need to be done. The purpose of the post cure is to increase Tg of the resin by complete cross-linking. The properties of the polyester resin are affected by the type and amount of reactant, catalyst and monomers as well as the curing temperature. The higher the molecular weight of polyester and the more points of unsaturation in molecules, the higher is the strength of the cured resins (Bagherpou, 2012).

Orthophthalic polyesters are environmentally sensitive and have limited mechanical properties. They have been replaced in some applications by isophthalic polyesters due to the excellent environment resistance and improved mechanical properties of the latter. The unsaturated polyester amounts to about 75% of all polyester resins used in the world. It is produced by the condensation polymerization of dicarboxylic acids and dihydric alcohols.

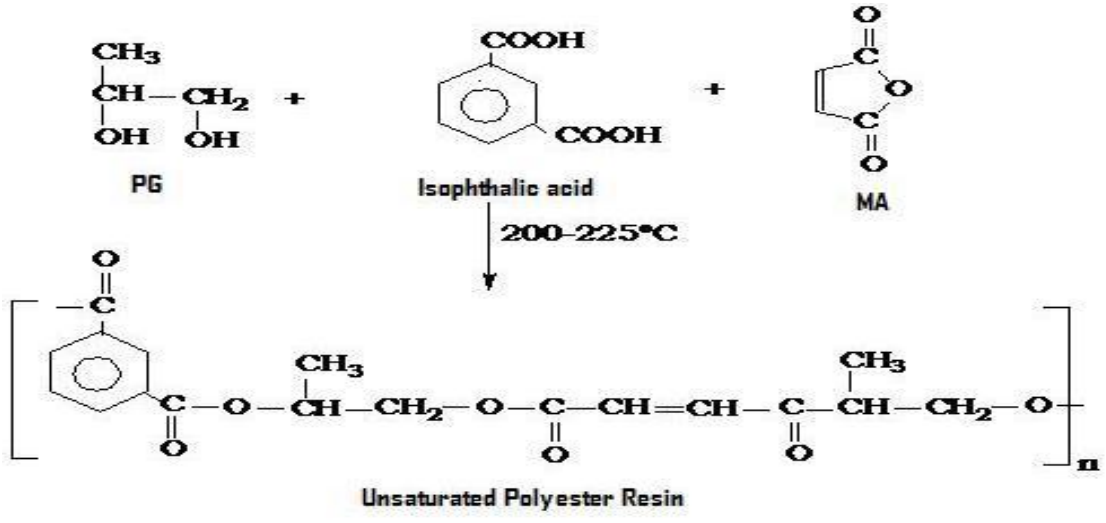
The formulation contains an unsaturated material such as maleic anhydride or fumaric acid which is a part of the dicarboxylic acid component. The formulation affects the viscosity, reactivity, resiliency and heat deflection temperature (HDT). The viscosity controls the speed and degree of wet-out (saturation) of the fibres. The reactivity affects cure time and peak exotherm (heat generation) temperatures. High exotherm is needed for a thin section curing at room temperature and low exotherm for a thick section. Resiliency or flexible grade composites have a higher elongation, lower modulus and HDT. The HDT is a short term thermal property which measures the thermal sensitivity and stability of the resins. The advantages cited in the unsaturated polyester are its dimensional stability and affordable cost. Other advantages include ease in handling, processing, and fabricating. Some of the special formulations are high corrosion resistant and fire retardants. This resin is probably the best value for a balance between performance and structural capabilities (Bagherpou , 2012).

2.8.8.1 Unsaturated Polyester Resin

Unsaturated polyester resins are the condensation products of unsaturated acids or anhydrides and diols with/without diacids. The unsaturation present in this type of polyesters provides a site for subsequent cross-linking.since 1930, unsaturated polyester resins have been used remarkably for wide range of applications making them a thermosetting system of major importance. These resins are compounded with varied fillers, reinforcements and cured by using free radical initiators to yield thermoset articles having a wide range of chemical and mechanical properties depending upon the choice of diacids, diols, cross- linking agents, initiators and other additives (Dholakiya, 2012). This versatility in the properties of the final thermoset

product associated with comparatively low cost has renewed the interest in these resins as an important matrix material for wide range of applications.

In 1929, Arvin and Carothers developed unsaturated polyester resins from maleic acid and ethylene glycol reacted at temperature 175^0-185^0C. Ford Motor Co. Ltd. synthesized unsaturated polyester resin by reacting maleic anhydride and phthalic anhydride with propylene glycol at 100^0C and then at 250^0C till the acid number diminished to the value less than 50 (mg of KOH per gm of sample). Corrado and his assistants synthesized low viscosity unsaturated polyester resins by reacting maleic anhydride, phthalic anhydride and dipropylene glycol at 200^0C. Ochsenbein and Olliver synthesized storage stable unsaturated polyester resin by reacting maleic anhydride, propylene glycol and dipropylene glycol at 185^0C (Dholakiya, 2012) under inert atmosphere. General purpose unsaturated polyester resins were prepared by using maleic anhydride, phthalic anhydride and propylene glycol with the molar ratio of phthalic anhydride: maleic anhydride ranging from 1:2 to 2:1. For thermoset products, the resultant resin was blended with styrene for cross-linking and small amount of peroxide as initiator. These types of resins are useful in making trays, shower stalls, boats, swimming pool, water tanks e.t.c. (Dholakiya, 2012).



Chemical synthesis of unsaturated polyester resin. (Dholakiya, 2012)

The reinforcement of polyesters with cellulosic fibres has been widely reported. Polyester-jute (De Albuquerque, 1999 and Sahoo *et al.,* 2007), Polyester-sisal (Pal, 1988 and Mishra *et al.*, 2002), polyester-coir (Owolabi, 1985) polyester-banana-cotton (Satyanarayana, 1983), polyester-straw (White and Ansell, 1983), polyester-pineapple leaf (Devi, 1997), and polyester- cotton-kapok (Mwaikambo and Bisanda, 1999), are some of the promising systems.

The possibility of using date palm fibres as reinforcement in polyester composites was investigated (Al-Kaabi *et al.,* 2005). Flexural properties and impact strength of date palm fibre/polyester composites were found to be influenced by fibre content and fibre treatment method. Soda treated fibres exhibited higher mechanical properties compared to untreated fibre/polyester composites. The fibre fraction and fibre length for this system were optimized for 9 wt% and 2 cm, respectively. Water absorption was lightly affected by surface modification of the fibres and was relatively low. The hybrid effect on the mechanical properties of abaca and sisal fibre-reinforced polyester composites was evaluated (Idicula, *et al.,* 2005a and 2005b). A positive hybrid effect was observed for the

flexural and tensile strength to increase when the volume fraction of banana fibres was increased and a negative effect was observed for the impact properties of the composites. Recent studies regarding the use of sisal and jute fibre (Mathur, 2006), abaca (Herrera-Estrada *et al.,* 2008), abaca and sisal (Idicula *et al.,* 2005), flax (Wambua *et al.,* 2003), sisal, abaca and bamboo fibre (Rao *et al.,* 2010) in reinforced polyester composites have also been reported.

2.9 Fabrication Techniques

In many cases, polymer composite processing utilizes the same technique as polymer processing which includes injection moulding, compression moulding and extrusion moulding. There are some other techniques which are unique only to polymer composite processing. These include filament winding, pultrusion and hand lay-up. In spite of the fact that some techniques are used commonly with polymer processing, the operational conditions can be very different; thus, it is important not to directly transfer knowledge without careful consideration.

2.9.1 Compression Moulding

The compression is the most famous method for producing natural fibre-reinforced thermosets and thermoplastic composites (Khan *et al,*. 2012; Sreekala *et al.,* 2002; LU *et al.,* 2006). Natural fibre and resins are taken into mould after well mixing. The mould is then placed in between two plates. Those plates are joined with heating and cooling apparatus. Pressure can be fixed by using hydraulic press. A multiple type of finish products can be found with very simple operation.

2.9.2 Hand Lay-Up

The hand lay-up technique is extensively used for natural fibre-thermosetting polymer composites for simple processing procedures. In this process, short fibres, unidirectional and woven fabrics can be fabricated in resin. The resins are laid on the fabric by using a roller. Sreekala *et al.,* (2002) have prepared the short random oil palm/glass hybrid fibre-PF resin composites by very simple hand lay-up along with compression moulding methods.

2.9.3 Vacuum-Assisted Resin Infusion Moulding

This method is used for making multilayer laminated composite. Unidirectional, non woven or woven mats are sized according to the shape of the mould chamber used in this process. The mould is set in between the resin container and pump. The resin and hardener are infused slowly for wetting mats by vacuum pump. Thereafter, curing is continued for a long time at ordinary temperature (Yuhazri *et al.,* 2010).

2.9.4 Resin Transfer Moulding

Resin transfer moulding is a profitable technique for natural fibre composites. In this technique, resin and hardener are pumped separately to the mixing head before injection into the mould. The required amount of resins injected into the prearranged fabrics. Pressure can be applied from the opposite direction of injected mould. The wastage of resin is very less and good shape of product is found by this method. Indira *et al.,* (2013) studied the effect of fibre length and fibre loading on the properties of banana fibre-reinforced phenol formaldehyde resin (PFR) composites. They found greater tensile and flexural properties in resin transfer moulded composites than in

compression moulded composites. However, it needs big investment for large-scale production.

2.9.5 Bulk Moulding

A male–female combination of a closed mould is needed for bulk moulding. Short fibres are specially used in this process. Nowadays, thermo set bulk moulded product occupied the market of thermoplastic injection moulded compounds such as motor parts, electrical component and housing appliances.(Samivel and Babu 2013). The surface smoothness of finish product is good but contains some void portion which reduces composite strength.

2.9.6 Sheet Moulding

The sheet moulding process is completed in two steps. First, long sheet of composites is prepared with unique thickness, and then the sheets are passed through the different dimensional moulds to prepare various products. Short natural fibre is added with constant weight on carrier foil (conveyer). Resins and fillers are laid from a hopper on fibre. The mixture is then passed through the several ball mills. The prepared composite sheets are rolled with foil. The rolled composite sheets are further moulded for producing different products. (Behzad and Sain 2005) established a novel processing method of natural fibre-thermoset acrylic resins with very short curing step (10 min). The final product has higher mechanical properties but needed huge establishment cost.

2.9.7 Pultrusion

The pultrusion technique is often investigated for natural fibre and thermoset resins (Akil *et al.,* 2009). The yarn rolled roving is passed through the resin bath. A hot die helps them convert into profile (Van de Velde and Kiekens 2001). The profile then flows in profile drawer for completing cure process. The prepared composite has greater mechanical properties, but the production rate is not satisfactory. Among the above-mentioned processes, the compression moulding is a more viable method for fabrication of short OBF–PF resins in small-scale production. Short dried OBF with different weight fraction (9, 19, 29 and 38 wt %) is initially mixed thoroughly with phenol formaldehyde (PF) resin. Composites are made using a stainless steel mould at 150 °C and 50 kN pressure. The curing is completed at room temperature for 24 h keeping the constant pressure 10 kN. The specimens for tensile and flexural tests are made by a cutting machine.(Ariffulzaman,*et al.,* 2014).

2.10 Mechanical properties of fibre reinforced composite

The mechanical properties of fibre reinforced composite depend on many parameters such as, fibre strength, fibre length and orientation, in addition to fibre matrix interfacial bond strength, a good interfacial bond is required for effective stress transfer from matrix to the fibre where maximum utilization of the fibre strength in the composite is achieved. Mechanical properties of natural fibre such as jute ,hemp, flax, sisal are good can compete with glass fibre in terms of specific strength and modulus (Azam ,*et al.,* 2009).

2.10.1 Mechanical Properties Of Short Fibre Composites

Mechanical properties of short fibre reinforced composites depend critically on the fibre length distribution (FLD) and the fibre orientation distribution (FOD) (Fu, and Lauke, 1996). In particular, the strength of short fibre reinforced composites increases with the increase of the mean fibre length and with the decrease of the mean fibre orientation angle (angle between the fibre axis and the loading direction) The elastic modulus (E) of misaligned short fibre reinforced polymer composites depends on the distributions of fibre lengths and orientations within the composite structure. In general, the composite elastic modulus increases with the decrease of the mean fibre orientation angle and with the increase of the fibre orientation coefficient; and the elastic modulus increases with the increase of mean fibre length when the mean fibre length is small. When the mean fibre length is large, it has nearly no influence on the elastic modulus of short fibre reinforced composites (Fu, and Lauke, 1996).

2.11 MORPHOLOGICAL ANALYSIS

2.11.1 Scanning Electron Microscopic Analysis:

Scanning electron microscope is a type of microscope that produces image of a sample by scanning it with a focused beam of electron. The electron interacts with the atoms in the sample, producing various signals that can be detected and that contain information about the samples surface topography. Sub-surface information and compositional difference. SEM is used to determine the size, distribution and orientation of fibres or particles, it identify and characterize any defects (voids, de bond fibre pullout and fibre bonding) . it also gives information on the degree of bonding between the matrix and reinforcement or between layers.

To prepare samples for SEM, insulating samples are coated with thin layer of Gold, Aluminum or Carbon by evaporation or sputtering. Samples can be viewed with different Kilo Voltage and can be obtained at different magnifications (webinar, 2011).

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

Okro stems which was extracted by water retting.

Sodium hydroxide (Johnson chemical company Kano Nigeria)

Acetic acid

Unsaturated polyester resin) (Olasco Chemical company Zaria, Nigeria)

Methyl Ethyl Ketone peroxide (MEKP) (Olasco Chemical company Zaria, Nigeria)

Accelerator (cobalt)) (Olasco Chemical company Zaria, Nigeria)

3.1.1 Equipment

i. Comb
ii. Universal Material Testing Machine (Cat Nr. Model 261). At mechanical engineering department of Ahmadu Bello University Zaria.
iii. Analytical Balance (Sartorius.Model :ED2245)
iv. Type "W" Mosanto Tensometer(Made in UK by Monsanto) At Ahmadu Bello university Zaria
v. Phenon Scanning Electron Microscope (Model: Pro X) by World Eindhoven Neither Land At Department of chemical engineering, Ahmadu Bello university Zaria.
vi. Charpy Impact Tester "15 Joules capacity (Cat Nr. Model 412). At department Ahmadu Bello university Zaria
vii. Indentec Universal Hardness Testing Machine,(Model: 8187.5) LKV "B"

viii. Glass mould of 200x120x80 (mm)

3.2 Methods

3.2.1 Okro Fibre Extraction

The Okro stems were obtained from a small farm in Gwarzo local government area of Kano state. The fibres samples were extracted by water retting method. The okro bark was bundled in ribbon form and immersed in water retting bath, little pressure was applied to the soaked bundle to ensure that the bark were fully submerged for a period of 10 days during which the cementing materials such as pectin, lignin, cellulose, and hemicelullose must have loosen and softened. On 10^{th} day, the retted ribbon was removed and washed with sufficient quantity of water untill the pulp was completely detached from fibres; the fibres were shredded and combed to have finer fibres. Then the fibres were dried at room temperature for a period of seven days (7). After drying, the fibres were chopped into short lengths (5-10mm) which were used for production of the composites samples.

3.2.2 Alkaline Treatment Of Okro Bast Fibre

Sodium hydroxide solutions by weight concentration of 5%, 10% and 15%were prepared using distilled water and sodium hydroxide. The fibres were soaked in the respective solutions for a period of thirty minutes with continuous stirring. They were later removed from the solution and rinsed with water, then each batch neutralized with 1% acetic acid and then finally rinsed with distilled water.

3.2.3 Preparation Of The Composites Samples

The alkaline treated Okro bast fibre reinforced unsaturated composites were prepared using hand-lay-up method. The required amount of Unsaturated Polyester Resin, accelerator (cobalt) and catalyst (MEKP) 2% to the resin volume were weighed out and thoroughly mixed. The corresponding amount of Okro fibres were weighed out and added to the UPR. It was thoroughly mixed to ensure even distribution of fibres. The mixture was then poured in to a mould in a glass mould (200 x 120x80 mm) on which foil paper has been laid for ease of removal of the produced composites. On addition of the promoters, (MEKP and Cobalt) the resin becomes more viscous until it reaches a state where its lost ability to flow (gel point). Exothermic reaction takes place which speed the whole process, and later on the resin obtained its full hardness and properties. The produced composites were cure under laboratory conditions before subjecting them to morphological, physical and mechanical analysis. One control sample, three untreated samples and nine alkaline treated samples were produced for this research.

3.3. Okro Bast Fibre/Unsaturated Polyester Resin Composites

Composites of Okro bast fibre/Unsaturated polyester resin composites were prepared by weighing out the required volume of unsaturated polyester resin, accelerator and catalyst. The corresponding amount of Okro fibre as shown in table 3.1 below

Table 3.1: Okro bast fibre/unsaturated polyester resin composites

S/N	% OF OBF	% of UPR	Mass of OBF	Mass of UPR	Total mass
1	0 (control)	100	0 (control)	90.40	90.40
2	5	95	4.52	85.88	90.40
3	10	90	9.04	81.36	90.40
4	15	85	13.56	79.84	90.40

3.4 Characterization of Composites

3.4.1 Tensile Testing

Tensile strength and elongation at break were carried out using Mosanto Tensometer machine. The test was performed according to ASTM D638 (2014). The test samples where clamped between the upper and lower jaw of the tensometer and the machine was started. The sample was stretched gradually with application of force until it reached the breaking point. Reading of maximum load and elongation at break were taken accordingly. The test was repeated Three (3) times for each of the composites and the average values were recorded.

3.4.2 Flexural Test

The flexural test was conducted according to ASTM D790 (2010) using the Cat Nr Universal Testing Machine using the three point bend test. The flexural strength was calculated using Equation

$FS=3PL/2bd2$ …….. (1)

Where L =is the span length of the sample (guage length);

P is the load applied;

b and t are the width and thickness of the specimen respectively.

3.4.3 Impact Test

The impact test was performed according to ASTM D256 (2010) standard using Charpy Impact Testing Machine. The composites samples were cut to a length not more than 10cm and a uniform width, the sample was placed on the machine and held tightly with the aid of knots, the ends were observed to be of equal length and a hammer of 15j capacity was raised and then released to hit the sample. Work done on breaking the samples was recorded, test was repeated three times and an average value was recorded.

3.4.4 Hardness Testing (Rockwell Hardness)

In this study, the Indectec Universal testing Machine was used in measuring the hardness of the samples according to ASTM 2240 (2015). The sample with parallel flat surface was placed on the avail of the apparatus and lowering the steel ball onto the surface of the sample. The dial was adjusted to zero on the scale under minor load (10kg) and the major load (60kgf) was immediately applied by releasing the trip lever. After 15 seconds the major load was removed and the specimen was allowed to recover for 15 seconds the test was repeated three times and an average value of each test result was recorded.

3.4.5 Water absorption

Water absorption was carried out according to ASTM 2842 (2012) Thirteen (13) samples were cut to a rectangular shape and size (2x2cm) and weighed using digital weighing balance. The weighed samples were placed in a plastic container and enough water was

added so completely covered, the composites samples and left in water for 48 hours. The samples were removed from the water, wiped with a clean cloth to eliminate moisture, and then re- weighed and the process continues for thirty days. The percentage water absorbed was calculated using Equation

$$percentage\ water\ absorption = \frac{final\ weight - initial\ weight}{initial\ weight} \times 100 \qquad \text{.....................} (2)$$

3.4.6 Density

The weight of the test sample was measured after which it was submerged in to a known volume of water. The volume of the water displaced was recorded; densities of the composites were determined according toASTMD792-13 (2013).

The densities of the composites were then determined using the equation below in grams per centimeter cube (g/cm3).

$$\rho = \frac{m}{v} \qquad \textbf{.. (3)}$$

(Danladi and Shuibu 2014)

3.4.7 Scanning Electron Microscopic Analysis

The composite samples were analyzed by Phenon SEM Machine, using the tensile fractured surface of the alkaline treated and untreated samples. The fractured surfaces were coated with 5mm of gold using a sputter machine, the coated samples were then placed on a sample holder and into the machine column then viewed through a navigating camera. However, adjustment was made before transferring into the electronic code, viewing voltage was set using 10KV, magnification were increased from (300 to 500 and

1000) and the sample morphology were stored in the electronic mode. The machine setting was changed from electronic mode to navigation camera before ejecting the samples from the machine.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

In this section, the results obtained are presented and discussed. The analyses of the results were done to establish whether significant differences existed between the mechanical and physical properties of the various composites as a result of the variation in their compositions.

Result Of Tensile Strength Of Control Sample Treated And Untreated OBF/UPR Composites

The tensile strength of a material is the maximum amount of tensile stress that it can take before failure or breaking. The tensile strength of a natural fibre reinforced composite is determined by many parameters, such as fibre strength, modulus, fibre length and orientation, in addition to the fibre-matrix interfacial bond strength. A strong fibre-matrix interface bond is very critical for composites to have high tensile strength. A good interfacial bond is also required for effective stress from the matrix to the fibre whereby maximum utilization of the fibre strength in the composite is achieved (Karnani, *et al,*. 1997). Figures 4.1 show the effects of alkali treatment on tensile strength at different fibre loading.

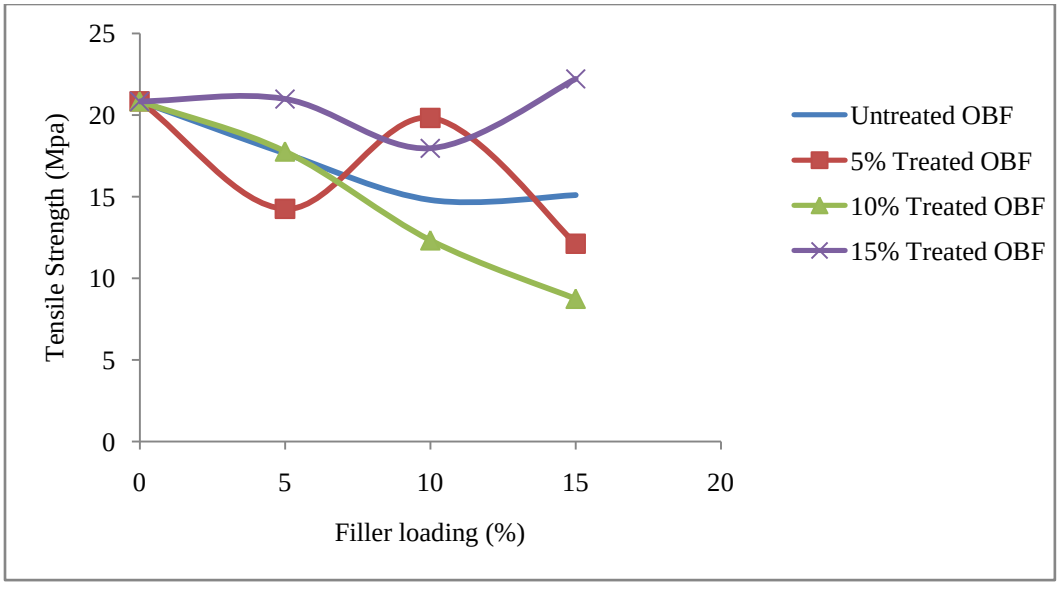


Figure 4.1: Tensile Strength Against Filler Loading Of Untreated And Treated OBF /UPR Composites.

The test result showed that 15% alkali treated, and 15% filler loading has the maximum tensile strength with a Value of 22.21MPa when compared to control samples and other samples reinforced with 5%, 10% filler loading. However maximum tensile strength for treated okro bast fibre /unsaturated polyester resin composites indicate that the alkali treatment has improved the adhesive characteristics of the fibre surface by removing the natural and artificial impurities there by producing rough surface topography. Similar result was results were reported by Sirgar *et al.*, (2010) On their study of effect of alkaline treatment on the mechanical properties of short pineapple fibre (PALF) reinforced high impact polystyrene (HIPS) composites. This result also agrees with the findings of Pickering *et al*.2007 who studied the optimizing of industrial hemp fibre for composites.

As shown in Figure 4.1, the tensile strength of 5% alkali treatment and 5% filler loading was observed to decrease to 12.25 Mpa the decrease in strength of these composites

could be due to agglomeration formation which resulted in formation of stress centres in the composites which initiate failure on application of stress. This is in line with the findings of Shoney and Melo (2007). The strength further increases to 19.82 Mpa when 10% filler content was added to the matrix. The strength later dropped to 12.12 Mpa respectively. Similar trend was observed for 10% alkali treatment at 5%, 10% and 15% filler content with a decrease in tensile strength corresponding to 17.76, 12.23 to 8.74 Mpa. The present study agrees with the findings of Sumaila, *et al*., 2013, which explains the observed decrease in tensile strength could be attributed to improper fibre wetting due to increased tendency of fibre entanglement with increasing short fibre as well as the possibility of increasing fibre-rich and/or matrix-rich areas within the composites.

It was also noticed that values obtained for the untreated okro bast fibre/unsaturated polyester resin composites is lower than that of the control sample and some treated okro bast fibre/ unsaturated polyester resin composites with the observed values 17.64, 14.79 and 15.09 Mpa respectively. The observed decrease in strength of the untreated okro fibre/unsaturated polyester resin (UPR) composites could be attributed to poor fibre/ matrix adhesion which leads to micro cracks formation at the interface under loading and non uniform stress transfer due to fibre agglomeration in the matrix. This is in line with the findings of (Sanjay *et al.,* 2009) who studied the hybridization effect of glass fibre on mechanical, morphological and thermal properties of polypropylene-bamboo/glass fibre hybrid composites.

4.2: Result Of The Percentage Elongation Of Treated And Untreated Composites Samples

Figure 4.2 presents the results of the effect of alkaline treatment and filler loading on elongation of Okro bast fibre / unsaturated polyester resin composites. It was observed that there was great improvement in the percentage elongation of the reinforced composite to those unreinforced with the various filler loading of the okro fibre. The 15% alkaline treated fiber and 5% filler loading composite sample exhibited the highest extension at break corresponding to 17% elongation. The value is observed to be higher than the values obtained for the control, untreated, 5% and 10% alkaline treated okro bast fibre samples. The enhanced elongation on treatment was due to the fact that more hemicelluloses are removed from the fibre as a result of treatment, which provided more surface roughness, hence giving rise to better interlocking with the matrix. Such result was obtained by Isa *et al.*, (2014) on their study on effects of fibre treatment on mechanical and water adsorption of short okro fibre hybridize epoxy composites.

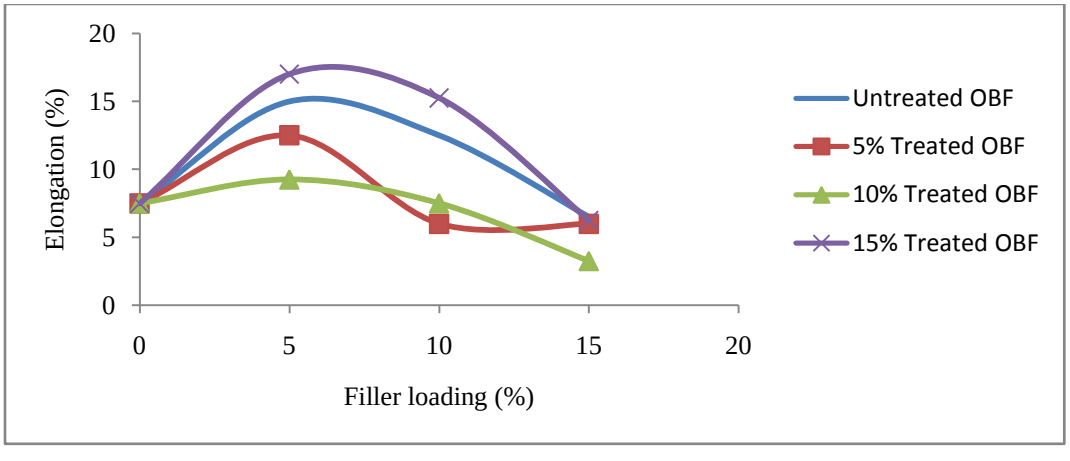


Figure 4.2: %Elongation against Filler loading of Untreated and Treated OBF/UPR Composites

The percentage elongation was observed to decrease with increase in filler loading content for all the composites, the increase of filler loading in the matrix (UPR) resulted in stiffening and hardening of the composites which reduced its ductility and led to lower elongation property. However the reduction in elongation at break indicates the incapability of the filler to support the stress transfer from the matrix to the fibre. Similar result was obtained by (Shuhadan and supri 2009) The values obtained for 5% alkaline treatment were 12.5% to 6% and 9.25%, 7.5% to 3.35% for 10% alkali treatment. The untreated OBF/UPR composites have its highest elongation at 5% and later dropped to 6.5% respectively.

4.3: Results Of Young's Modulus Of Treated And Untreated Composites

Young's modulus is also known as the tensile modulus which is referred to as measure of stiffness of material. It is defined as the ratio of stress along its axis over the strain along its axis. Young's modulus may have different values depending on the direction of the applied force with respect to the material structures. A material whose Young's modulus is very high is to be rigid. The presence of Okro fibres in UPR matrix contributes effectively to enhance the tensile modulus of UPR resin. It is known according to composite theory that the tensile modulus /young's modulus of a fibre- reinforced composite depends on the modulus of the fibre and the matrix, the fibre content and orientation. (Tran *et al.*, 2011).

Figure 4.4 illustrated the results of effects of alkaline treatment and filler loading on young's modulus of the okro bast fibre/ unsaturated resin composite. The 15% alkaline treated okro bast fibre and 15% filler loading exhibited the highest modulus of (287.36MPa) and degree of stiffness, which is similar to the result obtained for tensile

strength in this research. However, the 15% alkaline treated okro bast fibre /unsaturated polyester resin composites appears to be greater than the control sample (259.09), untreated samples (125.75, 227.53 and 137.5 Mpa), 5%alkaline treated sample (137.5,158.36, and 202) and 10% alkaline treated sample with the values of (133.18, 236.81 and 268.92) which were loaded with 5%,10% and 15% filler loading respectively.

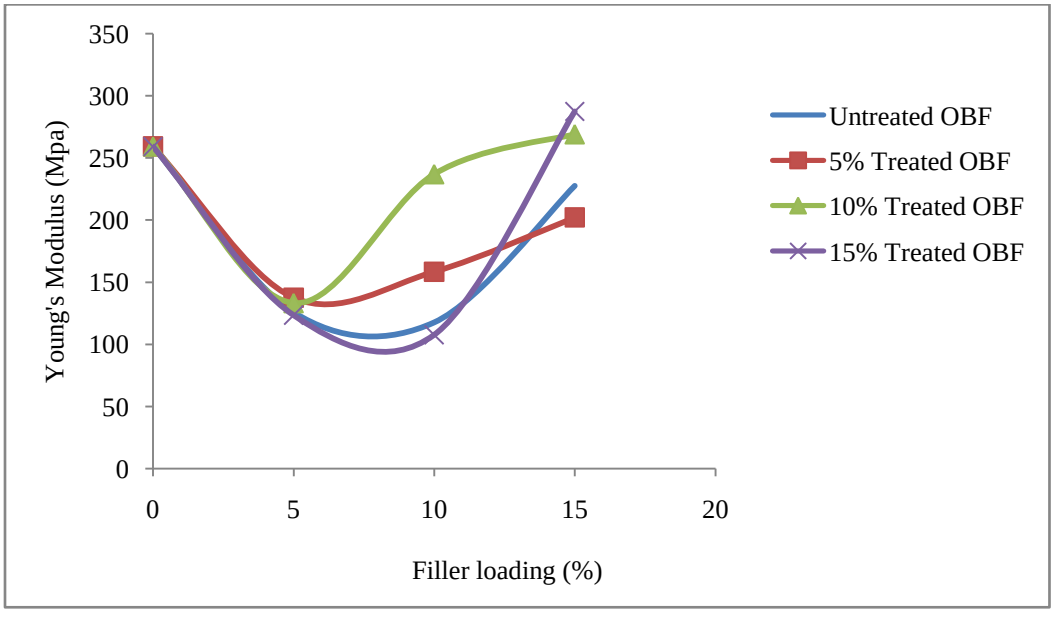


Figure 4.3: Young's Modulus Against Filler Loading Of Untreated And Treated OBF/UPR Composites.

Nara *et al*., (2012) observed that the young's modulus, which is an indication of load bearing capacity increases with fibre weight fraction. As fibre is the stiffer component in the composite, resistance towards deformation increases with increase in fibre content, this consequently increases the stiffness of the composite. Hence the variation in the modulus observed in the various researches could be attributed to the variation in the weight fractions of the fibres, the difference in matrix and the nature of the fibres used. Similar study was conducted by (Sreekala *et al*., 2002).

4.4: Result Of Flexural Rigidity Of Treated And Untreated Composites

Figure 4.4 shows a variation in flexural strength with increase in filler loading and NaOH concentration for treated okro bast fibre /unsaturated polyester resin composites. It was observed that flexural strength of all the composites increases with increase in filler loading. The increase in flexural strength of composites is primarily due to effective stress transfer from the fibres to the matrix which may be an indication of better adhesion between the fibre and the matrix. The improvement of the flexural strength was also reported by (Sanjay *et al.*, 2009) who studied the hybridization effect of glass fibre on mechanical, morphological and thermal properties of polypropylene-bamboo/glass fibre hybrid composites.

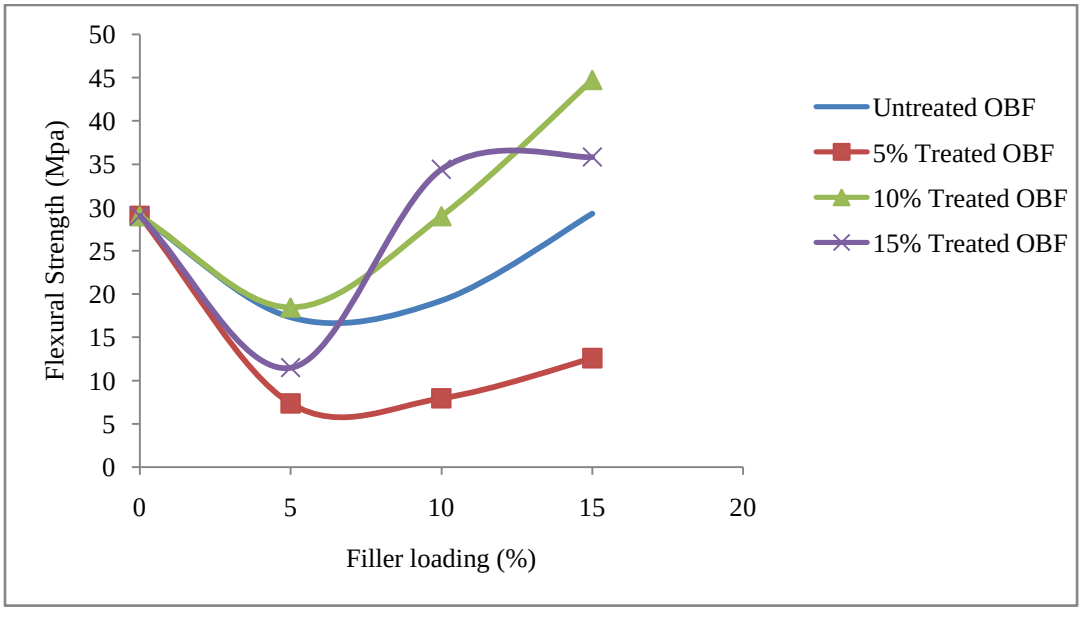


Figure 4.4: Flexural Strength Against Loading Of Treated And Untreated OBF Composites.

At 10% NaOH treatment the flexural strength increases from (18.45, 28.45 to 44.72 Mpa) which is the highest value obtained for all the samples. However, 15% NaOH treatment also exhibit similar trend that is the flexural strength increases with increase in filler loading corresponding to 11.48Mpa, 34.39Mpa to 35.81Mpa respectively. This could be due to good intermolecular interaction between the okro bast fibre and the resin. Similar results were obtained by Tran, et *al.,* (2011) in their study on effect of alkali treatment on interfacial and mechanical properties of coir fibre /poly (butylenes succinate) biodegradable composites. 5% alkaline treatment was observed to have the lowest value of flexural strength corresponding to (7.35, 7.95, 12.58 Mpa). However, the observed decreased in flexural strength could be due air trap into which leads to micro crack formation in the interface under loading and non uniform stress transfer due to the fibre agglomeration in the matrix. Similar result was reported by (Sanjay *et al.*, 2009). The untreated okro bast fibre /unsaturated polyester resin composites are said to have the observed value 17.3, 19.23 to 29.27Mpa respectively.

4.5: Result Of Hardness Values Of Treated And Untreated Composites Samples

Hardness is the measure of the resistance of material to surface indentation, it is also a function of the stress required to produce specific type of surface deformation and the values that were obtained are used to estimate the mechanical strength of each composite. This shows that fibres that increase the modulus of composite are expected to increase the hardness of the composite because hardness is a function of the relative fibre volume (Obasi, *et al.,* 2014). Figure 4.5 shows the variation in hardness values of treated and untreated okro bast fibre /unsaturated polyester resin composites. Hence the highest value indicates greater resistance of the composites to indentation.

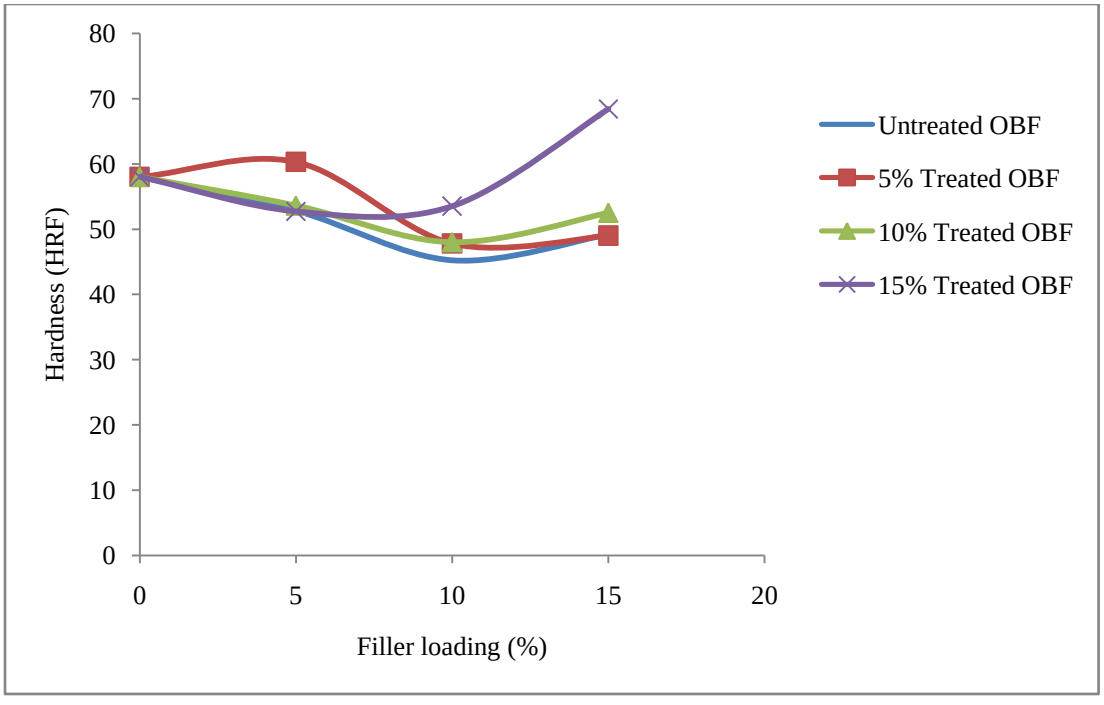


Figure 4.5: Hardness Against Loading Of Treated And Untreated OBF /UPR Composites.

An increase in hardness value for 15% alkaline treated okro bast fibre /unsaturated polyester resin composites was noticed with increase in filler loading content with the observed values of 52.3, 53.5 and 68.4 HRF. The increase in hardness value could be attributed to the increase in stiffness and the dispersion of the fibres into the matrix, minimization of voids and stronger interfacial bonding between the matrix and the fibre as a result of the treatment. Similar findings were observed by Siregar *et al.*, (2010). The 5% alkali treated okro bast fibre/unsaturated polyester resin composites show average values of hardness with increase filler loading, the hardness value decreases from 60.3 to 47.8 HRF and further increase to 49.0 HRF for 5, 10, 15% filler loading. Similar trend was observed for 10% alkaline treated okro bast fibre /unsaturated polyester resin having a value of 53.6, 48.0 and 52.6 respectively. The untreated sample shows a moderate

improvement in hardness value from 52.8, 45.2 and 49.2 HRF. The decrease in hardness value could attribute to poor/weak interfacial bonding between the okro fibre and the unsaturated polyester resin matrix.

4.6: Result Of Impact Strength Of Treated And Untreated Composites Samples

Impact strength is the ability of the material to withstand shock loading, it is also the ability of the material to absorb mechanical energy in the process of deformation and fracture under loading. The impact properties of a composite material are directly related to its overall toughness. Composite fracture toughness is affected by inter laminar and interfacial strength parameter (Mishra *et al.*, 2003).

Figure 4.6 above show the impact strength of treated and the untreated okro bast fibre /UPR composites with variation in filler content. It has been reported that the impact strength of a composite is influenced by many factors including the matrix fracture, fibre/matrix de bonding and fibre pull out (Obasi *et al.*, (2014).

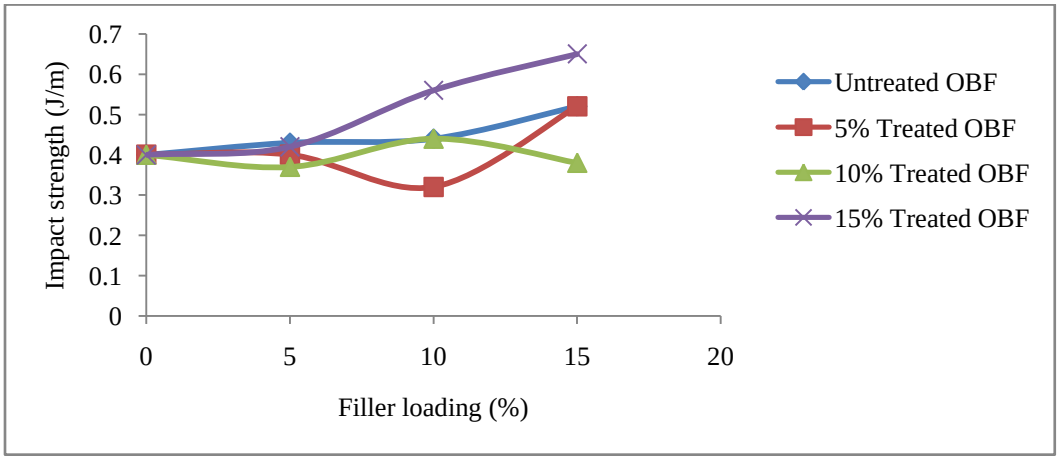


Figure 4.6: Impact Strength Against Filler Loading Of Treated and Untreated OBF /UPR Composites.

The impact strength of the unsaturated polyester resin was improved by about (62.5%) when reinforced with alkaline treated okro fibres. It can be seen that there is an increase in the impact strength of the composites reinforced with the treated fibre when compared with the untreated fibres showing the positive effect of the alkaline treatment composites. This shows that the treatment of the okro bast fibre/unsaturated polyester resin improves the compatibility and promotes the ability to dissipate energy during fracture of the composites.

It was observed that 15% alkaline treatment exhibit the maximum impact strength with the observed values 0.42 (J/m), 0.56 (J/m)and 0.65 (J/m), the increase in impact strength is primarily attributed to reinforcement effect imparted by the fibres, which allowed stress distribution from continuous polymer matrix to dispersed fibre phase. However, a slight improvement for 10% alkaline treatment was noticed having the following values respectively. 0. 37 to 0.44 which later decline to 0.38 J/m with increase in filler loading content. Similar trend was observed for 5% alkali treatment.

The observed decline in impact strength could be due to poor wetting of the fibre with the matrix, which in turn leads to composites with weak interface. Similar result was observed by Thwe and Liao, (2003) in their study on Durability of bamboo-glass fiber reinforced polymer matrix hybrid composites.

4.7: Result Of Density Of Treated And Untreated Composites Samples

The variation of density decreases with increase in filler loading of all the treated and untreated okro bast fibre /unsaturated polyester resin composites is shown in figure 4.7. The density of all the composites decreases with increase in filler loading. This is due to low density of the fibre than the matrix thereby resulting in composites with low density.

Such findings was observed by (Sirnivasababu,*et al*.,2009). Natural fibres have the advantage of having light weight, which proves density decreases with increasing fibre content this is similar to the result obtained by Danladi and Shuibu (2004) in their study of fabrication and properties of pineapple fibres /high density polyethylene composites.

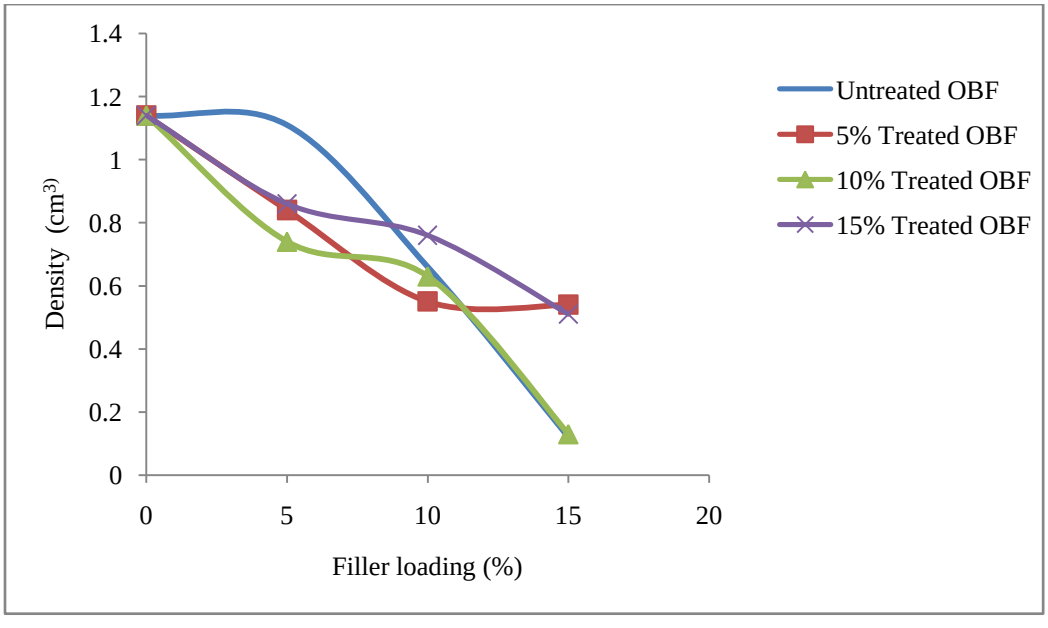


Figure 4.7: Density Against Filler Loading Of Untreated And Treated OBF/UPR Composites.

The value obtain for density of the okro bast fibre/unsaturated polyester resin composites shows slight decrease from 1.11,0.66, to 0.12 g/cm3 which is lower than the control sample (1.14g/cm3), similar trend was observed for 5% 10%,and 15% having the values of (0.84 ,0.55, 0.54g/cm3) (0.74,0.63 and 0.13g/cm3) and (0.86,0.76 and 0.51g/cm3) respectively.

4.9: Results Of Water Absorption (%) For Treated and Untreated Composites Samples

Water absorption is the amount of water uptake by a composites material when immersed in water for a stipulated period of time. The rate of water absorption depends on the

internal material states, nature of reinforcements, fibre matrix interface and environmental factors such as temperature and applied stress.

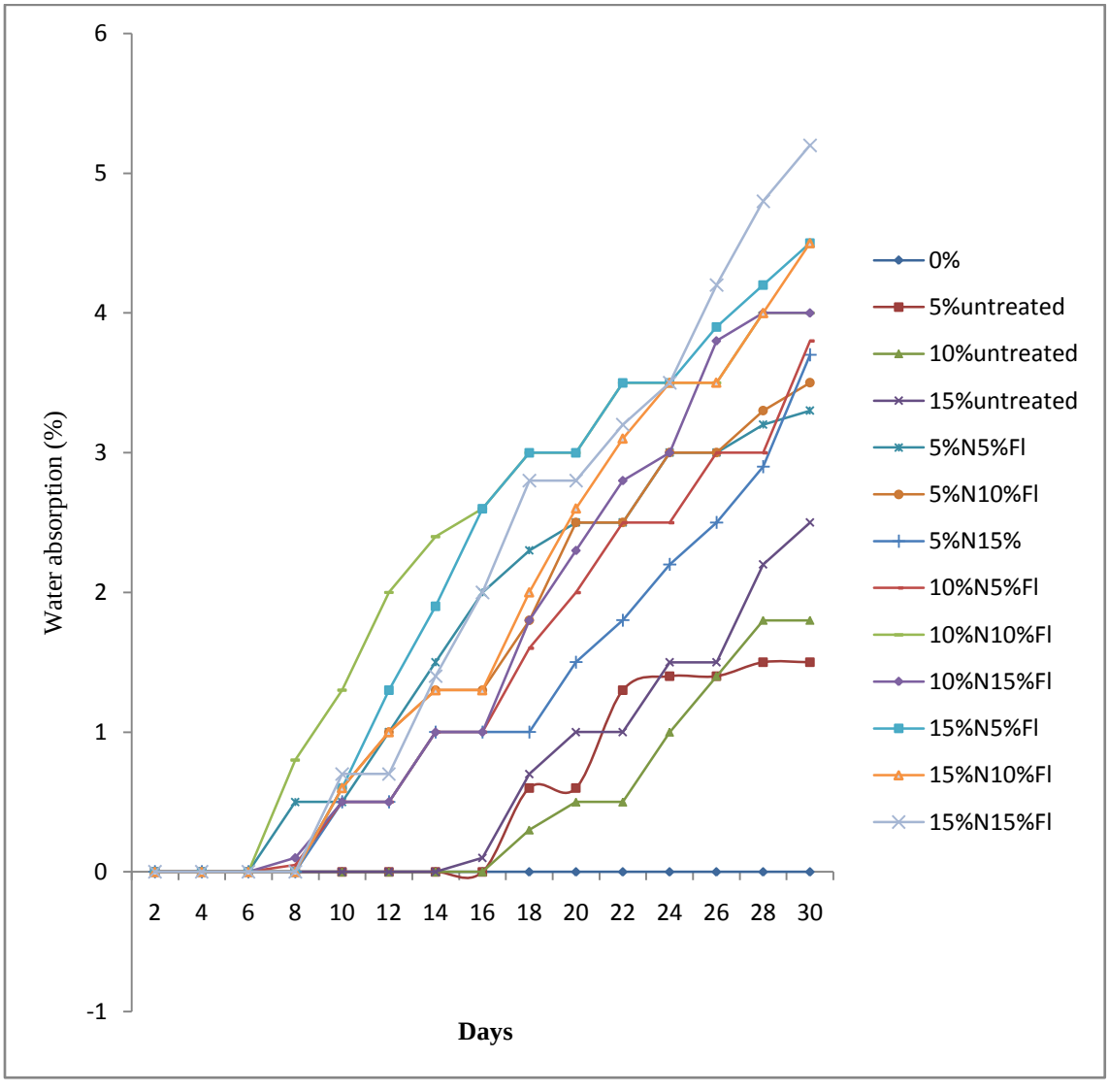


Figure 4.7: Water Absorption (%) Versus days Under Different NaOH Concentration/Filler Loading Of Treated And Untreated OBF /UPR Composites.

Figure 4.7 shows the percentage water absorption of treated and untreated okro bast fibre /unsaturated polyester resin composites. It was observed that the unreinforced sample (control sample) composite absorbed the minimum amount of water due to its hydrophobic nature and on reinforcing it with the okro fibres, moisture absorption

increased as a result of the introduction of the hydrophilic of okro bast fibres into the matrix (UPR). However, treatment of okro bast fibre with NaOH at varying concentrations of 5%, 10%and 15% increases, water absorption with increase in filler loading the highest water absorption was observed at 15% alkali treatment with a value of 5.2% and the lowest value for the treated OBF was observed at 5% NaOH treated OBF having the value of 3.3%. This implies that the denser solution of NaOH treatment increased the water uptake of the composite when compared to the lower concentration (5%) of NaOH solution treated fiber composites. This also showed that alkaline the treatment had removed the protecting cell fibres and swelling process was much faster, leading to higher water uptake this agrees with the findings of (Dhanalakshmi *et al.*, 2012) in their study on effects of chemical treatment on water absorption of areca fibres .

The reduction in the water absorption could be attributed to the greater adhesion between the fiber and the matrix, which could be possible due to the surface treatment of the fibers such results is reported by (Mishra *et al.*, 2003). Hetal et al., 2012 also concluded that by providing additional sites for mechanical interlocking, alkali treatment leads to improvement of interfacial bonding hence, promoting resin-fiber interpenetration at the interface. The hydrophobic resin pick-up could also account for the reduction in water absorption. However the untreated fibres absorbs less water from 0%to 1.5% for 5% filler loading, 0%to 1.8% for 10% filler loading and 0% to 2.2% for 15% filler loading, this Samples has the least value when compared to all the treated okro bast fibre / unsaturated polyester resin composites

4.8 Morphology

4.8.1 Scanning Electron Microscopy (SEM)

Fibre matrix interaction and fracture behaviour of untreated and treated Okro bast fibre /Unsaturated polyester composites were studied using scanning electron microscopy (SEM) of tensile fractured surface of 5%,10% untreated and 5%,10% treated samples of the composites were used. Plates 4.8 (a-d) shows the micrograph of 5% and 10% untreated fibre reinforced unsaturated resin composites and 5% 10% alkali treated okro fibre composites at the magnification of 500x

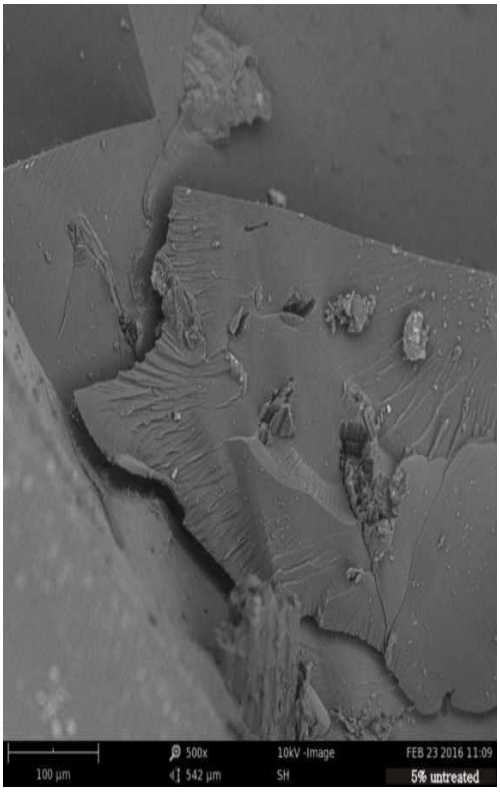


Plate 4.8(a): 5% UOBF/UPR at 500x

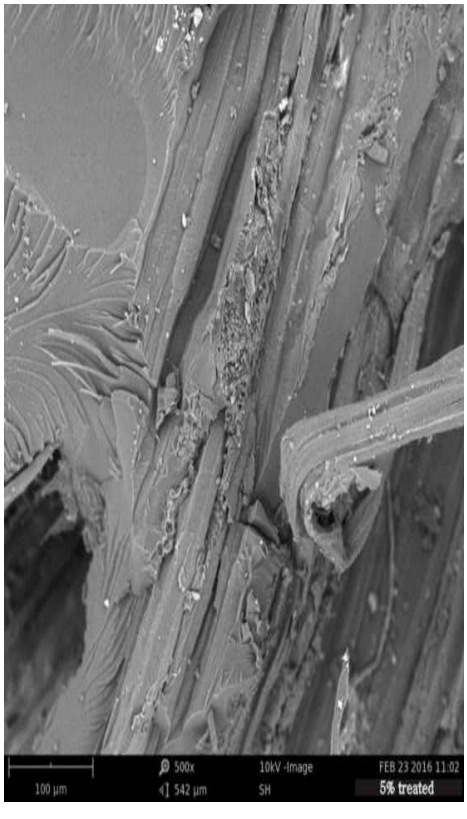


Plate 4.8(b): 5% TOBF/UPR at 500x

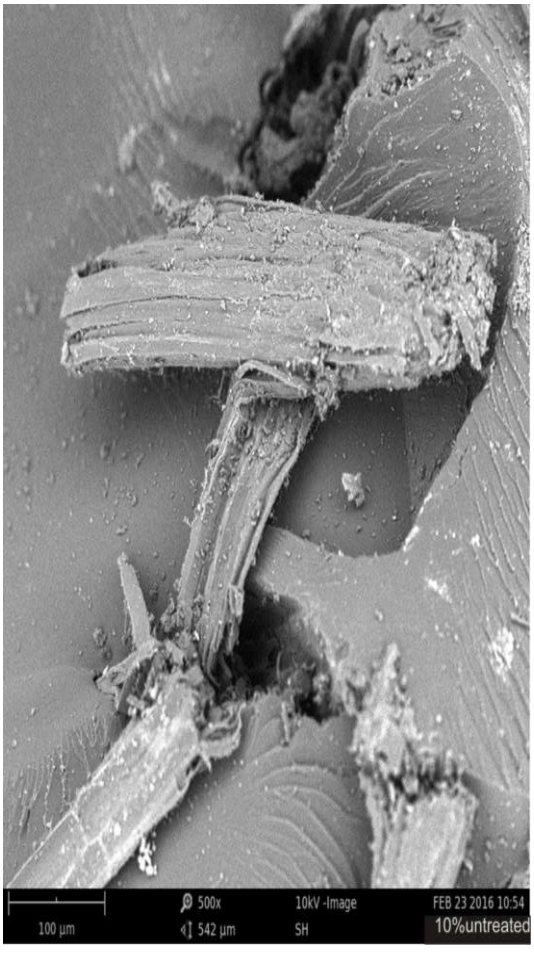


Plate 4.8(c): 10% UOBF/UPR at 500x

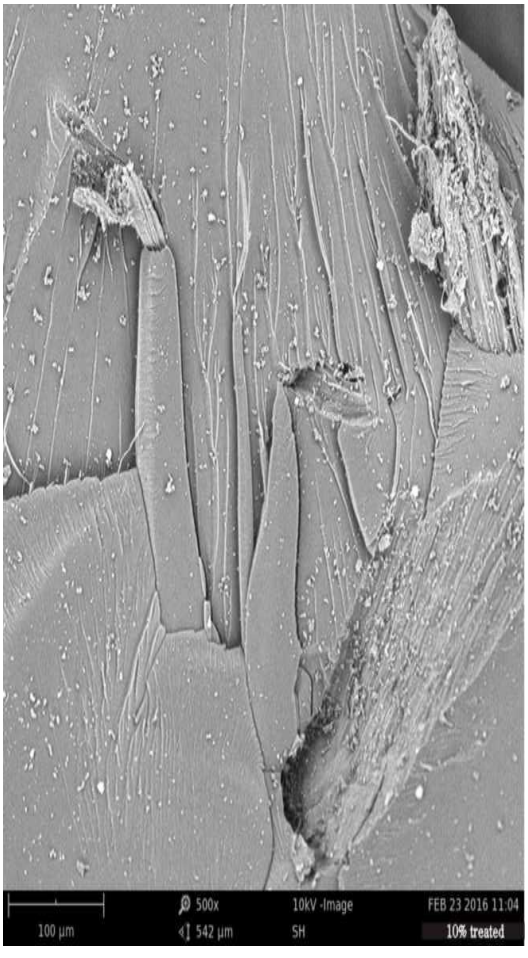


Plate 4.8(c): 10% TOBF/UPR at 500x

Morphology studies of okro bast fibre / unsaturated polyester resin revealed that the alkaline treatment of the okro bast fibre leads to fibre fibrillation that is breaking of fibre bundles into smaller fibres, which increase the chance of effective surface area to be available for contact with the matrix. Similar result was reported by Sirgar, *et, al* (2009). However, the use of alkaline treated fibre leads to better adhesion between the fibre and the matrix (UPR) indicating good wetting and strong interface. The evidence for this is provided from the matrix traces seen on the surface of the fibre. Very little fibre pull out suggests that the matrix has been efficient in holding on to the fibres.

This indicates that the composites have higher degree of fibre orientation under the stress, which results in higher fibre efficiency factor and hence improved mechanical strength. The okro fibres protruding from the composite indicate the degree of fibre pull out and crack deflection. The fibre surfaces that protrude are not clean with some adhering matrix material on it further indicating that the fibres are well adhered to the matrix. For the untreated fibre loaded with 5% and 10% Okro bast fibre composites shows fibre breakage, fibre pull out and voids or air entrapments which, is as a result of poor/matrix adhesion which leads to micro crack formation at the interface under loading and non uniform stress transfer due to fibre agglomeration in the matrix similar findings have been reported by Sanjay, *et al*,. (2009).

CHAPTER FIVE

5.0 SUMMARY, CONCLUSION AND RECOMMENDATIONS

5.1 SUMMARY

The aim of the whole research work was to investigate the effect of alkaline treated Okro bast fibres as fillers in unsaturated polyester resin composites. Some of the treated fibres were subjected to morphological, physical and mechanical properties. Simple hand layup technique was used for the production of the composites. Observation made was that alkali treatment and filler loading played a significant role in improving the mechanical and physical properties of the composites.

It was observed that tensile strength for 15% alkaline treated okro fibre /unsaturated polyester resin composites with 15% filler loading is 6.65% greater than the values obtained for the control sample and 25.9, 50.17 and 47.18% greater than the untreated samples loaded with 5,10 and 15%filler loading. A difference of (169, 12.08 and 83.25%) and (25.05, 80.28 and 154.11%) was observed for 5% and 10% alkaline treated okro fibres loaded with 5, 10 and 15% filler loading when compared to 15% alkaline treatment. It was found that this treatment improved greatly the tensile strength of the fibre to a certain degree; sodium hydroxide concentration gave impaired tensile strength. The treatment of okro bast fibre with alkali showed an increased proportion of the effective surface area available for contact with the unsaturated polyester matrix polymer thus leading to increase in the tensile strength and modulus of the reinforced composites (Klemm 1998). This gives the composites the ability to sustain higher loads before failure occurred. It was also observed that young's modulus increases with increase in filler loading having its maximum 15% alkali treatment with a value of 287.36Mpa

respectively. 15% alkali treatment and 10% filler loading of percentage elongation was also observed to be 103.3% higher than the control sample, (22 and 134%) greater than the untreated okro bast fibre/unsaturated polyester resin composites. It could also be seen that a difference of 22, 154% and 64.9, 103% was observed for 5 and 10% alkaline treated okro fibre /unsaturated polyester resin loaded with 5%, 10% and 15% filler loading respectively.

The flexural strength of the composites obtains it highest value at 10wt% alkaline treatment of okro bast fibre /unsaturated polyester resin composite indicating better adhesion between the fibre and the matrix, with the value of 44.72 Mpa. It can be noticed that the value (44.72) is greater than that of the control sample by 54.2% and greater than untreated samples with the observed values 158, 225.5 and 24.88% respectively.

The Charpy impact result obtained showed that the characteristic of the fibre is a crucial factor in determining the response of composite materials to impacting force; as the impact strength of the unsaturated polyester resin was improved by about 70.37% when reinforced with 15% filler loading of the untreated fibre composites. Composites with treatment have better response to impacting forces. The impact strength is found to be proportional to the volume of reinforcement as increase in filler loading improves the impact strength.

Rockwell hardness result of the composite specimens showed that the inclusion of the alkali treated Okro bast fibre/unsaturated polyester resin composites improved the hardness of the composites. The density of the control sample was observed to have the highest value of 1.14 g/cm^3, while the samples for treated and untreated OBF/UPR composites decreases with increasing filler loading.

The alkaline treated Okro bast fibre/unsaturated polyester resin composites absorbed the maximum amount of water which could be due to the removal of the cementing materials on the surface of the fibre with the following values for4.5% and 5.2%, while the control sample absorbs the least amount of water. The morphological analysis showed that the alkali okro fibre reinforced composite shows better adhesion between the matrix and the fibre indicating good wetting and strong interface adhesion between the fibre and the matrix.

5.2 CONCLUSION

This work resulted in the successful production of okro bast fibre/ unsaturated polyester composites using simple hand lay-up technique and the following conclusions were drawn:

The investigation of physical and mechanical properties as well as morphology of the composites is the most important techniques in studying the behavior of the composite material. The study investigated the effect of alkaline treatment on okro bast fibre /unsaturated polyester resin composites at varying concentrations of NaOH and filler loading. However The tensile strength ranging from (8.74Mpa to 22.21), young's modulus (107.59 to 287.37 Mpa), percentage elongation (3.25 to 19%), flexural strength,(7.95 to 44.72 Mpa) impact strength (0.32 to 0.65 to J/m) and hardness (45.2 to 68.4 HRF) have values dependent on varying NaOH concentration and filler loadings. The tensile modulus of Okro bast fibre/unsaturated polyester resin composites increased with increasing NaOH concentrations and filler loadings.

Further it was observed that there is an increase in impact and hardness value with increasing alkaline treatment and filler content. The properties of untreated Okro bast fibre/unsaturated polyester resin composite decreased with increase in filler loading. Reduction in densities with increase in filler loading was observed and moderates water absorption was observed for the untreated okro bast fibre while the treated Okro bast fibre /unsaturated polyester resin has the maximum uptake of water, Scanning electron microscope (SEM) showed better adhesion between the fibre and the matrix was observed for the treated Okro bast fibre/unsaturated polyester resin composites which result in moderate improvement in mechanical and physical properties of the composites.

5.3 RECOMMENDATIONS

1. Okro bast fibres should be used as an alternative to synthetic fibres in composites materials as it is found to be cost effective..
2. Other composite moulding techniques instead of local hand mixing should be used in the preparation of these composites to ensure optimal quality.
3. 15% alkaline treatment should be used for composites using okro bast fibre so as to have highest strength that can make them good for indoor home and light weight applications.

5.4 CONTRIBUTIONS TO KNOWLEDGE

The research work established the following:

Okro bast fibre has been regarded as waste product, with this research work, value has being added to it, because it shows that they can be used to produce composites that can be of light weight and environmental friendly.

This work also shows that the optimal value was obtain at 15% alkali treatment which has highest strength that can make them good for indoor home and light weight applications such as ceiling board, office portioning board, particle board and for light weight shoe racks.

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APPENDICES

APPENDIX A: TENSILE STRENGTH AND MODULUS OF COMPOSITES

Table A1: Effects Of Alkaline Treatments On The Tensile Strength Of Treated And Untreated OBF/UPR Composites

S/N	NaOH Concentration/Filler Loading	Tensile strength (Mpa)
1	0% control sample	20.83
2	5% Untreated	17.64
3	10% Untreated	14.79
4	15% Untreated	15.09
5	5%NaOH 5%Fl	12.25
6	5%NaOH 10%Fl	19.82
7	5%NaOH 15%Fl	12.12
8	10%NaOH 5%Fl	17.76
9	10%NaOH 10%Fl	12.32
10	10%NaOH 15%Fl	8.74
11	15%NaOH 5%Fl	20.98
12	15%NaOH 10%Fl	17.96
13	15%NaOH1 5%Fl	22.21

Table A2: Effects Of Alkaline Treatments On The Tensile Strength Of Treated And Untreated OBF/UPR Composites

S/N	NaOH concentration/Filler loading	Young's Modulus (Mpa)
1	0%	259.09
2	5% Untreated	125.75
3	10% Untreated	117.6
4	15% Untreated	227.53
5	5% NaOH 5%Fl	137.5
6	5% NaOH 10%Fl	158.36
7	5% NaOH 15%Fl	202
8	10% NaOH 5%Fl	133.18
9	10% NaOH 10%Fl	236.81
10	10% NaOH 15%Fl	268.92
11	15% NaOH 5%Fl	123.38
12	15% NaOH 10%Fl	107.59
13	15% NaOH 15%Fl	287.36

APPENDICE B: PERCENTAGE ELONGATION OF THE COMPOSITES SAMPLES

Table B1: Effects Of Alkaline Treatments on Percentage Elongation of Treated and Untreated OBF/UPR composite

S/N	NaOH concentration/Filler loading	Elongation %
1	Control	7.5
2	5%	15
3	10%	12.5
4	15%	6.5
5	5%NaOH5%Fl	12.5
6	5%NaOH 10%Fl	6
7	5%%NaOH 15%Fl	6
8	10%NaOH 5%Fl	9.25
9	10%NaOH10%Fl	7.5
10	10%NaOH15%Fl	3.25
11	15%NaOH 5%FL	17
12	15%NaOH10%Fl	15.25
13	15% NaOH 15%Fl	6.25

APPENDICE C: FLEXURAL STRENGTH OF THE COMPOSITES SAMPLES

Table C1: Effects Of Alkaline Treatments On Flexural Strength Of Treated And Untreated OBF/UPR Composites.

S/N	NaOH Concentration /Filler loading	Flexural strength (Mpa)
1	0%	29.0
2	5%Untreated	17.3
3	10% Untreated	19.23
4	15% Untreated	29.27
5	5% NaOH 5%Fl	7.354
6	5% NaOH 10%Fl	7.95
7	5% NaOH 15%Fl	12.58
8	10% NaOH 5%Fl	18.45
9	10% NaOH 10%Fl	28.99
10	10% NaOH 15%Fl	44.72
11	15% NaOH 5%Fl	11.48
12	15% NaOH 10%Fl	34.39
13	15% NaOH 15%Fl	35.81

APPENDICE D: HARDNESS OF THE COMPOSITES SAMPLES

Table D1: Effects of Alkaline Treatments On Hardness Of Treated And Untreated OBF/UPR Composites

S/N	NaOH Concentration/Filler loading	Hardness (HRF)
1	0%	58
2	5%Untreated	52.8
3	10% Untreated	45.2
4	15% Untreated	49.2
5	5% NaOH 5%Fl	60.3
6	5% NaOH 10%Fl	47.8
7	5% NaOH 15%Fl	49.0
8	5% NaOH 5%Fl	53.6
9	10% NaOH 10%Fl	48.0
10	10% NaOH 15%Fl	52.5
11	15% NaOH 5%Fl	52.7
12	15% NaOH 10%Fl	53.5
13	15% NaOH 15%Fl	68.4

APPENDICE E: IMPACT STRENGTH OF THE COMPOSITES SAMPLES

Table E1: Effects Of Alkaline Treatments On The Impact Strength Of Treated And Untreated OBF/UPR Composites

S/N	NaOH Concentration/Filler loading	Impact strength (J/m)
1	0%	0.4
2	5%Untreated	0.43
3	10% Untreated	0.44
4	15% Untreated	0.52
5	5% NaOH 5%Fl	0.4
6	5% NaOH 10%Fl	0.32
7	5% NaOH 15%Fl	0.52
8	10% NaOH 5%Fl	0.37
9	10% NaOH 10%Fl	0.44
10	10% NaOH 15%Fl	0.38
11	15% NaOH 5%Fl	0.42
12	15% NaOH 10%Fl	0.56
13	15% NaOH 15%Fl	0.65

APPENDICE F: DENSITY OF THE COMPOSITES SAMPLES

Table F1: Effects Of Alkaline Treatments On The Density Of Treated And Untreated OBF/UPR Composites

S/N	NaOH Concentration/Filler loading	Density (g/cm^3)
1	0%	1.14
2	5%Untreated	1.11
3	10% Untreated	0.66
4	15% Untreated	0.12
5	5% NaOH 5%Fl	0.84
6	5% NaOH 10%Fl	0.55
7	5% NaOH 15%Fl	0.54
8	5% NaOH 5%Fl	0.74
9	10% NaOH 10%Fl	0.63
10	10% NaOH 15%Fl	0.13
11	15% NaOH 5%Fl	0.86
12	15% NaOH 10%Fl	0.76
13	15% NaOH 15%Fl	0.51

APPENDICE G: WATER ABSORPTION (%) OF THE COMPOSITES SAMPLES

Table G1: Effects Of Alkaline Treatments On Water Absorption (%) Of Treated And Untreated OBF/UPR Composites

No of days	0	2	4	6	8	10	12	14	16	18	20	22	24	26	28	30
	Samples water absorption (%)															
100%UPR	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
5%UOBF	0	0	0	0	0	0	0	0	0	0.6	0.6	1.3	1.4	1.4	1.5	1.5
10%UOBF	0	0	0	0	0	0	0	0	0	0.3	0.5	0.5	1	1.4	1.8	1.8
15%UOBF	0	0	0	0	0	0	0	0	0.1	0.7	1	1	1.5	1.5	2.2	2.2
5%N5%Fl	0	0	0	0.5	0.5	1	1.5	2	2.3	2.5	2.5	3	3	3.2	3.3	3.3
5%N10%Fl	0	0	0	0	0.6	1	1.3	1.3	1.8	2.5	2.5	3	3	3.3	3.5	3.5
5%N15%Fl	0	0	0	0	0.1	0.1	0.5	1	1	1.5	1.8	2.2	2.5	2.7	3.2	3.8
10%N5%Fl	0	0	0	0	0.05	0.5	0.5	1	1	1.6	2	2.5	2.5	3	3	3.8
10%N10%Fl	0	0	0	0	1.3	2	2	2.4	2.6	3	3	3.5	3.5	3.5	4	4
10%N15%Fl	0	0	0	0	0.1	0.1	0.15	1	1.5	1.8	2.3	2.8.	3	3.8	4	4.5
15%N5%Fl	0	0	0	0	0	0.6	1.3	1.9	2.6	3	3	3.5	3.5	3.9	4.2	4.5
15%N10%FL	0	0	0	0	0.6	1	1.3	1.3	2	2.6	3.1	3.5	3.5	3.5	4	4.5
15%N15%FL	0	0	0	0	0.7	0.7	1.4	2	2.8	2.8	3.2	3.5	4.2	4.8	4.8	5.2