CHAPTER ONE

INTRODUCTION

1.1. Background of Study

In recent years, polymers have replaced many of the conventional metals/materials in various applications. This is possible because of the advantages polymer offer over conventional materials (Lipsa and Sweta 2007). The most important advantages of using polymers are the ease of processing, productivity, and cost reduction. In most of these applications, the properties of polymers are modified using fillers and fibers to suit the high strength/high modulus requirements (Lipsa and Sweta 2007).

Composite material is made of the combination of two different materials to achieve certain properties different from each material on its own. One of the materials acts as a matrix, while the other acts as reinforcing material. The reinforcing material is imbeded in the matrix material to improve its mechanical and physical properties (Irene et al., 2010). Natural fibers as reinforcements have attracted the interest of many material scientists, researchers, and industries because of their specific advantages as compared to conventional or synthetic fibers. Because of their biodegradable nature, natural fibers have been increasingly adopted to replace synthetic fibers in the industrial application (Nierul and Ishak, 2012). The development of natural fiber reinforced composite became an attractive research lines due to the non recyclability, high density and health hazards of composites reinforced with synthetic fibers such as glass, carbon and aramid fibers (Corrales et al., 2007; Herrera and Valdez, 2005). The greatest problem of using such materials is how to conveniently dispose of them once they have come to the end of their useful life span (Bodros et al., 2007). Several natural fibers such as sisal (Chow et al., 2007), jute (Ahmed et al., 2007) flex (Baley et al., 2006), pinneapple (Tran, 2006) bamboo (Shih, 2007), kenaf (Elshekeil et al., 2012) bagasse (Cao et al., 2006; Vilay et al., 2008) fibers and so on have been studied as a reinforcement and fillers in polymer composite. However, these natural fibers are not problems free as reinforcements on composite. The performance and stability of fiber reinforced composite materials depends on the development of coherent interfacial bonding between fiber and matrix. In natural fiber reinforced composite there is a lack of good interfacial adhesion between the hydrophilic cellulose fibers and hydrophobic resins due to their inherent incompatibility (Mwaikambo and Ansele, 1999).

This incompatibility is caused by the hydrophilic nature of the fibers and the hydrophobic nature of many polymers used in this field (Elshekeil et al., 2012). The structural compositions of natural fibers, such as cellulose, hemicelluloses, lignin, pectin and waxy substances allow moisture absorption from the environment which leads to poor bonding with the matrix materials (Doan et al., 2006).

Certain chemical treatment on natural fibers is needed to enhance their performance as reinforcements in polymer composite materials. Natural fibers are amenable to modification as they bear hydroxyl group from cellulose and lignin. The hydroxyl group may be involved in the hydrogen bounding within the cellulose molecules thereby reducing the activity towards the matrix. Chemical modifications may activate the groups or can introduce new moieties that can effectively interlock with the matrix.

In this work *Ampelossisus cavicaulis* (Nwogbe), *Adenia lobata* (Usoro), *Morinda Morindoides* (Ogbuebo) and *Ampelocissus leonensis* (Okpowoko) were each treated with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride. Effects of the chemical treatments of the fibers were studied by obtaining their tensile strengths after the fiber treatments. The process factors (chemical concentration, contact time and type of chemicals) for the chemical treatments of the fibers were optimized using central composite design (CCD). The compounding conditions (rotation speed, mixing temperature and compounding time) of the fibers with high density polyethylene (HDPE) were optimized using Box Behnken design (BBD). The composite manufacturing process conditions (temperature, time and pressure) were optimized using central composite design (CCD).

1.2. Statements of the Problem

In the past, products used in engineering applications have typically been made from materials such as timber, metals, concrete, masonry and plastics. Although these materials have good performance characteristics, they are often very inefficient.

This research is intended to solve the following problems.

(a) To chemically modify the surface of natural fibers for purposes of enhancing a very good mechanical interlocking between the reinforcement and the matrix for composite production.

(b)To use the composite so produced as a material for engineering applications.

(c) To produce a viable alternative, with comparable mechanical properties, low weight, cost, density, high specific properties, good electrical resistance, thermal, acoustic insulating properties and higher resistance to fracture.

(d) To replace non biodegradable, high density and hazardous synthetic fibers with biodegradable, low density and environmentally friendly natural fibers.

By converting these fibers into useful products, their commercial values will increase. By efficient utilization of the fibers, environmental problems will be solved in an effective ways. At the same time, it will also contribute to the economic growth in the area by producing new materials that can fulfill global needs.

1.3. Significance of the work

In this research, four natural fibers were used as the main reinforcements with high density polyethylene (HDPE) as the matrix. This research will solve the problems of using natural fibers as reinforcements by investigating the optimum conditions for treating the fibers for efficient use in composite production. Fibers as reinforcements with high density polyethylene will upgrade their natural characteristics and can improve their commercial values.

These natural fibers are environmentally friendly compared to synthetic fibers. Synthetic fibers usually derived from petroleum as their main sources are non biodegradable, non renewable and have limited sources. Also burning of synthetic fibers may release toxic gases which are harmful to health due to green house effect. The development of natural fibers to replace the synthetic ones is important step to overcome all these issues. The advantages of using these natural fibers over the synthetic ones are low weight, recyclability, biodegradability and renewability.

This research gives the best condition to synthesize polymer composite to suit industrial needs.

1.4. Aim and Objectives

The aim of this research is to study the effects of chemical treatments of fibers on the physio-mechanical properties of reinforced high density polyethylene composites. To achieve this main goal the following specific objectives were set for the research:

- To characterize the raw fibers; (*Ampelocissus Cavicaulis*, *Adenia lobata*, *Morinda Morindoides* and *Ampelocissus leonensis)* and treat each with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride.
- To optimize the chemical treatment processes involving categoric and numeric factors using central composite design (CCD).
- To study effect of chemical type, pretreatment time and strength of chemicals on the physical and mechanical properties of each of the treated fibers.
- Optimize the compounding conditions (compounding time,temperature and rotation speed) using Box Behnken design (BBD) and study the effect of these conditions on the tensile strength of the composites
- To produce compression molded composite using HDPE with the four chemicals and Optimize the process conditions (molding temperature, pressure, molding time and fiber conditions) on the mechanical property of the composite using BBD.
- To study the effect of fiber loading, molding temperature, time, and pressure on the tensile strength of the composite and analyse the Physico-chemical properties (water absorption, swelling thickness, Density, and chemical resistance).
- To study the microstructural properties of the composites using SEM and FTIR

1.5. Scope of the Work

The scope of the work is as follows:

- The extraction of the natural fibers from the plants; Ogbuebo, Usoro, Nwogbe and Okpaowoko plants and their characterization.

- Chemical treatments of the fibers with sodium hydroxide, acetic anhydride, nitric acid and zinc chloride.
- Optimization of the chemical treatment process using CCD
- Effect of pretreatment time, chemical strength and chemical types on the tensile strength of the fibers.
- To study the properties of the modified fibers
- Optimization of the compounding conditions using BBD
- Effect of temperature, rotation speed, and time on the tensile strength of the composite
- Production of compression molded composite using untreated and treated fibers with HDPE as matrix
- Optimization of the process conditions using BBD for compression molding process
- Effect of fiber loading, temperature, pressure, and time on the tensile strength of the composite
- Determination of the mechanical, physical (water absorption, swelling thickness, chemical resistance) and microstructural (FTIR, and SEM) properties of the composites.

CHAPTER TWO

LITERATURE SURVEY

2.1. Overview

The term reinforced polymer is oft en used for composites with fibrous materials while filled polymer is the description used for polymers with particulate fillerszaq Examples of the reinforced polymers are fiber-glass reinforced polyesters and carbon fiber reinforced polyamides. A vast number of filled polymers are used today, including calcium carbonate filled poly (vinyl chloride), clay-filled polyethylene and carbon black-filled polybutadiene. The role of fibers is relatively obvious and simple; reinforcing the strength of weak polymers. The properties tend to be anisotropic. However, fillers have complex roles and many of them are interactive. Some are added to homogeneously increase modulus and strength, and others are for dimensional stability, viscosity control, increased hardness, pigmentation, and even for cost reduction. For example, calcium carbonate is often added to unsaturated polyester resin for cost reduction, rheology control, and dimensional stability, but in sacrifice of the strength.

Reinforced composites can be further classified into high-performance composites like filament wound plastics with glass fibers, or carbon fiber reinforced high temperature polymers made by hand lay-up method; and low-performance composites such as injection molded bulk molding compound (BMC) or composites made by the spray-up method. Extremity of the performance, whether it be the strength or use temperature, is the key factor for high performance composite. In general, high-performance composites tend to use high fiber content as the mechanical and physical properties of reinforcing fibers are generally much greater than the matrix resin. Filler is a term used for materials other than continuous fibers and, in general, implies that the reinforcement effect is rather low. Fillers may be particulate or fibrous but the size is usually less than a millimeter. For the reinforcement effect of fillers, the aspect ratio, i.e. the length-to-diameter ratio, is a critical important factor. Extender is the word used for filler which has almost no reinforcement effect and is used simply to reduce the amount of a plastic required.

A hierarchy exists in the terminology of reinforcing fibers. The thinnest component is a filament whose diameter is typically between several micrometers to several tens of micrometers. A few hundred to few thousand filaments are bundled together, which is assisted by a film former, to form a strand. Finally, many tens of strands make up a roving.

2.2**.** Composites

A composite is a heterogeneous substance consisting of two or more materials which does not lose the characteristics of each component. This combination of materials brings about new desirable properties. Naturally occurring composites include tendon, bone, bamboo, rock, and many other biological and geological materials.

2.2.1**.** Advantages of Composite

Composites have many engineering advantages over synthetic polymers and copolymers.

Some of these advantages are:

i. Reinforcement of the polymers result in increased tensile strength, flexural strength, compression strength, impact strength, rigidity and combination of these properties (Irene et. al., 2010).

ii. Increased size stability.

iii. Improved fire retardancy.

iv. Corrosion protection.

v. Improved electrical properties; reduction of dielectric constant.

vi. Coloring.

v. Improved processibility; controlled viscosities, good mixing, controlled orientation of fibers.

2.3. Applications of fiber reinforced composites

Biocomposite materials are already being applied in industries in areas such as geo-textiles, transportation, building/housing, packaging and consumer products (Schloesser, 2004; Suddell and Williams, 2005; Hu et al., 2007; Dweibb et al., 2006).

2.3.1. Transportation/Automotive

As a result of their good mechanical properties, large thin walled interior and exterior automobile parts have been made from glass fiber reinforced polymer (GFRP) composites. However, the technological, economical, and ecological benefits of natural fiber reinforced polymer (NFRP) composites over GFRPs provide many opportunities for these materials in the automotive industry. There are several useful publications on the current and potential uses of biocomposite in the automotive industry (Schloesser, 2004; Zah et al., 2007; Rowell, 1997). Even with increased research efforts in bio-based materials over the last several decades, the biocomposite industry still faces many challenges. Most of these in 1996, the use of natural fibers in the European automotive industry was reportedly around 4,300 mt. By 1999 that number had increased to over 24,000 mt and is projected to continue increasing to more than 100,000 mt by 2010 (Suddell and Williams, 2005). Initial applications were for interior door panels and trunk liners; however in recent years automotive applications for biocomposites have expanded to both interior and exterior applications. Interior composite parts using natural plant fibers typically include door panels, instrument panels, and glove boxes, whereas exterior applications are predominately floor panels (Schloesser, 2004).

Daimler Chrysler achieved the first global introduction of flax fiber reinforced polyester composites as engine and drive train covers for Travego busses and Mercedes passenger cars and has since increased the number of automotive parts containing natural fibers .The Ford Company, whose use of biocomposite materials in car parts dates back to the 1940s when the hemp fiber reinforced ‘soy based car’ was being advertised, recently unveiled a Model U concept vehicle that uses PLA for the canvas roof and carpet mats (Oksman, and Selin, 2004). Additionally, soy based materials are already in commercial use for composite parts on John Deere tractors (Petrovic et al., 2004).

2.3.2. Building/Housing

Lignocellulosic composite materials in the form of laminated lumber, veneers, fiberboards, particleboard and molded particulate reinforced plastics, are commonly used for structural and non-structural applications in the building and housing industries (Kozlowski and Wladyka-Przyblak, 2004).The United States alone consumes over one billion pounds of formaldehyde-based resins every year in the manufacture of particleboard, oriented strength board, and plywood (Chiou et al., 2005). The toxic nature of these resins has led to initiation of research into replacement adhesive systems derived from bio-based sources such as starches and plant oils. Additionally, in many cases these traditional wood based composites can be replaced by biocomposites derived from renewable plant fibers (Youngquist et al., 1994). Biocomposite panel binders may be synthetic or bio-based polymers. Youngquist et al. (1994) reviewed the use of nonwood plant fibers for building materials and panels. More recently, Kozlowski et al. (2004) reported that composite boards made from fibers and shives of non-wood lignocellulosic sources such as hemp, flax and kenaf are competitive with traditional wood based materials. Although in some cases the properties of the products are competitive with traditional wood based composites, further studies to improve the performance and investigate the durability and weatherability of these materials is clearly needed. Their uses include doors, door and window frames (fenestration products), floor, ceiling or wall boards, and even furniture.

In addition to being lighter than many conventional materials, biocomposites have also been shown to have excellent insulation properties due to the cellular structure of the fibers or fillers.

2.3.3. Packaging

The packaging industry is one of the most widely known uses for fibrous materials. Paper-based packaging is centered on the use of cellulose pulps, whether they be from wood or other agro-based resources (Laufenberg, 1997). It is clear then that molded containers with complex or simple shapes as well as lightweight packaging fillers all have the potential to be made from biocomposites (Rowell, 1997). NEC Corporation has developed a fully biodegradable flax fiber reinforced PLA composite which is anticipated to see a use in electronics packaging (Netravali, 2004). The use for biopolymers and biocomposites in the food packaging industry has also seen increased interest in recent years. Most notable was the introduction of PLA based biodegradable plastic bags and containers by Dow Cargill under the NatureWorks™ trademark (Oksman and Selin, 2004). For packaging materials, the ease of biodegradation of bio-based materials proves advantageous in reducing abundant waste material.

2.3.4. Consumer products

Through various molding techniques, biopolymers and natural fiber reinforced biocomposites can be processed to meet the needs of consumer products, often at lower costs and lower weights (Rowell, 1997). It is expected that virtually any injection molded part currently made by neat synthetic polymers and those with inorganic fillers could be replaced by biocomposites made in a similar fashion. Examples of such applications for consumer goods already being replaced by biopolymer and biocomposite materials include tool handles, clothing hangers, food trays, compostable planting cups, hygiene products, laptop computer casings, and furniture such as tables and chairs (Netravali and Chabba, 2003; Rowell, 1997).

2.4. Bio-composite Processing Technologies

The processing parameters for bio-composites depend largely on the type of polymer being used as a matrix material. Thermoplastic polymers, often with high melt viscosity, require the application of heat and pressure to ensure good melt flow and consolidation of the polymer around the fiber reinforcements. Thermosetting polymers, however, generally have low viscosity prior to curing which allows for easier fiber wet-out, therefore the application of heat and/or pressure to these composites is typically used to initiate cure reactions in the polymer. A known challenge for processing of natural fiber composites is to keep processing temperatures below 200°C as temperatures above this point have been shown to cause degradation of natural fibers (Ochi, 2006; Goda et al., 2006; Madsen et al., 2007). An increasing number of biopolymers that require lower processing temperatures such as plasticized starch, cellulose and soy-based polymers have, however, offered greater a selection of composite matrix materials such that the detrimental effects of high temperature composite processing can be avoided.

Composite processing techniques are also chosen based on the desired structure of the fiber reinforcements. Natural fibers come in the ‘raw’ unprocessed form or, after varying levels of mechanical and/or chemical processing, result in more refined and purified structures. Natural fiber reinforcements can range in length from short to long lengths and can be in a random or highly oriented structure. For short fiber reinforced composites where random orientation is desired, injection molding (IM), resin transfer molding (RTM) and various hot press techniques have all been shown to yield composites with good physical and mechanical properties. However, when long fiber reinforcements with high levels of orientation are desired composite processing techniques such as extrusion, filament winding and pre-preg formation followed by compression molding are typically implemented. As a result of the wide variety materials and desirable performance characteristics in bio-composites, highly tailored processing techniques are not uncommon for the production of specific products.

2.5. Manufacturing of Composites

A systematic study of the process variables for composites based on different natural fibers like flax, jute, sisal, ramie, and pineapple has been made with a view to determine the most suitable processing conditions for such composites by some researchers. Some major methods of molding are rotational molding, compression molding, injection molding and extrusion. Alternative processing methods of natural fiber-reinforced composites are an important advancement necessary for their increased use.

2.5.1 Compression molding

Compression molding is a well known technique to develop variety of composite products. It is a closed molding process with high pressure application. In this method, as shown in the figure 2.1, two matched metal molds are used to fabricate composite product. In compression molder, base plate is stationary while upper plate is movable. Reinforcement and matrix are placed in the metallic mold and the whole assembly is kept in between the compression molder. Heat and pressure is applied as per the requirement of composite for a definite period of time. The material placed in between the molding plates flows due to application of pressure and heat and acquires the shape of the mold cavity with high dimensional accuracy which depends upon mold design. Curing of the composite may be carried out either at room temperature or at some elevated temperature. After curing, mold is opened and composite product is removed for further processing. In principle, a compression molding machine is a kind of press which is oriented vertically with two molding halves (top and bottom halves).

Generally, hydraulic mechanism is used for pressure application in compression molding. All the three dimensions of the model (pressure, temperature and time of application) are critical and have to be optimized effectively to achieve tailored composite product as every dimension of the model is equally important to other one. If applied pressure is not sufficient, it will lead to poor interfacial adhesion of fiber and matrix. If pressure is too high, it may cause fiber breakage, expulsion of enough resin from the composite system. If temperature is too high, properties of fibers and matrix may get changed. If temperature is low than desired, fibers may not get properly wetted due to high viscosity of polymers especially for thermoplastics. If time of application of these factors (pressure and temperature) is not sufficient (high or low), it may cause any of defects associated with insufficient pressure or temperature. The other manufacturing factors such as mold wall heating, closing rate of two matched plates of the plates and de-molding time also affect the production process.

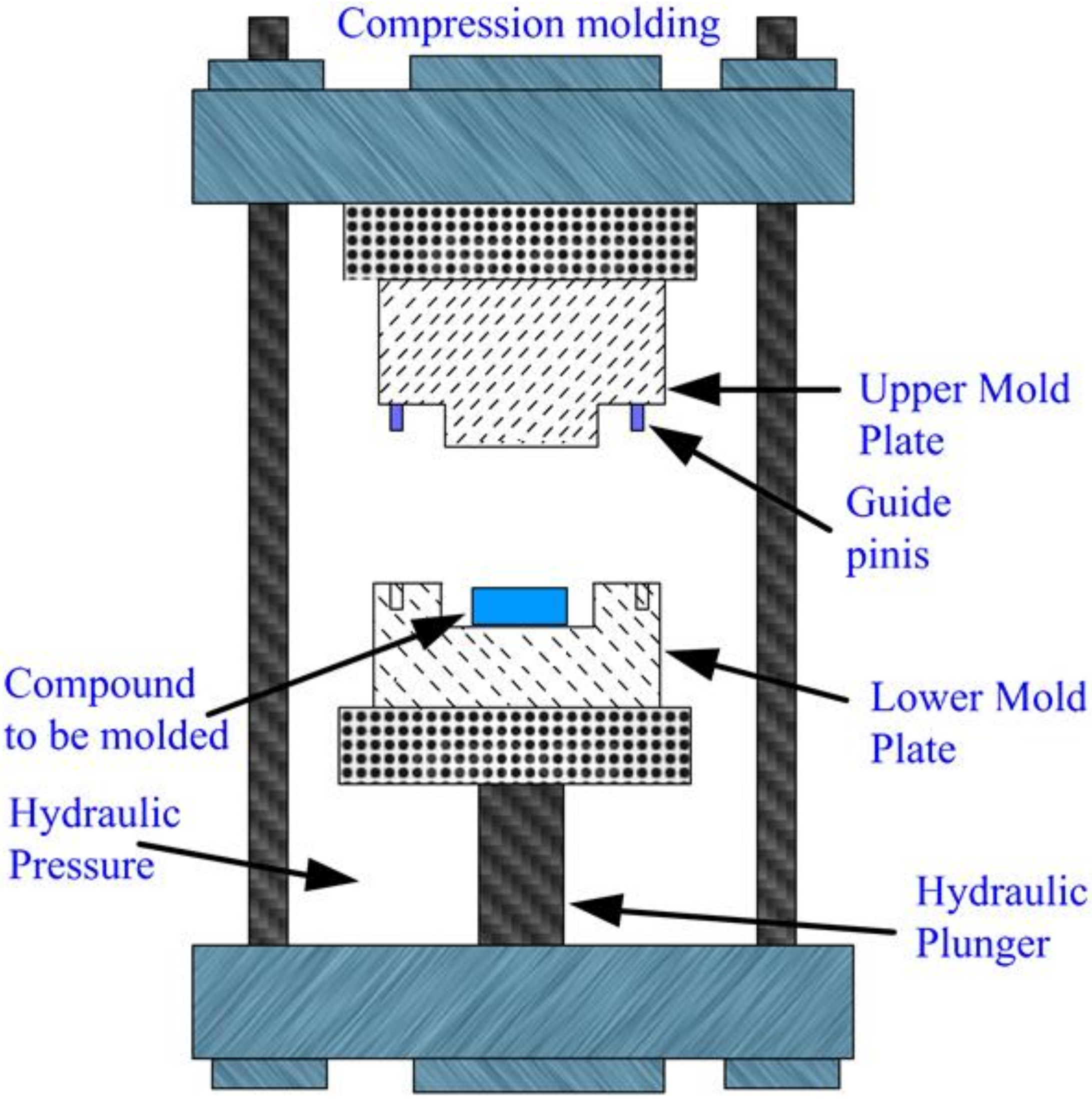


Fig. 2.1. Compression molding method (Inderdeep, 2012)

Advantages of the compression molding process

1. Production rate is high as the mold cycle time is in few minutes.
2. Good surface finish with different texture and styling can be achieved.
3. High part uniformity is achieved with compression molding process.
4. Good flexibility in part design is possible.
5. Extra features like inserts, bosses and attachment can be molded in during the processing.
6. Raw material wastage is minimum.
7. Maintenance cost is low.
8. Residual stresses are absent or negligible in the molded component.

9. Twisting and shrinkage in product is reduced therefore dimensional accuracy is good.

Disadvantages of compression molding process

1. Due to expensive machinery and parts, the initial capital investment associated with compression molding is high.
2. The process is suitable for high production volume. It is not economical for making a small number of parts or for prototyping applications.
3. It is a labour intensive process.
4. Sometimes secondary processing (trimming, machining) of product is required after compression molding.
5. Sometimes uneven parting lines are there.
6. There is limitation on mold depth.

2.5.2 Injection molding

Injection molding is a manufacturing process for producing parts by injecting material into a mould. Injection molding can be performed with a host of materials, including metals, glasses, elastomers, confections, and most commonly thermoplastic and thermosetting polymers. Material for the part is fed into a heated barrel, mixed, and forced into a mould cavity, where it cools and hardens to the configuration of the cavity (Merrill, 1955).After a product is designed, usually by an industrial designer or an engineer, moulds are made by a mold maker (or toolmaker) from metal, usually either steel or aluminum, and precision-machined to form the features of the desired part. Injection molding is widely used for manufacturing a variety of parts, from the smallest components to entire body panels of cars. Advances in 3D printing technology, using photopolymers which do not melt during the injection molding of some lower temperature thermoplastics can be used for some simple injection molds.

Parts to be injection moulded must be very carefully designed to facilitate the moulding process; the material used for the part, the desired shape and features of the part, the material of the mould, and the properties of the moulding machine must all be taken into account. The versatility of injection moulding is facilitated by this breadth of design considerations and possibilities (Bryce, 1996).

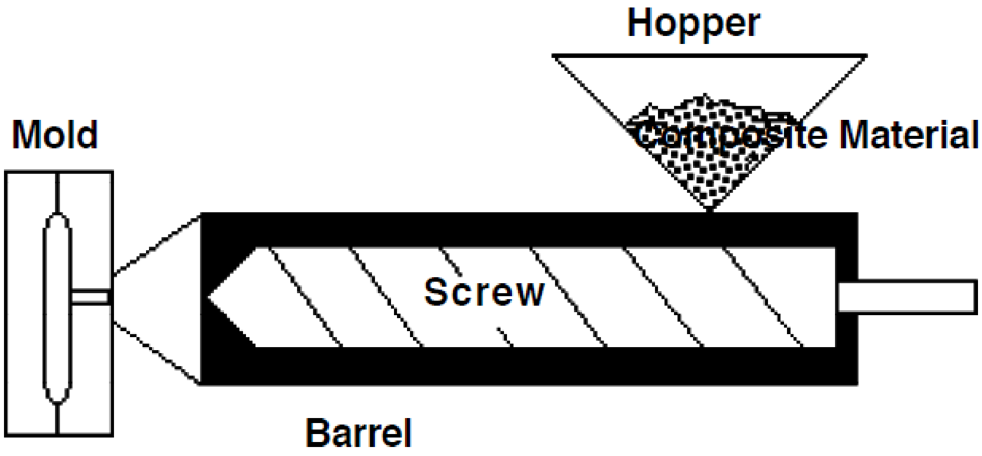


Fig. 2.2. Schematic diagram of an injection molding machine

Injection molding uses a ram or screw-type plunger to force molten plastic material into a mold cavity; this solidifies into a shape that has conformed to the contour of the mold. It is most commonly used to process both thermoplastic and thermosetting polymers, with the former being considerably more prolific in terms of annual material volumes processed. Thermoplastics are prevalent due to characteristics which make them highly suitable for injection molding, such as the ease with which they may be recycled, their versatility allowing them to be used in a wide variety of applications, and their ability to soften and flow upon heating. Thermoplastics also have an element of safety over thermosets; if a thermosetting polymer is not ejected from the injection barrel in a timely manner, chemical crosslinking may occur causing the screw and check valves to seize and potentially damaging the injection molding machine.

Injection molding consists of high pressure injection of the raw material into a mold which forms the polymer into the desired shape. Molds can be of a single cavity or multiple cavities. In multiple cavity molds, each cavity can be identical and form the same parts or can be unique and form multiple different geometries during a single cycle. Molds are generally made from tool steels, but stainless steels and aluminum molds are suitable for certain applications. Aluminum molds typically are ill-suited for high volume production or parts with narrow dimensional tolerances, as they have inferior mechanical properties and are more prone to wear, damage, and deformation during the injection and clamping cycles; but are cost-effective in low-volume applications as mold fabrication costs and time are considerably reduced. Many steel molds are designed to process well over a million parts during their lifetime and can cost hundreds of thousands of dollars to fabricate.

When thermoplastics are molded, typically pelletized raw material is fed through a hopper into a heated barrel with a reciprocating screw. Upon entrance to the barrel the thermal energy increases and the Van der Waals forces that resist relative flow of individual chains are weakened as a result of increased space between molecules at higher thermal energy states. This process reduces its viscosity, which enables the polymer to flow with the driving force of the injection unit. The screw delivers the raw material forward, mixes and homogenizes the thermal and viscous distributions of the polymer, and reduces the required heating time by mechanically shearing the material and adding a significant amount of frictional heating to the polymer. The material feeds forward through a check valve and collects at the front of the screw into a volume known as a shot. A shot is the volume of material that is used to fill the mold cavity, compensate for shrinkage, and provide a cushion (approximately 10% of the total shot volume, which remains in the barrel and prevents the screw from bottoming out) to transfer pressure from the screw to the mold cavity. When enough material has gathered, the material is forced at high pressure and velocity into the part forming cavity. To prevent spikes in pressure, the process normally uses a transfer position corresponding to a 95–98% full cavity where the screw shifts from a constant velocity to a constant pressure control. Often injection times are well under 1 second. Once the screw reaches the transfer position the packing pressure is applied, which completes mold filling and compensates for thermal shrinkage, which is quite high for thermoplastics relative to many other materials. The packing pressure is applied until the gate (cavity entrance) solidifies. Due to its small size, the gate is normally the first place to solidify through its entire thickness. Once the gate solidifies, no more material can enter the cavity; accordingly, the screw reciprocates and acquires material for the next cycle while the material within the mold cools so that it can be ejected and be dimensionally stable. This cooling duration is dramatically reduced by the use of cooling lines circulating water or oil from a thermolator. Once the required temperature has been achieved, the mold opens and an array of pins, sleeves, strippers, etc. are driven forward to demold the article. Then, the mold closes and the process is repeated.

For thermosets, typically two different chemical components are injected into the barrel. These components immediately begin irreversible chemical reactions which eventually crosslinks the material into a single connected network of molecules. As the chemical reaction occurs, the two fluid components permanently transform into a viscoelastic solid. Solidification in the injection barrel and screw can be problematic and have financial repercussions; therefore, minimizing the thermoset curing within the barrel is vital. This typically means that the residence time and temperature of the chemical precursors are minimized in the injection unit. The residence time can be reduced by minimizing the barrel's volume capacity and by maximizing the cycle times. These factors have led to the use of a thermally isolated, cold injection unit that injects the reacting chemicals into a thermally isolated hot mold, which increases the rate of chemical reactions and results in shorter time required to achieve a solidified thermoset component. After the part has solidified, valves close to isolate the injection system and chemical precursors, and the mold opens to eject the molded parts. Then, the mold closes and the process repeats.

Pre-molded or machined components can be inserted into the cavity while the mold is open, allowing the material injected in the next cycle to form and solidify around them. This process is often used to create plastic parts with protruding metal screws, allowing them to be fastened and unfastened repeatedly. This technique can also be used for In-mould labelling and film lids may also be attached to molded plastic containers.

A parting line, sprue, gate marks, and ejector pin marks are usually present on the final part. None of these features are typically desired, but are unavoidable due to the nature of the process. Gate marks occur at the gate which joins the melt-delivery channels (sprue and runner) to the part forming cavity. Parting line and ejector pin marks result from minute misalignments, wear, gaseous vents, clearances for adjacent parts in relative motion, and/or dimensional differences of the mating surfaces contacting the injected polymer. Dimensional differences can be attributed to non-uniform, pressure-induced deformation during injection, machining tolerances, and non-uniform thermal expansion and contraction of mold components, which experience rapid cycling during the injection, packing, cooling, and ejection phases of the process. Mold components are often designed with materials of various coefficients of thermal expansion. These factors cannot be simultaneously accounted for without astronomical increases in the cost of design, fabrication, processing, and quality monitoring. The skillful mold and part designer will position these aesthetic detriments in hidden areas if feasible.

2.5.3. Rotational molding

Rotational molding is a process for manufacturing hollow plastic products. Rotational molding involves power mixing, melting, sintering and melt solidification. Various aspects of the rotational molding process have been studied by several researchers (James and Marcel, 1979). Fundamental research on rotational molding has been directed to reduce the molding cycle time and to optimize the mechanical properties of final parts. Polyethylene accounts for more than 80% of the total production (Bellehumeur et al., 1998). Rotational molding has particular advantages in terms of relatively low levels of residual stresses and inexpensive molds. Rotational molding also has few competitors for the production of large (>2 m^3) hollow objects in one piece. Currently, the rotational molding industry is in its exciting development.

Important new market sectors are opening up as rotational molders are able to deliver high quality parts at competitive prices.

2.5.4. Hand lay-up molding

Hand lay-up molding is the method of laying down fabrics made of reinforcement and painting with the matrix resin layer by layer until the desired thickness is obtained. This is the most time and labor consuming composite processing method, but majority of aerospace composite products are made by this method in combination with the autoclave method. Due to the hand assembry involved in the lay-up procedure, one can align long fibers with controlled orientational quality. Another advantage of this method is the ability to accommodate irregular-shaped products. Such advantages are utilized in low performance composites including fiber-glass boat and bath tub manufacturing.

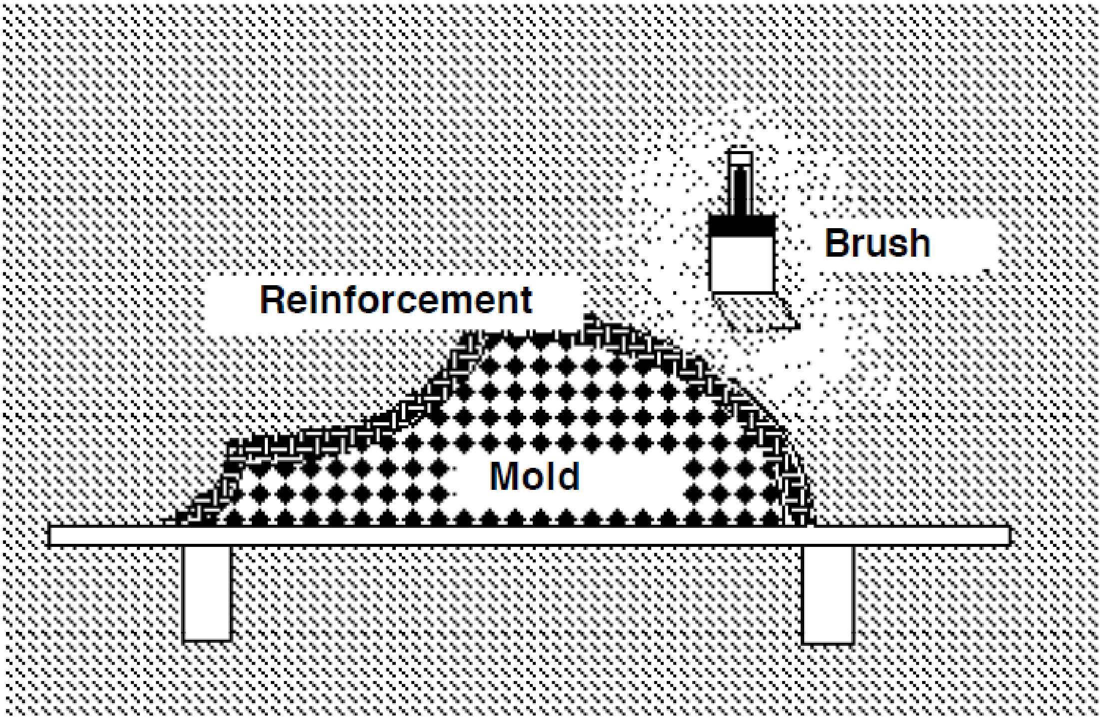


Fig. 2.3. Schematic diagram of hand lay-up operation.

2.5.5. Spray up molding

Spray-up molding is much less labor intensive than the hand lay-up method by utilizing a spray gun and a fiber cutter. However, only short fiber reinforced composites can be made. A continuous fiber is fed into the cutter and chopped. The chopped fiber is sprayed upon a mold with the stream of resin mist and catalyst delivered through separate nozzles. The sprayed mixture of fiber and resin soon cures on the mold at room temperature and the product is produced. Because of the spraying operation, large and complex-shaped objects can be easily made.

2.5.6. Reaction injection molding

Reaction injection molding (RIM*)* is one of the newest processing methods. Instead of using already polymerized materials as matrices, highly reactive monomeric or oligomeric ingredients are placed in two tanks which are then quickly mixed by impingement, and injected into the mold cavity. As soon as the two materials are mixed, chemical reaction begins to form a polymeric matrix, which completes typically within 5-30 seconds. Thus, the major portion of the RIM machine is a high pressure pump and a metering system. Again, with high intensive shear, only short fibers and fillers can be used as reinforcements. However, RIM utilizes low viscosity chemicals and this allows the preplacement of continuous fiber-woven fabrics in the mold in the same manner as resin transfer molding. Distinction is made between these two methods based on the preparation of the resin precursor. When the resin formulation is already made, the method is called resin transfer molding while if the resin is prepared in-situ by an impingement or static mixer, the method is termed RIM.

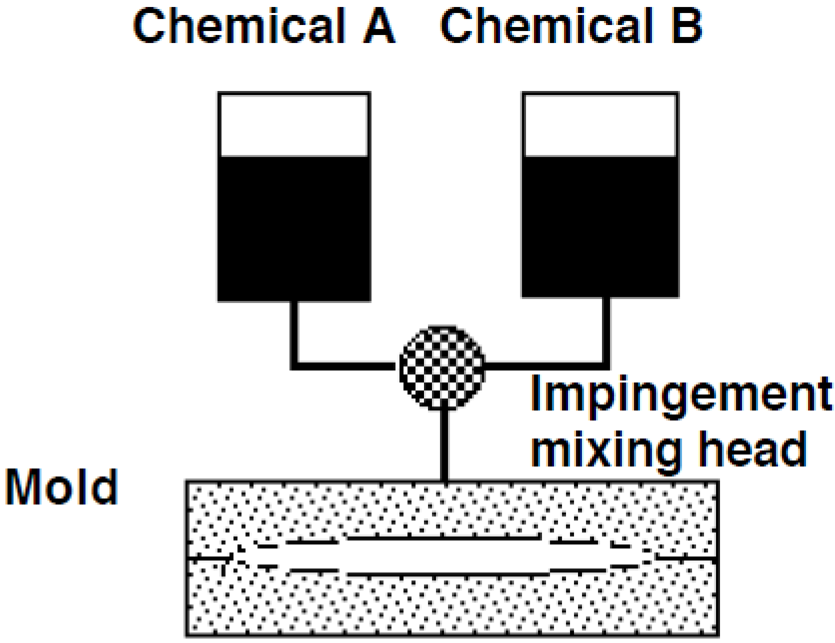


Fig.2.4. Schematic diagram of a reaction injection molding machine.

2.5.7. Pultrusion

Pultrusion is used only for polymer composite processing. A bundle of fiber rovings is passed through a wet resin bath, squeezed into a desired shape, passed through a heated die, and cured into a final composite. The solidified composite, typically reinforced unidirectionally with continuous fibers or sometimes bidirectionally, is pulled by a puller to continuously feed the uncured portion of the wet fibers into the hot die, thus the name, "pultrusion". This is one of very few continuous processing methods for continuous fiber reinforced composites. Only constant cross-sectional products can be made; the shape of the cross-section does not necessarily have to be the same, however .

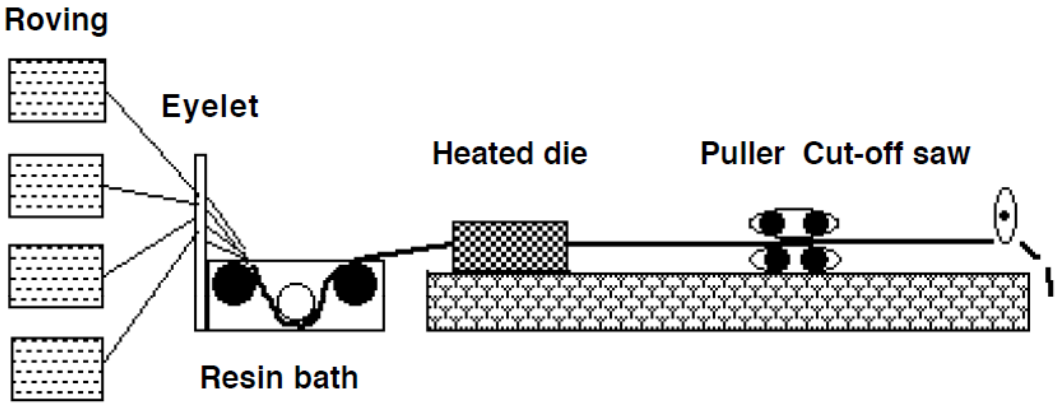


Fig.2.5. Schematic diagram of a pultrusion machine.

2.5.8 Filament winding

Filament winding is also a unique processing method for polymer composite processing with a continuous reinforcing fiber. Resin-wet rovings are wound with a certain pattern around a mandrel. The wound mandrel is then placed into an oven and cured to a solid composite.

Due to the controlled tension, squeezing action and controlled winding pattern, the fiber content can be very high to produce composites with one of the highest mechanical properties. The winding process is time consuming and is the cause of low productivity. However, due to its very high mechanical properties with automated operation, it is actively evaluated by aerospace industries.

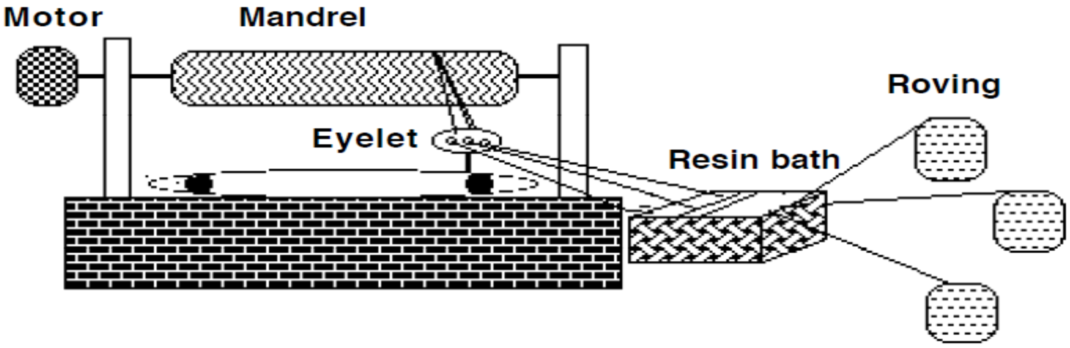


Fig. 2.6. Schematic diagram of a filament winding machine.

2.6. Biopolymers

The plastics industry has been continually thriving due to the seemingly endless applications for these materials in industries such as packaging, automotives, building products, furniture, and consumer goods. Traditionally, the majority of these materials are derived from petroleum feed-stocks because of their high mechanical properties and durability. Biocomposites made from traditional synthetic and petroleum derived polymers have been developed extensively in the last few decades. Thermoplastic polymers such as polypropylene (PP) and polyethylene (PE) as well as thermosetting polyester, epoxy and polyurethane polymers are the most common matrix materials used in natural fiber reinforced composites. The incorporation of natural fiber reinforcements in these resins clearly makes then better from an environmental perspective to traditional glass-fiber reinforced polymer (GFRP) composites; however, they do little to solve the problems of diminishing petroleum resources and landfill space.

The petroleum supply is not renewable and has also been shown to be unreliable in recent years making the cost of these materials rise appreciably. Additionally, petroleum derived plastics do not degrade which means they must either be incinerated, causing harmful emissions, or disposed of in diminishing landfill space.

Research interests in biopolymers, broadly defined as polymers which are biodegradable and/or derived from renewable resources (Mohanty et al., 2005), have increased worldwide in the last several decades. Biodegradable petroleum derived plastics are some scientist’s response to the concern regarding the accumulation of non-degradable plastics waste. These polymers have offered scientists a possible alternative to the waste disposal problems typically associated with traditional petroleum derived polymers often times without having to sacrifice the exceptional properties of non-degradable plastics. However, this does not address the issue of rising costs of plastics due to fossil fuel shortages. The instability of the petroleum supply and environmental issues related to its products has led many researchers to look into alternate feed stocks for plastics, the majority of which are renewable and agricultural based. Biopolymers derived from renewable resources offer a similar solution to waste disposal, but have the added advantage of reducing dependence on the diminishing petroleum resources.

Clearly, both can be seen as advantageous in advancing and sustaining the technology of environmentally friendly materials. For this study, it was of interest to develop a biocomposite that optimized properties of environmental friendliness and high performance, therefore, only biopolymers with good properties and biodegradable capabilities were investigated for use as composite matrix materials. Mohanty and co-workers (Mohanty et al., 2000) reviewed the state of biopolymer technologies and presented structures, synthesis and properties of many of the common biodegradable polymers from synthetic and renewable resources. Since the time of this review, however, several notable research efforts have been reported on new biopolymer materials for composites. Figure 2.7 shows a broad breakdown of the emerging technologies in the field of biopolymers. An overview of the previous review as well as current findings in biopolymer technology is presented below.

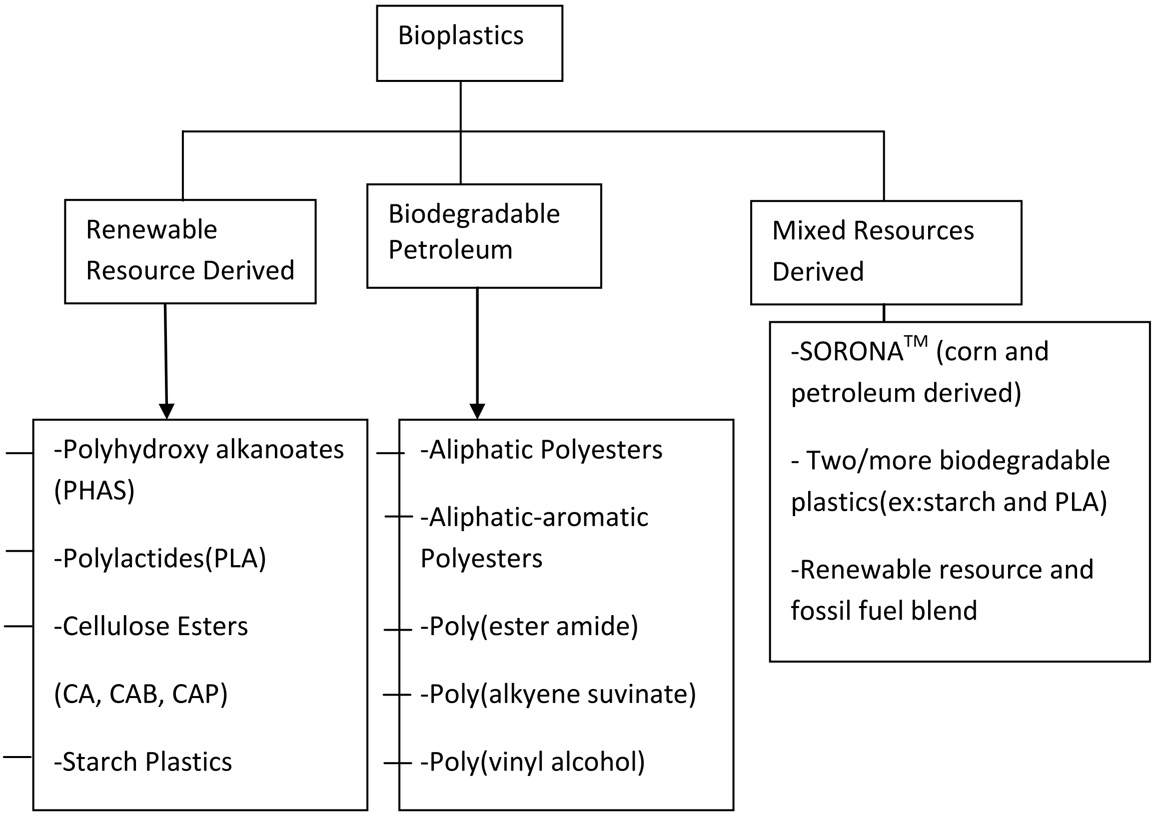


Fig. 2.7. Broad classification of materials termed “bioplastics” (Mohanty et al., 2000).

2.7. Characteristics of Thermoplastic Polymers

Thermoplastic polymers constitute an important class of materials with a wide variety of applications (Kosaka et al., 2007). They are capable of being re-melted without any change in chemical structure or properties. Polyethylene, polypropylene, polystyrene and polyvinyl chloride are the most common thermoplastic polymers and are frequently called commodity polymers. As long as processing does not mechanically damage the thermoplastic polymer structure, these polymers are considered recyclable. Besides the use of “pure” polymer for structural purposes, it is also used as a matrix for fiber-reinforced composites. These composites are mostly based on the traditional reinforcement fibers such as glass fibers. However, natural fibers can also be used as reinforcement. Polymers have a different affinity towards the fiber owing to the difference in their chemical structure. In the search for the most suitable thermoplastic matrix for a flax fiber-reinforced composite, density and temperature related properties seems to be limiting criteria (Mohanty et al., 2001). Low-density polyethylene (LDPE), high-density polyethylene (HDPE) and linear low density polyethylene (LLDPE) are normally used as major matrix for reinforcement. These thermoplastic polymers are widely used and have a melting point compatible with natural fibers. Their low melting points also allow processing below the degradation temperature of the fibers. Table 2.1 lists typical properties of three major thermoplastics

Table 2.1. Typical properties of three major thermoplastics (www.azom.com).

Polymer type	Density (g/cm^3)	Degree of crystallinity	Glass transition temperat. (^{0}C)	Crystal melting temperat. (^{0}C)	Tensile streng. (Mpa)	Elong. at break (%)	Flexural modulus (Gpa)
High-density Polyethylene (HDPE)	0.95-0.97	High	- 120	137	20-30	10-1,000	1-1.5
Low-density Polyethylene (LDPE)	0.92-0.93	Moderate	- 120	110	8-20	100-650	0.25-0.35
Linear low density Polyethylene (LLDPE)	0.91-0.94	High	- 74	122-124	2.0	100-500	-.35

2.7.1. High-density polyethylene (HDPE)

High-density polyethylene (HDPE), also known as linear polyethylene or low-pressure polyethylene is the preferred polyethylene for chemical containers of all sizes primarily due to its exceptional environment stress crack resistance (Crawford and Throne 2000).It has excellent stiffness from room temperature to the boiling point of water. Even though HDPE is frequently called linear polyethylene, it still has some short chain branching. Nevertheless, its linear nature and its high backbone mobility allow it to crystallize from 75% to 90% of theoretical. The crystalline structure causes the product to have a milky, translucent appearance. Since the crystallite is more ordered and more tightly packed than the amorphous phase, the density of HDPE is typically around 960 kg/m^3 approaching the theoretical value of 1000 kg/m^3. Many HDPEs are formulated for extrusion and blow molding applications (Crawford and Throne 2000).

2.7.2. Linear low-density polyethylene (LLDPE)

LLDPE has side chains similar to those of LDPE but with proper catalysts and coreactive agents, the chains are dramatically reduced in length (Crawford and Throne, 2000). LLDPE has a density range of 910 kg/m^3 to about 940 kg/m^3, and is 65% to 75% crystalline at room temperature. Compared with LDPE, the ‘linear low’ materials have found rapid acceptance because of their high toughness (at low, normal and high temperatures), improved stiffness, chemical resistance, tensile strength, elongation at break and puncture resistance. However, it has somewhat poorer impact strength when compared with LDPE and HDPE. Suppliers have emphasized more specifically the improved resistance to environmental stress cracking (Brydson, 1989).

In Lee and Joo’s study (1999), a thermoplastic LLDPE resin was used as the matrix for fiber composites. Its low processing temperature (less than 130ºC) made composite fabrication possible without partial melting or annealing of the fibers. The high toughness of LLDPE yielded a good impact-resistant composite and had advantages of thermoplastic composite processing, such as short processing time, unlimited storage time and solvent free processing.

2.7.3. Low-density polyethylene (LDPE)

Low-density polyethylene (LDPE), also referred to as high-pressure polyethylene or branched polyethylene, has extensive side chains, up to about 100 ethylene units in length. The long branches tend to inhibit molecular organization during cooling. As a result, LDPEs typically have relatively low densities of 910 kg/m^3 to 925 kg/m^3 and relatively low crystallinity of 45% to 66%. LDPEs are relatively soft polyethylene with flexural modulus ranging between 0.24 and 0.35 GPa. Owing to the high number of tertiary hydrogen, LDPE does not have good environmental stress crack resistance (ESCR). Nevertheless, LDPEs mold well at low temperatures that accurately replicate mold surfaces (Crawford and Throne, 2000).

2.7.4. Biodegradable petroleum based polymers

There are currently a great number of commercially available biodegradable polymers derived from petroleum resources (Mohanty et al., 2000). In particular, aromatic polyesters such as PCL and PBS, aliphatic-aromatic polyesters such as Eastman's Eastar Bio® and BASF's Ecoflex®, and polyester amides such as Bayer's BAK 1095 and 2195, are the most recognized biodegradable polymers (Mohanty et al., 2005). These biodegradable polymers exhibit mechanical properties similar to those of traditional petroleum derived polymers which can be beneficial when attempting to use them in place of conventional polymers. It is important to consider the processing parameters for these polymers when selecting them for a specific application, as they can vary greatly. Poly (ε-caprolactone), PCL, is a tough and semi-rigid semi-crystalline thermoplastic polymer with a modulus between that of low- and high-density polyethelylene (Mohanty et al., 2000). PCL has a glass transition of -60°C, a low melting point around 60°C, and low viscosity making it easily melt processed. As a result of its low melt temperature it is typically blended with other polymers. Additionally, PCL possesses good resistance to water, oil, solvents, and chlorine.

Commercially available polybutylene succinates (PBS), have been developed by Showa Highpolymer under the trade name Bionelle®. These materials are reportedly high molecular weight white semi-crystalline thermoplastic polymers with a glass transition temperature between -45 and -10°C and a melting point of about 90 to 120°C (Mohanty, 2000). These polymers have a density around 1.25 g/cm^3 and tensile strength and stiffness properties between that of PE and PP, and LDPE and HDPE, respectively.

Polyester amides were first introduced in 1995 by the Bayer Corporation under the name BAK 1095, two years later the injection molding grade polyester amide, BAK 2195, was introduced. These polymers are noted for their high toughness and tensile strain at break with mechanical and thermal properties similar to those of polyethylene. The BAK 1095 and 2195 have melting points of 125 and 175°C, respectively (Mohanty et al., 2005). These polymers are unique in that they break down into water, carbon dioxide, and biomass under aerobic conditions at rates comparable to other combustable materials.

2.7.5. Bio-resource derived polymers

It has been recognized that plastics derived from biobased renewable resources offer both economic and environmental advantages for the United States. Firstly, by utilizing renewable resources such as agricultural crops the country's dependence on foreign petroleum supplies will be reduced. Additionally, the use of natural materials is in line with increasing support for environmentally friendly alternatives in science and technology. In the past decade several government based initiatives have offered economic incentive to consider bio-based alternatives to petroleum based materials (Mohanty et al., 2000).

Some of the most developed technologies in bio-based plastics are aromatic polyesters such as PHAs from bacterial fermentation and PLA from corn as well as polymers derived from renewable resources such as cellulose (cellulose acetates), starches, (starch esters), and proteins or oils from plants such as soybeans. Demand for these materials is expected to increase by more than 20% a year along with improved economics as production and sales increase (Mohanty et al., 2005).

2.8. Fibers

Fibers are class of hair-like materials that are in discrete elongated pieces, similar to pieces of thread (Muhammad Jannah, 2008). They can be spun into filaments, thread or rope. They can be used as a component of composite materials. Fiber can be classified into two main groups, which are man-made fiber and natural fiber. In general, natural fibers can be sub-divided as to their origin such as plants, animals, or minerals; while man-made fibers can be subdivided to synthetic and natural polymers. The first fibers used by man were natural fibers such as cotton, wool, silk, flax, hemp and sisal. The first man-made fiber was probably glass (Muhammad Jannah, 2008).

Both natural and synthetic fibers (commonly known as man-made fibers) are now available and always being used as fillers in making good properties of composites. The major fibers used till now can be classified into the groups given in Figure 2.8. Reinforcing fibers in a single-layer composite may be short or long compared to its overall dimensions. The long fibers and short fibers are called continuous fibers and discontinuous fibers, respectively (Agarwal and Broutman, 1990). The continuous fibers in a single-layer composite may be all aligned in one direction to form a unidirectional composite. The unidirectional composites are very strong in the fiber direction but are generally weak in the direction perpendicular to the fibers. The continuous reinforcement in a single layer may also be provided in a second direction to provide more balanced properties. The bidirectional reinforcement may be provided in a single layer in mutually perpendicular directions as in a woven fabric. The orientation of short or discontinuous fibers cannot be easily controlled in a composite material. In most cases the fibers are assumed to be randomly oriented in the composite (Agarwal and Broutman, 1990).

Alternatively, short fibers, sometimes referred to as chopped fiber may be converted to a lightly bonded form or mat that can be later impregnated with resin to fabricate single-layer composites. Chopped fibers may also be blended with resins to make a reinforced molding compound. These fibers tend to become oriented parallel to the direction of material flow during a compression or injection molding operation and thus get a preferential orientation. Figure 2.9 represents an accepted classification of single layer alignments.

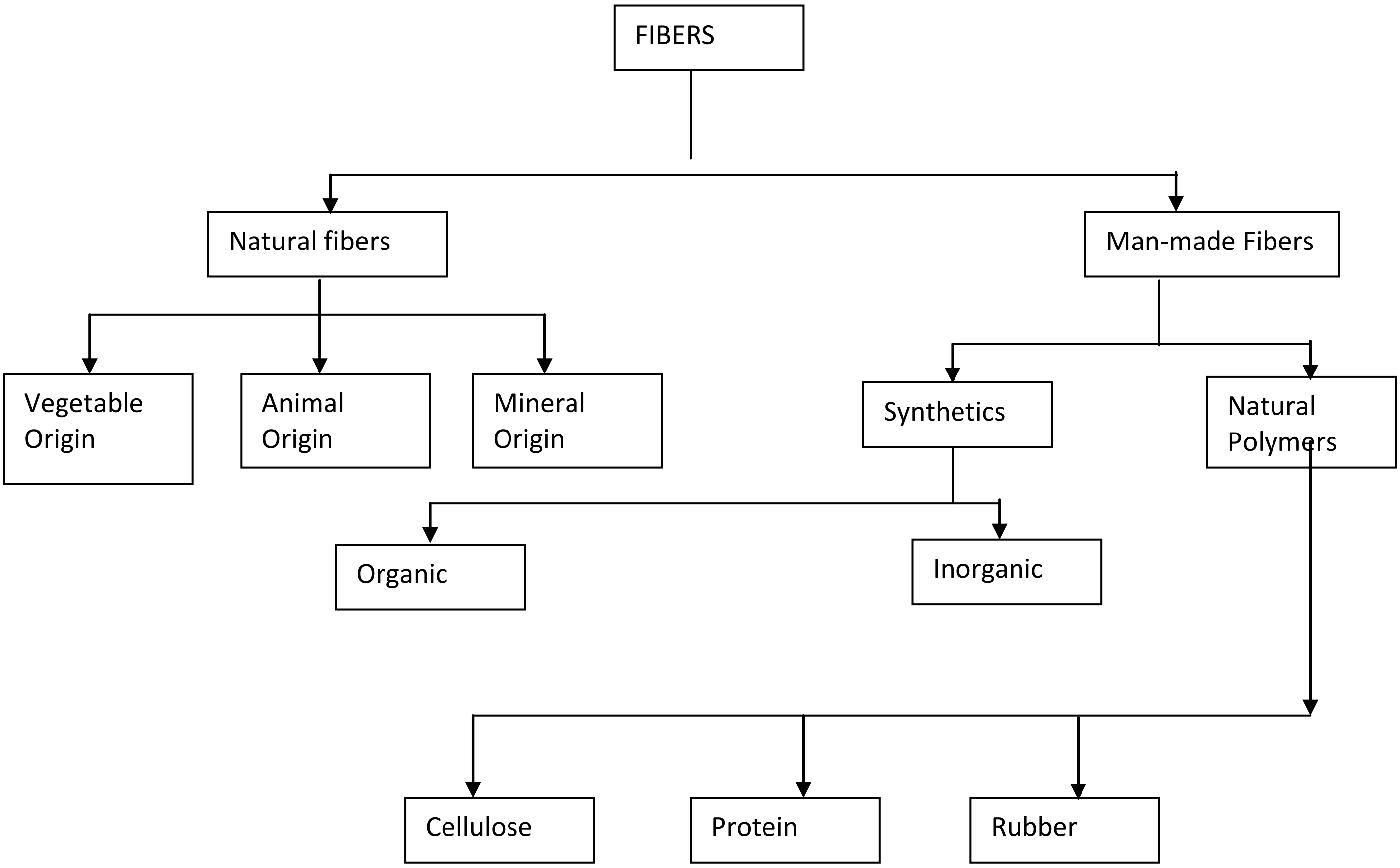


Fig. 2.8.Classification of fibers (Muhammad Jannah, 2008)

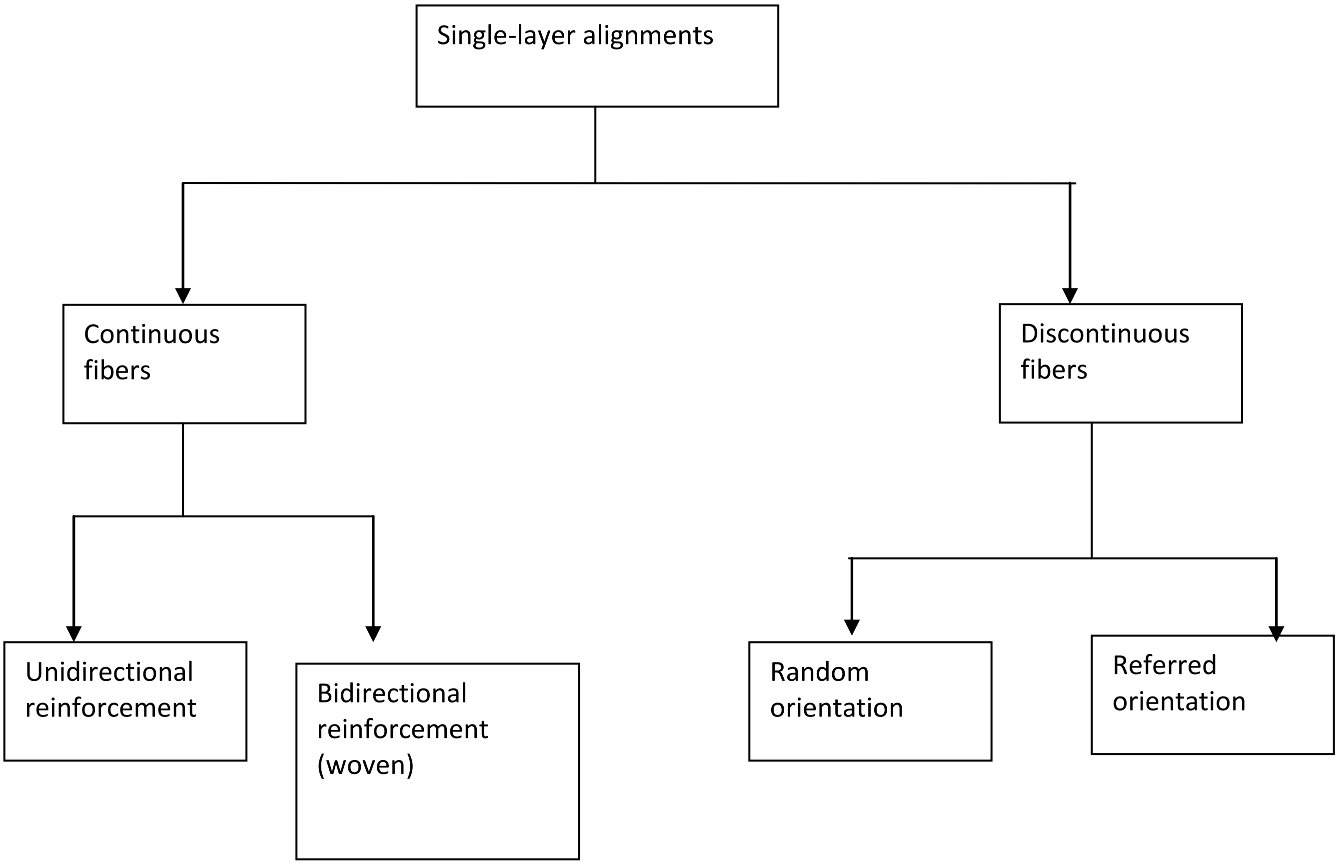


Fig. 2.9: Single – layer alignments (Agawal and Broutman, 1990)

2.8.1 Synthetic fibers

Synthetic fibers are the result of extensive research by scientists to improve upon naturally occurring animal and plant fibers used in making cloth and rope (Muhammad Jannah, 2008). Figure 2.10 shows the classification of synthetic fibers. A large number of synthetic fibers with a variety of properties have been produced from polymers by various spinning techniques, including melt, dry, wet and emulsion spinning. Before synthetic fibers were developed, artificial (manufactured) fibers were made from cellulose, which comes from plants. At the beginning of the twentieth century, synthetic fibers started supplementing and replacing natural fibers. The first truly synthetic fiber was nylon, followed by polyesters, polyacrylics and polyolefins. Also synthetic elastomeric, glass and aramid fibers became important commercial products (Muhammad Jannah, 2008).

Synthetic fibers are now available, ranging in properties from the high elongation and low-modulus elastomeric fibers, through the medium-elongation and medium-modulus fibers such as polyamides and polyesters, to the low-elongation, high modulus carbon, aramid and inorganic fibers. With such a wide variety of synthetic fibers available, the volume of synthetic fibers consumed worldwide is now greater than that of natural fibers. Most synthetic fibers have relatively smooth surfaces and they are frequently subjected to various mechanical and heat-setting processes to provide crimp (Muhammad Jannah, 2008).

The modern synthetic fiber that was made from older artificial materials and become the most common of all reinforcing fibers for polymer matrix composites is glass fiber (Agarwal and Broutman, 1990). Glass fiber is the dominant fiber and is used in 95 % of cases to reinforce thermoplastic and thermoset composites (Mohanty et al., 2005). The principal advantages of glass fibers are low cost and high strength compared with others synthetic fibers. The disadvantages are low modulus and poor adhesion to polymer matrix resins, particularly in the presence of moisture. The classification of synthetic fibers is shown in Figure 2.10.

2.8.2. Natural fibers

Natural fibers are subdivided based on their origins, for example vegetable/plants, animals, or minerals. Vegetable or plant fibers include bast or stem fibers, leaf or hard fibers, seed, fruit, wood, cereal straw and other grass fibers (Alexander et al., 2005). According to Jeronimidis (1989), plants can stand up because of cellulose and lignin.

Structural materials in animals are mainly made of proteins such as collagen, elastin and keratin in combination with various polysaccharides, calcium minerals (in bone and teeth) or complex phenolic compounds (in hard insect cuticles). Mineral fibers are naturally occurring fibers or slightly modified fibers procured from minerals. Mineral fibers such as asbestos fibers had been used historically for insulating houses.

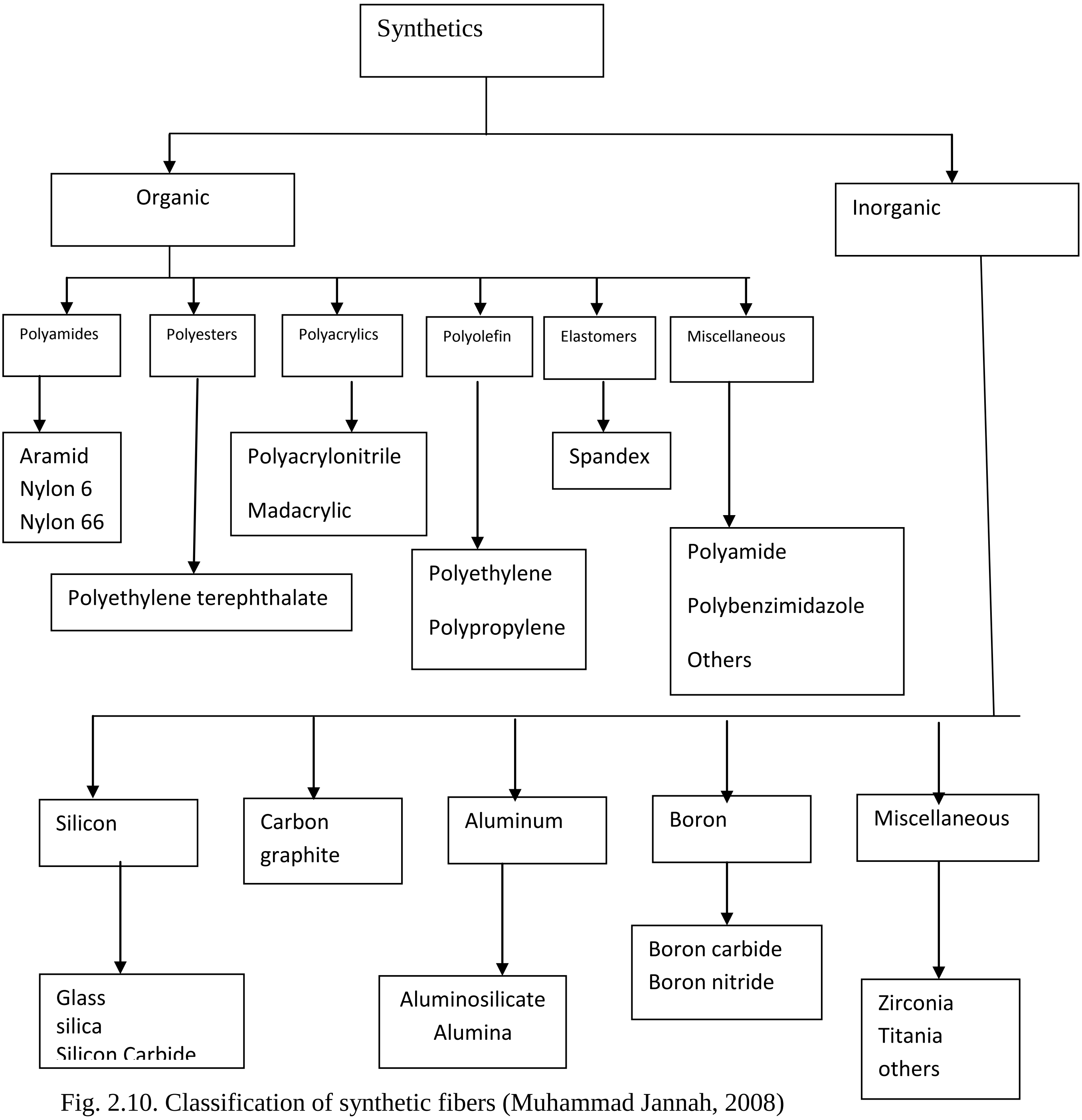


Fig. 2.10. Classification of synthetic fibers (Muhammad Jannah, 2008)

However, since January 1997, to provide protection of workers and consumers, the manufacture and transformation of asbestos fibers became forbidden (Bilba et al., 2007).

Natural fibers are now emerging as viable alternatives to glass fibers either alone or combined in composite materials for various applications. The advantages of natural fibers over synthetic or man-made fibers such as glass are their relatively high stiffness, a desirable property in composites, low density, recyclable, biodegradable, renewable raw materials, and their relatively low cost (Mohanty et al., 2005; Frederick and Norman, 2004; Joseph et al., 2002). Besides, natural fibers are expected to give less health problems for the people producing the composites. Natural fibers do not cause skin irritations and they are not suspected of causing lung cancer (Bos, 2004). The disadvantages are their relatively high moisture sensitivity and their relatively high variability of diameter and length. The abundance of natural fibers combined with the ease of their processability is an attractive feature, which makes it a covetable substitute for synthetic fibers that are potentially toxic (Pothan et al., 2006).

Definition of Natural fiber

“Natural” fibers in the strict meaning of the word are produced through agriculture (Schuster et al., 2004). Natural fibers are composite materials designed by nature. The fibers are basically a rigid, crystalline cellulose microfibril-reinforced amorphous lignin and hemicelluloses matrix. Most plant fibers, except for cotton, are composed of cellulose, hemicelluloses, lignin, waxes and some water-soluble compounds, where cellulose, hemicelluloses and lignin are the major constituents.

From a structural point of view, natural fibers are multi-cellular in nature, consisting of a number of continuous, mostly cylindrical honeycomb cells which have different sizes, shapes and arrangements for different types of fibers (Dipa and Jogeswari, 2005). These cells are cemented together by an intercellular substance which is isotropic, non cellulosic and ligneous in nature, with a cavity termed the lacuna, whose position and dimensions differ in composition and orientation of cellulosic microfibrils.

There is a central cavity in each cell called the lumen (Dipa and Jogeswari, 2005). The microfibrils in the central walls form a constant angle (microfibrillar or helical angle) for each type of fiber with the fiber axis, so that the crystallites are arranged in a spiral form, the pitch of which varies from one fiber to another. Thus, each fiber is a ‘natural composite’ by itself, wherein crystallites (mostly cellulosic) remain rooted in a matrix in a given orientation. Hence the properties of the single fibers depend on the crystallite content, their sizes, shape, orientation, length/diameter (L/D) ratio of cells, thickness of cell walls, and finally, their defects such as lumen and lacuna (Dipa and Jogeswari,2005).

Dipa and Jogeswari (2005) summarized that the most important factor controlling the different types of natural fibers is their species because the properties of fibers are different between different species. In addition, the properties of fibers within a species vary depending on area of growth, climate and age of the plant. Lastly, the properties of natural fibers vary greatly depending on their processing method used to break down to the fiber level.

Advantages of natural fibers

The majority of natural fibers that have been investigated for composite reinforcement are ligno-cellulosic materials, or those derived from plants. The strengths of plant fibers have long been recognized and utilized. Roping, textiles, tools, and even housing materials are just some of the past and present applications that take advantage of the unique mechanical properties of plant fibers. In addition to their good mechanical properties, there are several other advantageous qualities that plant fibers have over traditional composite fiber reinforcements. These fibers are typically derived from fast-growing renewable plants and therefore are not only significantly cheaper and much less subject to economic fluctuations, but also reduce environmental concerns associated with the depletion of natural resources. Additionally, the density of cellulose based fibers can be as much as half that of traditional reinforcing fibers such as those made from glass (Mohanty et al., 2000). The resulting natural fiber reinforced polymer (NFRP) composites can therefore be lighter than traditional composites allowing for more efficient systems and reduced costs associated with material transportation. Natural fibers are also non-toxic materials making them occupationally safer than glass fibers which produce glass particles during processing that can cause allergic reaction, skin irritation, or irritation to the respiratory system if inhaled. Plant fibers are also known to be less abrasive than traditional fibers allowing for less damage to the equipment required to process them.

Composites made with natural fibers are alleged to have considerably improved life cycle assessments (LCA) as they require less energy and have a lower carbon footprint than those made with glass fibers (Schloesser, 2004). This is in part due to the fact that only the amount of carbon dioxide that a plant assimilates during its growth phase is released during its degradation or combustion, thereby making plant fibers a carbon neutral material (Joshi et al., 2004). Additionally, the energy consumption to produce a natural fiber mat (9.55 MJ/kg), including cultivation, harvesting, and fiber digestion, is less than a quarter of the energy required to produce a comparable glass fiber mat (54.7 MJ/kg). A study by Patel and co-workers (Zah, et al., 2007) performed LCAs for several current biocomposite systems and determined that the potential environmental benefits of biocomposites are significant and can be expected to be a valuable contributor toward a more environmentally friendly and sustainable world.

Table 2.2. Comparisons between natural and glass fibers (Mohanty et al., 2002).

	Natural fibers	Glass fibers
Density	Low	Twice that of natural fibers
Cost	Low	High
Distribution	Wide	Wide
Energy consumption	Low	High
Renewable	Yes	No
Recyclable	Yes	No
Co_2 neutral	Yes	No
Abrasion to machines	No	Yes
Health risk when disposed	No	Yes

Additionally, a study by Joshi et al. (2004) defined four general drivers for the superior environmental performance of natural fiber reinforced composites as compared to glass fiber reinforced composites for automotive applications;

(i) the production of natural fibers has lower environmental impact,
(ii) substitution of base polymers by higher volume of natural fibers,
(iii) lower energy use during lifecycle as a result of reduced material weight,
(iv) bio-degradation or energy and carbon credits from end life incineration. This study determined that in most cases natural fiber composites are likely to be environmentally superior to glass fiber composites.

Disadvantages of natural fibers

The transition from conventional fibers like glass, carbon, and aramid to plant based fibers for composite reinforcement faces some difficulties. Firstly, the processing and manufacture of natural fibers is different from that of traditional fiber reinforced polymers (FRP). This can act as an obstacle for widespread implementation of natural fiber reinforced composite technology as it may require companies to develop new machines and methods for manufacture of broad goods and fabrics. Additionally, conventional fibers can be repeatedly produced with a defined range of properties. In contrast, the properties of natural fibers are subject to a higher level of variability. Some factors affecting fiber properties, such as chemical composition and physical structure, are inherent to the fiber. Other factors like growing conditions, harvesting and processing techniques, and even storage are more variable and therefore can change the properties of fibers from one batch to the next. Perhaps the most serious problem with using natural fibers in composites is their moisture sensitivity. Their hydrophilic nature causes the fibers to take on water causing degradation and swelling potentially leading to fiber/matrix interface problems. For bio-composite technologies to be successful, it is necessary that this problem of fiber weakness be thoroughly researched and addressed in terms of efficient resolution.

2.9. Types of natural fiber

Plant fibers used for composite reinforcement are commonly categorized by their origin:

(1) Bast fibers, obtained from the fibrous bundles within the inner bark of a plant stem;

(2) Leaf fibers, obtained from fibers running the length of plant leaves;

(3) Seed fibers;

(4) Reeds and grass stems;

(5) Fruit fibers; and

(6) Wood fibers from the core of trees.

The majority of research in the field of natural fiber composite reinforcements has been conducted using plant fibers from the bast and leaf categories. These fibers, also known as hard fibers, are the most common in natural fiber composites because of their combination of high strength and stiffness as well as low elongation to break. Bast fibers are also of particular interest because of their long lengths, providing for easier fiber alignment and relatively low moisture uptake, reducing fiber swelling and consequently giving better fiber/matrix adhesion in humid environments (Pott, 2004).

Bast fibers. Bast fiber crops have rigid stalks with nodes regularly spaced along their length. Between each node, the stalks have a hollow core followed by a thick woody pith layer, a cambium transition layer, a phloem with short chlorophyll-containing cells and long bast fiber cells, and lastly a thin protective layer consisting of the cortex and epidermis (Bismark et al., 2005). The bast fibers of interest for composite reinforcement are the long fibers which run lengthwise in the phloem regions, just inside of the protective bark. Although the shape and size of the stem of various bast fiber crops are different, the structure of the individual bast fibers is similar. High yields of the long individual fibers or fiber bundles can be obtained from bast fiber crops at relatively low cost.

A summary of the most commonly used bast fibers for natural fiber reinforcements as well as their geographic origins, physical characteristics, and corresponding mechanical properties are described in the following sections.

Flax. According to Bismark,et al., 2005, Flax (*Linum usitatissimum L., Linaceae*) is typically grown in temperate regions such as southern Europe, Argentina, India, and

China and has been cultivated for nearly 10,000 years. Common uses of flax plants are to provide important products such as oil seed, paper and pulp, and textile yarns and fabrics. Flax plants are quite fast growing and can grow to heights of 80 to 150 cm in less than 110 days. The bast fibers come from the central portion of the plant and are in bundles between 60 and 140 cm long with diameters ranging from 40 to 80 μm. Flax fibers are one of the strongest and stiffest plant fibers; however, they have relatively low longitudinal extension to failure when subjected to tensile loads.

Hemp. Hemp (*cannabis sativa L., Cannabaceae*) is typically grown in moderate climates such as those of Central Asia and Northern America. Although it has been cultivated for more than 12,000 years, its cultivation has been limited by strict legislation as a result of its physical resemblance to the narcotic drug, marijuana. Hemp cannot be used as a narcotic, however, as it produces less than 1% of the narcotic 9- tetrahydrocannabinol (THC) in comparison to 3-20% produced by marijuana. There are a wide variety of products made from hemp plants including specialty paper, textiles, construction materials, plastics and composites, food, medicine, and fuel. Hemp crops are advantageous to other fiber crops in that they are very resilient crops requiring no or minimal herbicides, fungicides, pesticides, and fertilizers. Hemp plants also have quick growth rates reaching heights of up to 5 m with bast fiber contents between 28 and 46 percent. The fiber strands have lengths of 1.8 m or longer with elementary fibers averaging 13 to 25 mm in length. Hemp fibers, although hygroscopic, are highly resistant to moisture degradation and rot very slowly in water. Hemp fibers also have excellent mechanical properties such as strength and stiffness, with low elongation to break as a result of their low cellular microfibril angle.

Jute. Jute (*Corchorus capsularis, Tiliaceae*) plants thrive in hot humid environments and although it originated in the Mediterranean it spread to the Near and Far East (Bismark et al., 2005) . Today it is grown mostly in the delta formed by the Ganges River and Bramhaputra River in India and Bangladesh as well as in Thailand, China and Brazil. The use of jute plants by humans dates back to prehistoric times. These annuals grow to heights between 2 and 3.5 m, with stalk diameters ranging from 2 to 3 cm. Unlike flax and hemp, jute plants are grown entirely for its fibers which are between 1.5 and 3 m in length. These bast fibers vary widely in size and although they are strong, their tensile properties are lower than those of other bast fibers such as hemp and flax. Additionally, they are fairly brittle and exhibit low elongation to break as a result of high lignin content. Jute fibers are resilient to attacks by microorganisms; however, they are quite hygroscopic and are sensitive to moisture as well as chemical and photochemical attack.

Ramie. Ramie (*Beohmeria nivea L. and Boehmeria viridis, Urticaceae*) is a hardy perennial crop that is cultivated primarily in Indonesia, China, Japan, and India. The plants grow to heights of 1.2 to 2.5 m and can be planted and harvested up to six times per year. Ramie fibers are primarily used in textile industries as they are very fine (diameters from 10 to 25 μm) and silk-like as well as strong. They also have very good resistance to bacteria, mildew and insect attack. Unlike flax, hemp, and jute fibers they are also stable in both alkaline media and mild acids. Ramie fiber also has excellent strength and stiffness properties which are most likely a result of their high cellulose content. A disadvantage of ramie fibers is their often high cost in comparison to other natural fibers.

Kenaf. Kenaf (*Hibiscus cannabinus L., Malvaceae*) is an annual cane-like crop originating in Asia and Africa. Kenaf plants are fast growing and reach heights of 2.4 to 6 m in 5 months. Products made from kenaf plants include paper, textiles, and composites.

The kenaf plant has both short and long fibers within its stalks; however the elementary fibers are quite short, having lengths of 1.5 to 6 mm. The fibers have a striated surface as well as an irregular shape. Additionally, the fibers are coarse and brittle and can be difficult to process. Their mechanical properties are similar to those of jute fibers; however, the density of kenaf fibers is typically less than that of jute as a result of the lower cellulose content. Kenaf has been said to have the highest carbon dioxide absorption of any plant making it a valuable tool in the reduction of CO_2 contents in the atmosphere.

2.9.1. Leaf fibers

In many monocotyledons, long strand leaf fibers are embedded in parenchymatous tissue to provide mechanical support for the long and broad leaves. As in the bast, these composite fiber strands are composed of many smaller ultimate fibers

(Muhammad Jannah, 2008). In general, leaf fibers are more coarse than bast fibers and are commonly used as cordage, mats, rugs, and carpet backings rather than clothing fabrics. Common examples of these materials are Manila hemp (abaca) and sisal, both of which are used for twines and ropes worldwide. In addition, abaca and sisal have been both used historically for paper fibers (Muhammad Jannah, 2008). According to Alexander et al. (2005), abaca and sisal are hard fibers obtained from the leaves of their plant and they are considered to be the strongest of all plant fibers. Whereas the leaf fiber strands display the characteristically long length of bast fibers, the ultimate fibers comprising these strands are typically less than 12 mm long as shown in Table 2.3. Like most of the plant fibers, the leaf ultimate fibers have typical diameters of approximately 30 mm.

Table 2.3. Dimensions of bast and core fibers.

Type	Property	Flax	Hemp	Jute	Kenaf
Bast strand	Length (cm)	25-120	100-400	150-360	200-400
	Width (mm)	0.04-0.6	0.5-5	-	-
Bast Ultimate	Length (mm)	4-69	5-55	0.7-6	2-11
	Diameter (mm)	8-31	16	15-25	13-33
Core Utimate	Length (mm)	0.2	0.7	1.06	0.6
	Diameter (mm)	-	-	26	30

Table 2.4. Dimension of leaf fibers.

Type	Property	Abaca	Henequen	Phormium	Palm	Sisal
Strand	Length (cm)	365	60-150	150-240	30-60	60-120
	Width (mm)	0.2-1	0.1-0.5	0.1-0.5	0.15-0.25	0.1-0.5
Ultimate	Length (mm)	2-12	15-4	2-11	-	0.8-7
	Width (mm)	6-40	8.3-33	5.25	18	8-48

2.9.2. Seed-hair fibers

Unlike the bast and leaf fibers, seed-hair fibers are single celled. These fibers are attached to the seeds of certain plants for aid in wind-dispersal (Muhammad Jannah, 2008). One exception to this is coir, a fiber produced from the husk of coconuts. Like bast fibers, coir is produced by separation technique after husks undergo retting. Coir can produce long fiber strands, depending on the production process. The ultimate coir fibers lengths are quite small which are less than 1 mm long and 6 mm in diameter as shown in Table 2.4. The more typical seed-hair fibers are all similar in morphology to cotton, with long lengths (20 mm) and small diameters (20 mm). Cotton fibers consist of the unicellular seed hairs of the bolls of the cotton plant (Alexander et al., 2005).

Innumerable products are made from cotton, primarily textile and yarn goods, cordage and automobile tire cords. Cotton fibers are the backbone of the textile trade of the world. Short fibers that are left on the seed after processing are termed linters (Muhammad Jannah, 2008). Cotton linters are commercially available and are currently used as rag content in fine papers. These waste fibers have similar diameters to the textile cotton, with much shorter lengths (less than 7 mm). Kapok and milkweed both have thin cell walls and large diameter lumens. Since kapok is difficult to spin, it is primarily used as loose fill for buoyancy or insulation purposes. Milkweed has been used only on a limited basis for fill; however, it has been studied for its papermaking and textile spinning qualities (Muhammad Jannah, 2008).

Table 2.5. Seed-hair fiber dimensions.

Property	Coir	Cotton	Cotton linters	Kapok	Milk weed
Length (mm)	0.21-1	10-50	3-7	15-30	5-40
Diameter (mm)	6.24	12-25	30	10-30	6-38

2.9.3. Cereal straws

Straw is produced throughout the world in enormous quantities as a by-product of cereal cultivation. Half the straw is simply burned on the fields just to get rid of it or else it is buried in greater quantities than what is needed to replenish organic matter in the soil (Alexander et al., 2005). Historically, straws from wheat, rye, and rice were widely used as a pulp source for paper-making. Whereas this practice became extinct in North America and much of Europe by 1960, it is still practiced in southern and eastern Europe as well as many Asian, Mid-Eastern, and Southern American countries.

Although corn stalks are widely available and have been studied as a pulp source, their commercial use has been limited (Muhammad Jannah, 2008; Foyle et al., 2007). According to Alexander et al. (2005), straw consists of stem and leaves branching off the stem at the nodes. Straw also was found to have less cellulose and lignin but more hemicelluloses as compared to wood. In general, wheat straw and corn stalk fibers have small diameters and average lengths producing a favorable aspect ratio of 110 as shown in Table 2.6. Fibers from rice straws typically have smaller diameters (5 to 14 mm) with a long aspect ratio of 170 (Muhammad Jannah, 2008). Straw offers some technological advantages over wood because it allows better packing of the fibers or strands and the degree of bonding between them is much better.

Table 2.6. Cereal straw fiber dimensions.

Property	General	Corn	Rice	Wheat
Length (mm)	0.68-3.12	1.0-1.5	0.65-3.48	1.5
Diameter (mm)	7-24	20	5-14	15

2.9.4. Grass fibers

Many other grasses have been and are considered as a fiber source. Because of limited availability or processing difficulties, most of these fibers (except sugarcane bagasse) have never become widely used; however, they are often common in certain localities. These fibers include sugarcane bagasse, bamboo, esparto, and sabai grass.

In these grass fibers, sugarcane bagasse is the most commonly and widely studied among researchers. Sugarcane is grown as a source of sucrose in many tropical and sub-tropical countries in many continents. Bagasse is the residue remaining after the sugar has been extracted (Muhammad Jannah, 2008). Currently, this material is used for paper in India and Mexico and in boilers to generate process steam for the sugar production. Paper and material fiber provide the more economical use for this material (Muhammad Jannah, 2008). The bagasse consists of pith and rind materials.

The rind consists of 50% of the dry bagasse weight and contains most of the usable fiber. In general, these fibers are similar to hardwood fibers average 2.8 mm in length and 34 mm in diameter, producing an aspect ratio of 82. Two grasses, esparto and sabai, are widely available in North Africa and India, respectively. Both grasses are locally common as a paper fiber, with sabai at one time comprising 20% of the pulped material in India. Compared to softwood fibers, these grass fibers have small diameters (9 mm) and average lengths (1-2 mm), producing fibers with large aspect ratios between 100 and 200 (Muhammad Jannah, 2008).

Table 2.7. Other grass fiber dimensions .

Property	Sugar cane bagasse	Banboo	Espanto	Sabai
Length(mm)	2.8	2.7-4	1.1	0.5-4.9
Width (mm)	34.1	15	9	9-16

2.10**.** Structure and chemical composition of plant fibers

2.10.1**.** Chemical and Molecular Structure

Plant fibers are lignocellulosic in nature. The major constituents are cellulose, hemicellulose, and lignin. The amount and location of these constituents depends on a fiber's species and age and are important contributors to the overall fiber properties.

Cellulose is the essential component of plant fibers. Cellulose is a semi-crystalline polymer containing many hydroxyl functional groups along its long chain macromolecular structure. These hydroxyl groups are capable of forming hydrogen bonds with other hydroxyl groups along the chain as well as with other cellulose chains in fibers (Bledzki eta l., 1996). The amorphous and crystalline regions of cellulose are similar in structure; however, the amorphous regions have a lower frequency of inter chain hydrogen bonding making then more open to water molecules. In these regions the hydroxyl groups will form hydrogen bond with water vapor in the air making the cellulose naturally hydrophilic.

The chemical structure of cellulose is the same for all natural fibers; however, the degree of polymerization (DP) or length of the polymer chains of the cellulose varies. The DP of cellulose has a significant effect on the mechanical properties of a fiber as it is directly related to the molecular weight (MW) (Wambua et al., 2003). When the structure of cellulose has a high DP and MW, the fiber will likely have greater strength properties. In general, bast fibers, such as flax, hemp, jute, ramie and kenaf, have high cellulose contents and commonly show the highest DP among natural fibers, explaining their good use in composite materials (Lewin and Pearce,1985).

Hemicellulose is a fully amorphous branched polymer with a selection of sugar molecules as monomeric units. Unlike cellulose, the constituents of hemicellulose differ between plant species and crops. The open structure of hemicellulose, containing many –OH and acetyl groups, allows it to absorb significant amounts of water. In addition, the DP of hemicellulose is much lower than that of cellulose making it a weaker polymer (Pott., 2004). Lignin is a complex cross-linked polymer with a chemical network that is still not completely understood. It functions as a structural support material in plants by filling the spaces between the cellulose and hemicellulose regions cementing them together.

Mechanical properties of lignin are lower than that of cellulose (Bledzki and Gassan, 1999). Additionally, the aromatic nature of lignin, high carbon and low hydrogen content, make it hydrophobic (Bismark et al., 2005).

2.10.2 Physical Structure

Plant fibers are characterized by a cellular structure consisting of cellulose regions connected via lignin and hemi-cellulose fragments. A single cell consists of a series of cell walls, typically with a hollow center, giving the overall fiber a porous structure (Bledzki and Gassan, 1999).The microfibrillar angle of a plant fiber is an important characteristic and can be defined as the deviation in alignment of the microfibrils to the parallel axis of the fiber. The cell aspect ratio of a fiber (L/D) is defined as the relationship between the fiber cells length and diameter, with higher values therefore representing long thin fibers.

In addition to the chemical composition of a fiber, the structural parameters including microfibrillar angle and cell aspect ratio (L/D) have been suggested to be the most significant properties affecting the values of fiber strength, stiffness and elongation.

It has also been shown experimentally that in general the smaller the fiber angle, i.e., the more parallel the microfibrils are to the fiber axis, the higher the mechanical properties of the fiber (Lewin and Pearce, 1985). Theoretical models predicting fiber properties, such as modulus, based on these parameters have been demonstrated (Hearle and Sparrow, 1979).

2.10.3.Durability/Environmental Sensitivities

Plant fibers have varying sensitivities to thermal, environmental and chemical exposures. Research has suggested cellulosic fibers exhibit a loss in properties when subjected to extended thermal exposure, particularly at temperatures above 200°C (Herrmann et al., 1998).

Table 2.8. Physical and mechanical properties of natural fiber (Zah et al., 2007).

Material type	Type	Density (g/cm^3)	Diameter (µm)	Tensile strength (Mpa)	Elastic modulus (Gpa)	Elongat. at break (%)
Glass	Mineral	2.5-2.55	5-25	1800-3500	70-73	2.5-30
Flax	Bast	1.4-15	20-600	345-1500	27.6-80	1.2-3.2
Hemp	“	1.45-1.5	25-200	550-900	70	1.6
Jute	“	1.3-1.49	25-200	393-800	10-30	1.16-1.8
Ramie	“	1.5-1.55	10-25	400-938	44-128	1.2-3.8
Kenaf	“	1.193	90-100	375-930	22-53	1.5-1.6
Coir	Seed/hair	1.15-1.46	100-460	131-220	4-6	15-40
Cotton	Seed	1.5-1.6	12-38	287-800	5.5-12.6	7-8
Sisal	Leaf	1.33.1.45	50-390	468-700	9.4-38	2-7
RALF	“	1.44-1.53	20-80	413-1627	34.5-82.5	1.6-2.4
Curava	“	1.4		500-1150	11.8	3.7-4.3
Hardwood	Wood	0.6-0.9		90-110	11-13	
Softwood	“	0.3-0.7		60-90	8-14	

Plant fibers have varying levels of resistance to environmental conditions such as moisture, UV radiation, and attacks by microorganisms and bacteria. Environmental sensitivities are inherent to many natural fibers and aid in their ability to degrade upon disposal; however, these sensitivities must be considered when using materials incorporating natural fibers in applications that could require periods of environmental exposure. Some natural fibers are also known to exhibit a reduction in mechanical properties when exposed to chemical media such as alkaline or acids (Ochi, 2006). Although brief exposure to these media has been strategically used by researchers to enhance the properties of natural fibers (Kozloski and Wladyka-przybylak, 2004), in general, uncontrolled or extended exposure can be expected to greatly reduce the properties of natural fibers.

2.11. Main components of natural fibers

The major chemical component of a living tree is water, but on a dry weight basis, all plant cell walls consist mainly of sugar-based polymer (carbohydrates) that are combined with lignin with lesser amounts of extractives, protein, starch and inorganics (James and Jeffrey, 1997). The chemical components are distributed throughout the cell wall, which is composed of primary and secondary wall layers. Chemical composition varies from plant to plant, and within different geographic locations, ages, climate and soil conditions (James and Jeffrey, 1997). According to Sain and Panthapulakkal (2004), the age of the plant, climatic conditions and fiber processing techniques influence the structure of fibers as well as their chemical composition. Bledzki and Gassan (1999) summarized that the components of plant fibers are cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances, with cellulose, hemicellulose and lignin the major components with regard to the physical properties of the fibers. The percentages and properties of the components contribute to the overall properties of the fibers (Sain and Panthapulakkal, 2004).

2.11.1. Cellulose

Cellulose is the essential component of all plant fibers and is the most abundant organic chemical on the face of the earth. In 1838, Anselme Payen suggested that the cell walls of large numbers of plants consist of the same substance, to which he gave the name “cellulose” (Bledzki and Gassan, 1999).

According to Nishino (2004), cellulose is a natural linear homopolymer (polysaccharide), in which D-glucopyranose rings are connected to each other with β-(1-4)-glycosidic linkages. It is thus a 1, 4-β-D-glucan (Bledzki and Gassan, 1999). Sain and Panthapulakkal (2004) summarized that the basic chemical structure of cellulose in all plant fibers is the same whereas the cell geometry of each type of cellulose varies with the fiber; this is one of the factors which contribute to the mechanical properties of the green fibers. The molecular structure of cellulose can be seen from Figure 2.11. It can be seen that cellulose structure contains alcoholic hydroxyl group. These hydroxyl groups form intermolecular and intra molecular hydrogen bonds with the macromolecule itself and also with other cellulose macromolecules. Therefore, all natural fibers are hydrophilic in nature (Mohanty et al., 2005; Herrera and Valadez, 2005).

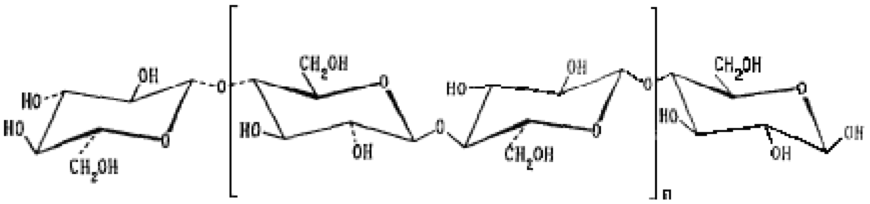


Fig. 2.11. Molecular structure of cellulose (Sitikhadijah Binti, 2010).

Although the chemical structure of cellulose from different natural fibers is the same, the degree of polymerization (DP) varies. The mechanical properties of a fiber are significantly dependent on the DP (Mohanty et al., 2005). According to James and Jeffrey (1997), the number of glucose units in a cellulose molecule is referred to as the degree of polymerization (DP), and the average DP for plant cellulose ranges from low of about 50 for a sulfite pulp to approximately 600, depending on the determination method used.

Most plant-derived cellulose is highly crystalline and may contain as much as 80 % crystalline regions. The remaining portion has a lower packing density and is referred to as amorphous cellulose. On a dry weight basis, most plants consist of approximately 45-50 % cellulose. This can vary from a high (cotton) of almost 90 % to a low about 30 % for stalk fibers (James and Jeffrey, 1997).

2.11.2 Hemicelluloses

Hemicelluloses are another component of plant fibers. Hemicelluloses are polysaccharides composed of a combination of 5- and 6-ring carbon ring sugars (Alexander et al., 2005). Figure 2.12 shows partial structure of hemicelluloses with a combination of 5-ring carbon ring sugars (Bledzki and Gassan, 1999).

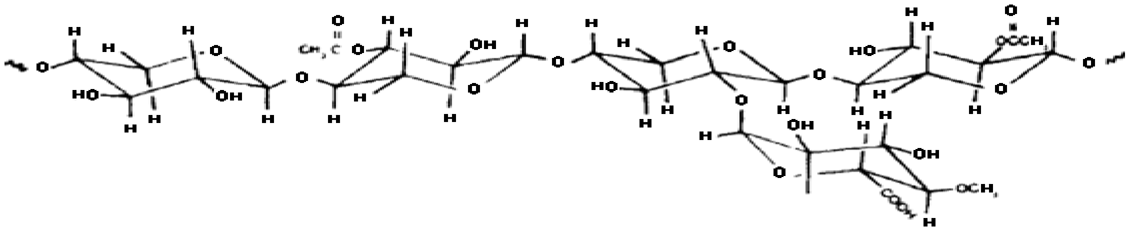


Fig. 2.12. Partial structure of hemicelluloses (Bledzki and Gassan, 1999).

Hemicelluloses has a random, amorphous structure with little strength, highly branched polymer compared to the linearity of cellulose, and has a degree of polymerization lower than that of cellulose (Sain and Panthapulakkal, 2004). They are also classically defined as alkali soluble materials after removal of pectic substances, very hydrophilic and easily hydrolyzed in acids (Xiao etal, 2001; Alexander et al., 2005).Unlike cellulose, which contains only a 1, 4-β-glucopyranose ring, hemicelluloses contains different types of sugar units such as D-xylopyranose, D-glucopyranose, Dgalactopyranose, L-arabinofuranose, D-mannopyranose, and D-glucopyranosyluronic acid with minor amount of other sugars (James and Jeffrey, 1997). These monomers of hemicelluloses are shown in Figure 2.13. Usually, all of the monomers are present. There may even be small amounts of L- sugars but Xylose is always the sugar present in the largest amount.

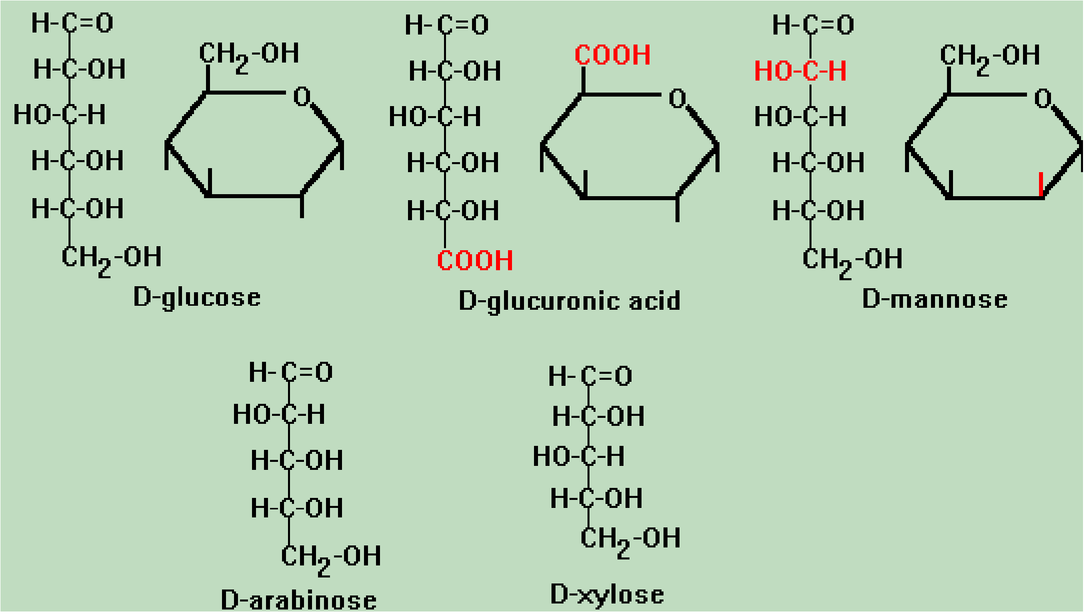


Fig. 2.13. Monomers of hemicelluloses (Sitikhadijah Binti, 2010).

Bledzki and Gassan (1999) have concluded that hemicelluloses differ from cellulose in three aspects. First, it contains several different sugar units whereas cellulose contains only 1, 4-β-D-glucopyranose units. Second, it exhibits a considerable degree of chain branching, whereas cellulose is a linear polymer. Third, the degree of polymerization of native cellulose is 10-100 times higher than that of hemicelluloses.

2.11.3 Lignin

Lignin is a complex chemical compound most commonly derived from wood and an integral part of the cell walls of plants. The term was introduced in 1819 by de Candolle and is derived from the Latin word *lignum*, meaning wood (Sitikhadijah Binti, 2010).

Lignin is the compound that gives rigidity to the plants. It is thought to be a complex, three-dimensional copolymer of aliphatic and aromatic constituents with very high molecular weight. Lignin is amorphous and hydrophobic in nature (Alexander et al., 2005; Mohanty et al., 2005; James and Jeffrey, 1997). The partial structure of lignin can be seen in Figure 2.14.

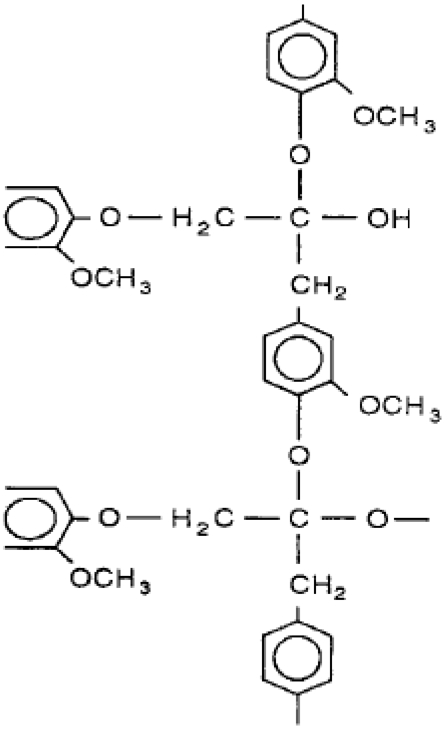


Fig. 2.14. Partial structure of lignin (Bledzki and Gassan, 1999).

Lignin can be classified in several ways but they are usually divided according to their structural elements. All plants lignin consist mainly of three basic building blocks of guaiacyl, syringyl, and p-hydroxyphenyl moieties, although other aromatic type units also exist in many different types of plants, which forms a randomized structure in a tri-dimensional network inside the cell walls (Xiao et al., 2001; James and Jeffrey, 1997). Figure 2.15 shows building blocks of lignin. There is a wide variation of structure within different plant species. The function of the lignin in plants is as an encrusting agent in the cellulose/hemicelluloses matrix or called plant cell wall adhesive. Therefore, lignin acts as a structural support material in plants by filling the spaces between the polysaccharide fibers, which hold the natural structure of the plant cell walls together (Sain and Panthapulakkal, 2004; James and Jeffrey, 1997). Lignin stiffens the cell walls and acts as a protective barrier for the cellulose, thus the carbohydrate is protected from chemical and physical damage. They are totally insoluble in most solvents and cannot be broken down to monomeric units. The properties of lignin vary with the fiber type, but it always has the same basic composition (Mohanty et al., 2005; Sain and Panthapulakkal, 2004).

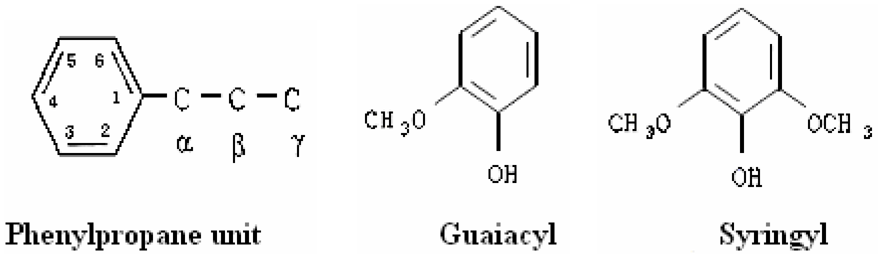


Fig. 2.15. Building blocks of lignin (James and Jeffrey, 1997).

Lignin plays a crucial part in conducting water in plant stems. The Polysaccharide components of plant cell walls are highly hydrophilic and thus permeable to water, whereas lignin is more hydrophobic. The cross linking of polysaccharides by lignin is an obstacle for water absorption to the cell wall. Thus, lignin makes it possible for the plant's vascular tissue to conduct water efficiently (Sitikhadijah Binti, 2010).

2.12. Engineering properties of natural fibers

The mechanical properties of plant fibers are very good and in some cases are comparable to those of glass fibers. Although the tensile strengths of these fibers are lower than that of glass fibers. The lower density of the natural fibers gives them comparable values of specific strength. Depending on the species of plant fibers, the fiber elongation to break can be above or below the range for traditional glass fibers, allowing for easily tailored composite properties through selection of natural fibers.

Additionally, the hollow tubular structure of natural fibers provides better insulation against heat (low thermal conductivity properties of between 0.29-0.32 /m-K) and noise). As explained in the previous sections, the mechanical properties of natural fibers are highly dependent on the chemical and physical characteristics of the fibers.

Several experimental research efforts have demonstrated these trends. Research by Mukherjee et al. (2000), on several bast, leaf, and fruit fibers determined that high ultimate tensile strength (UTS) and low elongation are typically correlated with high cellulose, low fiber angles, and high cell aspect ratios (L/D) in fibers. Similarly, low UTS and high elongation values were found in fibers with lower cellulose content, high fiber angles, and low L/D values (Netravali and Chabba, 2003). It is important to note, however, that the correlation between strength and the above parameters is weaker than for elongation and stiffness values as fiber strengths are strongly affected by the presence of defects (Green, et al., 1999). By understanding the effect of different chemical and structural parameters of fibers on their mechanical properties, the choice of fibers for composite reinforcement can be better tailored to meet the application needs of the composite. Some mechanical properties of natural fibers commonly used for composite reinforcement in comparison to conventionally used fibers as shown in Table 2.8 provides relative data on the physical and mechanical properties of natural fibers. A comparison between the mechanical properties and physical properties in Table 2.8 is useful when determining an ideal natural fiber for use as reinforcement in a composite.

2.13. Surface treatment of natural fibers

2.13.1 Chemical Modifications

All natural plant fibers are hydrophilic in nature, which lowers their compatibility with hydrophobic polymers. The incompatibility and weak interface between polar natural fibers and non-polar polymer matrix materials, due to their dissimilar chemical nature, is a shortcoming in natural fiber composites. Several chemical modifications have been shown to enhance the fiber/matrix adhesion in natural fiber reinforced composites by introducing a third material that has properties intermediate of the other two (Bledzki et al., 1996; Bledzki and Gassan, 1999). Chemical coupling agents, such as silanes acrylonitrile, isocyanates, and maleates, improve interfacial adhesion by treating the fiber surface or polymer with a compound that forms a bridge of chemical bonds between the fiber and matrix materials (Bledzki et al., 1996). The use of silanes as coupling agents for glass fiber reinforced polymer composites is extensive (Hamanda et al., 2000). Silane coupling agents have also been used to modify the surface and increase fiber/matrix adhesion of several natural fibers including big blue grass (Mohanty et al., 2004), banana (Facca et al., 2007), bamboo (Huda et al., 2007), PALF (Devi et al., 1997), sisal (Rong et al., 2001), henequen (Valedez-Gonzalez et al., 1999), kenaf (Huda et al., 2008), hemp (Mehta et al., 2006), and jute (Bledzki and Gassan, 1999). In general, these studies reflected little or no change in the physical properties of the fiber surface, but instead saw increased fiber/matrix adhesion through increased chemical bonding and decreased fiber swelling as a result of moisture uptake in the fibers. Improvements in the thermal stability of the fiber and composite were seen (Huda et al., 2008). Many of these studies compared the effect of silane treatments to alkaline treatments and found the chemical bonding of the silane treatment to have a greater influence on the composite properties than the increased mechanical interlocking offered by alkaline treatments.

The effect of grafting acrylonitrile (AN) on natural fibers has been studied by several researchers (Morye and Wool, 2005, Shibata et al., 2003, Mehta et al., 2006).The graft polmerization of AN on sisal fibers, studied by Mishra et al. (Mishra et al., 2002) showed decreased moisture absorption and increased tensile properties of the treated fibers. Similarly, AN treatment of flax fibers (Morye and Wool, 2005) in an soy oil-based resin has been reported to increase composite strength and decrease moisture absorption. In a study by Mehta et al. (Mehta et al., 2006) on the effect of various fiber surface treatments on hemp fiber reinforced UPE composites, AN grafting was shown to yield the best improvement in composite mechanical properties. In contrast, the use of AN coupling agent on abaca fiber PLA and PBS reinforced composites resulted in little improvement of composite properties (Shibata et al., 2003). In general, the effectiveness of AN grafting on natural fiber composites was shown to be highly dependent on the characteristics of the treatments such as reaction medium, treatment time, initiatior, AN concentration, and even fiber loading (Mishra et al., 2002). For thermoplastic fiber reinforced composites, maleated coupling agents are commonly used on fibers and polymers to increase compatibility between the two constituents (Mohanty et al., 2002, Zampaloni et al., 2007; Bledzki et al., 2004, Pickering et al., 2007). The most common use for maleic anhydride (MA) is with PP where the MA is grafted onto the backbone of the polymer chain. Fibers are then treated with the heated MAPP copolymer and covalent bonds form across the interface (Li et al., 2007). Studies on the use of MAPP for glass (Hamada et al., 2000; Bernhardsson and Shishoo, 2003; Gamstedt et al., 1999), bamboo (Okubo et al., 2004), kenaf (Mohanty et al., 2002; Zampaloni et al., 2007), henequen (Mohanty et al., 2002), sisal (Joseph et al., 2002; Mohanty et al., 2004), flax (Wielage et al., 2003), jute (Gassan and Bledzki,1997), and hemp (Bourmaud and Baley, 2007; Mutje et al., 2007) fibers have reported an increase in adhesion between the polar fibers and non-polar polymer matrices. The increase in fiber/matrix adhesion was also seen to correspond to increases in composite properties; in particular the tensile strength was seen to increase as much as 77 percent for natural fibers (Mutje et al., 2007). The use of maleated coupling agents in natural fiber reinforced PE (Sebe et al., 2000), PHB (Mohanty et al., 2004), CAB (Wibowo et al., 2006), and soy oil-based polymers (Williams and Wool, 2000) has also been shown to improve the fiber/matrix adhesion suggesting that the successful use of these agents is not limited to composites with a PP matrix.

Methyl methacrylate (MMA) grafted onto coir (Rout et al., 2001) and sisal (Mishra et al., 2002) fibers resulted in increased properties of polyester amide biocomposites. For MMA grafted coir fibers, composite properties exceeded those using AN grafted fibers, whereas for sisal fiber reinforced composites the AN grafted composite properties were superior. Joseph et al. (2002) studied the effect of coupling agents, such as MA and isocyanates, on moisture absorption of sisal/PP composites. Coupling agents decreased the hydrophillicity of natural fibers and increased bonding between the fibers and matrix resulting in significant reductions in composite moisture absorption. The reduction in moisture absorption is expected to increase long term performance of natural fiber reinforced composites. Similar results were seen for a study using bio-based coupling agents such as lysine-based diisocyanate (LDI) in natural fiber reinforced composites (Lee and Wang, 2006).

2.13.2. Physical methods

Physical methods such as steam explosion, thermo treatment, fiber fibrillation and electric discharge change the structural and surface properties of the fiber and thereby influence the mechanical bonding of the fibers and polymer matrix in a composite (Herrra-Franco and Valedez-Gazalos, 2005). Wet oxidation and hydrothermal treatments have been investigated to remove impurities from the surface of natural fibers resulting in natural fibers with high cellulose contents, increased surface roughness and a more fibrillated structure (Thomsen et al., 2006). Additionally, the steam explosion process has been used on wood products (Cai, 2006; Renneckar et al., 2006) and plants (Okubo et al., 2004; Wielage et al., 1999) to extract fibers with decreased lignin contents and increased surface roughness leading to increased adhesion between the fiber and matrix. Surface modifications by discharge methods such as plasma, sputtering, and corona discharge are of interest for improving the functional properties of natural fibers. Corona discharge is used for fiber surface oxidation activation and works by changing the surface composition and therefore surface properties of composite components typically increasing bonding ability between the fiber and matrix. Similarly, during plasma treatment (Ward and Ladizesky, 1985) the fiber surface energy can either be increased or decreased depending on the type and nature of the gasses used (Bledzki and Gassan, 1999) making these fibers more compatible with polymers of different chemical or polar nature.

2.13.3. Mercerization

Mercerization is an old method of cellulose fiber modification that involves the alkaline treatment of cellulose fibers. Alkali treatments gradually remove the cementing hemicellulose and lignin portions of natural fibers which in turn cause fibrillation of the fiber bundles thereby increasing the surface area of the fibers (Li et al., 2007; Ray et al., 2002). The dissolution of hemicellulose and lignin result in a more purified cellulose increased surface roughness. The efficiency of the treatment has been shown to depend on the type and concentration of the alkaline solution as well as the length and temperature of the treatment (Mohanty et al., 2002). Although the removal of cementing materials can improve the alignment of the fiber microfibrils, reductions in fiber strengths and stiffness by as much as 50 to 70 percent, respectively, have been reported (Bledzki et al., 2004). These studies have found that by stressing fibers during mercerization losses in fiber strength can be diminished. Additionally, it was found (Goda et al., 2006) that thorough rinsing of the fibers with water or acid solutions following mercerization is required to prevent further degradation of the fiber properties.

Some authors have also reported a change in the cellulose crystallinity as well as changes in thermal stability of natural fibers through alkaline treatment.They are Pickering et al., 2007; Ray et al., 2002; Gassan and Bledzki, 2001; Liu et al., 2004; Ouajai et al., 2004. Alkali treatments on natural fibers have been extensively used to increase the properties of composites with traditional petroleum-based polymers (Pickering et al., 2007; Aziz and Ansell, 2004; Mehta et al., 2006; Devi et al., 1997; Pothan et al., 2006) and biopolymers (Vilaseca et al., 2007; Shibata et al., 2003; Cao et al., 2006; Gomes et al., 2007) through better fiber/matrix adhesion. The improved adhesion is hypothesized to be a result of changes in fiber surface roughness allowing for better mechanical interlocking. In general, composites with alkali treated fibers showed improved strength properties, whereas stiffness and impact strengths were shown to either increase or decrease depending on specifics of constituent materials and mercerization conditions. Increased strength and stiffness as well as decreased impact strengths (toughness) are attributed to good transfer of stresses between the constituents resulting in composite failure controlled by fiber fracture. However, reductions in the fiber stiffness allowing for greater fiber elongations at failure have also been attributed to decrease in composite stiffness and increased impact strength.

2.13.4. Acetylation

Another surface treatment for natural fibers known as acetylation describes a reaction resulting from the introduction of acetyl functional group into an organic compound. Chemical modification with acetic anhydride substitutes the polymer hydroxyl groups of the cell wall with acetyl groups, making the polymers hydrophobic (Li et al., 2007). As a result, acetylation is commonly used to reduce the hygroscopic nature of natural fibers thereby improving the dimensional stability and fiber matrix interface of composites. Increased fiber/matrix adhesion as a result of acetylation on natural fibers such as abaca (Shibata et al., 2003), sisal (Mishra et al., 2002), PALF, coir (Rout et al., 2001), banana (Joseph et al., 2002), kenaf (Huda et al., 2007), and jute (Rana et al., 1997) has been reported. In addition, increased thermal stability of natural fibers as a result of acetylation has been reported (Rana et al., 1997). In general, however, the mechanical properties of these composites did not increase significantly. This may be a attributed to reduced mechanical interlock between the fiber and matrix as a result of bulking action of the acetyl groups on the fiber cell walls causing a smoother fiber surface. Fiber acetylation can, however, be challenging because the fibers must be free of moisture for the reaction to occur. Additionally, because the hydrophobicity of the fibers increases with increased acetyl content, long treatment time and the use of a catalyst or solvent are sometimes necessary to ensure that all available polymer hydroxyl groups are reached and able to be replaced with the acetyl groups.

2.14. Effects of fiber surface treatments on fibers

The chemical modification directly influences the cellulosic fine structure of natural fiber. This section reviews the effects of fiber modification on the stress-strain behavior and tensile properties of fibers.

2.14.1. Stress-strain behavior

The mechanical performance of fibers is dependent upon its chemical composition, chemical structure and cellular arrangement. Sreekala et al. (2000) performed tensile stress-strain test for untreated and modified oil palm fiber. Each individual fiber was composed of fibrils held together by non-cellulosic substances, such as lignin and pectin. Failure of the fiber was gradual upon the application of tensile stress. It showed intermediate behavior between brittle and amorphous. As stress gradually increased, some of the fibrils may have slipped out. The total of the stress was then sheared by fewer cells. Further increase of stress led to the rupture of cell walls and de-cohesion of cells. This resulted in a catastrophic failure of the fiber. Modifications led to major changes on the fibrillar structure of the fiber. It removed the amorphous components.

This changed the deformation behavior of the fibers. The brittleness of the fiber was substantially reduced upon treatments.

2.14.2. Tensile properties of fibers

Sreekala et al. (2000) measured the tensile properties of untreated and modified fibers, such as tensile strength, Young’s modulus and elongation at break. Many of the modifications decreased the strength properties due to the breakage of the bond structure, and also due to the disintegration of the non-cellulosic materials. Some of the treatments, like silane and acrylation, led to strong covalent bond formation and thereby the strength was enhanced marginally. Optimum mechanical performance was observed for silane-treated and acrylated fiber. The reinforcing ability of the fibers did not just depend upon the mechanical strength of the fibers but on many other features, such as polarity of the fiber, surface characteristics and presence of reactive centers. These factors control interfacial interaction. The Young’s modulus of the fibers improved upon acrylation, alkali and silane treatment. The improved stiffness of the fibers was attributed to the crystalline region (cellulosic) of the fiber. The fiber also showed very good elongation properties, with values increasing upon modifications. Lower elongation of the untreated fiber may be due to the three dimensionally cross-linked networks of cellulose and lignin. Treatment broke this network structure giving the fiber higher elongation and lower strength properties. Mishra et al. (2001) investigated the tensile properties of untreated, chemically modified and AN-grafted sisal fibers. Chemically modified fibers showed an appreciable decrease in the tensile properties. This decrease was attributed to the substantial delignification and degradation of cellulosic chains during chemical treatment. The extension at break of these fibers did not change much. In all the cases of grafting, it has been found that the tensile strengths were higher than that of untreated fiber

2.15. Effects of fiber surface treatments on composite properties

Chemical treatments will be necessary to strengthen the interface between fiber and matrix. Several studies have been conducted on the influence of various types of chemical modifications on the properties of natural fiber-reinforced thermoplastic composites (Mansour et al., 1983; Manrich and Agnelli, 1989; Kenaga et al., 1962). This section reviews the effects of fiber modification on the mechanical properties, thermal properties and macro-mechanical properties of composites.

2.15.1. Mechanical Properties of Composites

The mechanical properties of a natural fiber-reinforced composite depend on many parameters, such as fiber strength, modulus, fiber length and orientation, in addition to the fiber-matrix interfacial bond strength. A strong fiber-matrix interface bond is critical for high mechanical properties of composites. A good interfacial bond is required for effective stress transfer from the matrix to the fiber whereby maximum utilization of the fiber strength in the composite is achieved (Karnani et al., 1997). Modification to the fiber also improves resistance to moisture-induced degradation of the interface and the composite properties (Joseph et al., 2000). In addition, factors like processing conditions/techniques have significant influence on the mechanical properties of fiber reinforced composites (George et al., 2001).

Sapieha et al. (1989; 1990) have found that by the addition of a small amount of dicumyl peroxide or benzoyl peroxide into the cellulosic fiber-polymer (LDPE) systems during processing significantly improved the mechanical properties of the composite. Kokta etal. (1990a; 1990b) studied extensively the effect of different chemical modifications, such as silane treatment and grafting, on the mechanical properties and dimensional stability of cellulosic fiber-thermoplastic composites. They found that the chemically modified cellulosic fiber-reinforced thermoplastic composites offered superior physical and mechanical properties under extreme conditions even after recycling. Ray et al., (2001) have employed the technique on jute and found that the improvements occurred on the fiber properties. Münker and Holtmann (1998) studied different natural fibers (flax, ramie, curaua) and matrices (polyester, polypropylene).Their findings showed that mechanical properties of natural fiber-reinforced composites could be improved by the use of different coupling agents.

2.15.2. Tensile Properties of Composites

Natural fiber-reinforced composites often show enhancement in tensile properties upon different modifications owing to the increased fiber-matrix adhesion. Tensile properties can be explained on the basis of the changes in chemical interactions at the fiber-matrix interface. The tensile strength of flax fiber-reinforced composites is determined both by the tensile strength of the fibers and the presence of weak lateral fiber bonds.

Sreekala et al., (2000) performed one of the pioneering studies on the mechanical performance of treated oil palm fiber-reinforced composites. They studied the tensile stress-stain behavior of composites having 40% by weight fiber loading. Isocyananté-, 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 fiber was attributed to the changes in the chemical structure and bondability of the fiber. Alkali treated (5%) sisal-polyester biocomposite showed about 22% increase in tensile strength (Mishra et al., 2002). Ichazo et al., (2001) found that adding silane treated wood flour to PP produced a sustained increase in the tensile modulus and tensile strength of the composite. Joseph and Thomas (1993) studied the effect of chemical treatment on the tensile and dynamic mechanical properties of short sisal fiber reinforced low density polyethylene composites. It was observed that the CTDIC (cardanol derivative of toluene diisocyanate) treatment reduced the hydrophilic nature of the sisal fiber and enhanced the tensile properties of the sisal-LDPE composites. They found that peroxide and permanganate treated fiber-reinforced composites showed an enhancement in tensile properties. They concluded that with a suitable fiber surface treatment, the mechanical properties and dimensional stability of sisal-LDPE composites could be improved. Mohanty et al., (2000) studied the influence of different surface modifications of jute 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 fiber content also affected the bio-composite performance and about 30% by weight of jute showed optimum properties of the bio-composites.

2.15.3. Impact Properties of Composites

Fibers have a significant effect on the impact resistance through the principle of stress transfer. When an impact load is applied perpendicular to the reinforcing fibers, good fiber-matrix adhesion is required for even moderate impact strength (Nielsen, 1974). The impact properties of the polymeric materials are directly related to the overall toughness of the material (Shah, 1998). Toughness is defined as the ability of the polymer to absorb applied energy. Impact resistance is the ability of a material to resist breaking under a shock loading or the ability to resist fracture under stress applied at high speed.

Detailed studies have already been done on the impact resistance of short fiber reinforced composites (Kau, 1990; Jang et al., 1990; Reed and Bevan, 1993). The impact resistance of fiber-reinforced composite depends on fiber rigidity, interfacial stress resistance and fiber aspect ratio. The strength of the matrix, the weakest part of the material, should be related to the failure process. The involvement of fibers in the failure process is related to their interaction with the crack formation in the matrix and their stress transferring capability. The total energy dissipated in the composite before final failure occurs is a measure of its impact resistance. The total energy absorbed by the composite is the sum of the energy consumed during plastic deformation and the energy needed for creating new surfaces.

2.15.4. Macro-mechanical Properties of Composites

The macro-mechanical properties of composites are attributed to an increase in the interfacial shear strength (ISS) of the modified composites. The ability to control the chemical and mechanical properties of the fiber-matrix inter-phase is crucial. Interface studies of untreated and surface treated sisal-polyester composites has been investigated to determine fiber splitting, fiber pullout, de-bonding, matrix cracking and fiber-matrix interaction using scanning electron microscopy (Mishra et al., 2002). Scanning electron micrographs of the tensile fracture of the composites revealed the failure mechanisms and impact fracture morphology. Fiber breakage was the main failure criteria observed.

They also reported that in the untreated sisal composite, a clean pullout of fibers without any adhering resin matrix was observed. This proved that there was very poor adhesion between fiber and matrix. The 5% alkali-treated sisal composite showed better fiber matrix interaction as observed from the good dispersion of fibers in the matrix system predicting micropores at the interface. Morphological studies showed that the MAPP and silane treatment improved the polymer-wood flour (WF) adhesion and the dispersion of the particles while the alkaline treatment only improved the dispersion (Ichazo et al., 2001).

2.15.5. Thermal Properties of Composites

A quick method for determining the threshold values for processing temperature is done by thermal analysis. Thermogravimetric (TG) analyses are carried out with a thermal balance. The thermo gravimetric degradation curve provides information about the thermal stability of a material (Shah, 1998).

Wielage et al., (1999) used differential scanning calorimetry (DSC) to determine the melting point of the flax-reinforced polypropylene and to collect caloric data. DSC is a thermo-analytical technique in which the heat flow is measured as a function of temperature or time. They subjected the flax-reinforced polypropylene to a defined temperature regime under controlled atmosphere and reported that the melting range of the polymer matrix was displayed as an endothermic peak. An increased heating rate leads to a displacement of the melting range to higher temperatures. Powell et al., (2002) considered the effect of the matrix on the heating of flax fiber-reinforced composites. They reported that pure HDPE showed degradation beginning at approximately 410-430ºC. Therefore, the matrix absorbed a great amount of heat, taking the thermal stress -off of the flax fiber.

2.16. Response Surface Methodology (RSM)

As an important subject in the statistical design of experiments, the Response Surface Methodology (RSM) is a collection of mathematical and statistical techniques useful for the modeling and analysis of problems in which a response of interest is influenced by several variables and the objective is to optimize this response (Montgomery 2005). For example, the growth of a plant is affected by a certain amount of water $x1$ and sunshine $x2$. The plant can grow under any combination of treatment $x1$ and $x2$. Therefore, water and sunshine can vary continuously. When treatments are from a continuous range of values, then a Response Surface Methodology is useful for developing, improving, and optimizing the response variable. In this case, the plant growth y is the response variable, and it is a function of water and sunshine. It can be expressed as $y = f(x1, x2) + e$

The variables *x1* and *x2* are independent variables where the response *y* depends on them. The dependent variable *y* is a function of *x1*, *x2*, and the experimental error term, denoted as *e*. The error term e represents any measurement error on the response, as well as other type of variations not counted in *f*. It is a statistical error that is assumed to distribute normally with zero mean and variance. In most RSM problems, the true response function *f* is unknown. In order to develop a proper approximation for *f*, the experimenter usually starts with a low-order polynomial in some small region. If the response can be defined by a linear function of independent variables, then the approximating function is a first-order model.

If there is a curvature in the response surface, then a higher degree polynomial should be used. The approximating function with 2 variables is called a second-order model:

In general all RSM problems use either one or the mixture of the both of these models. In each model, the levels of each factor are independent of the levels of other factors. In order to get the most efficient result in the approximation of polynomials the proper experimental design must be used to collect data. Once the data are collected, the Method of Least Square is used to estimate the parameters in the polynomials. The response surface analysis is performed by using the fitted surface. The response surface designs are types of designs for fitting response surface. Therefore, the objective of studying RSM can be accomplish by

(1) Understanding the topography of the response surface (local maximum, local minimum, ridge lines), and

(2) Finding the region where the optimal response occurs. The goal is to move rapidly and efficiently along a path to get to a maximum or a minimum response so that the response is optimized. The RSM is important in designing, formulating, developing, and analyzing new scientific studies and products. It is also efficient in the improvement of existing studies and products. The most common applications of RSM are in industrial, biological and clinical science, social science, food science, and physical and engineering sciences. Since RSM has an extensive application in the real-world, it is also important to know how and where Response Surface Methodology started in the history. According to Hill and Hunter, RSM method was introduced by G.E.P. Box and K.B. Wilson in 1951 (Wikipedia 2006). Box and Wilson suggested to use a first-degree polynomial model to approximate the response variable. They acknowledged that this model is only an approximation, not accurate, but such a model is easy to estimate and apply, even when little is known about the

process (Wikipedia, 2006). Moreover, Mead and Pike stated origin of RSM starts 1930s with use of Response Curves (Myers et al., 1989). According to research conducted (Myers et al., 1989), the orthogonal design was motivated by Box and Wilson (1951) in the case of the first-order model. For the second-order models, many subject- matter scientists and engineers have a working knowledge of the central composite designs (CCDs) and three-level designs by Box and Behnken (1960). Also, the same research states that another important contribution came from Hartley (1959), who made an effort to create a more economical or small composite design. There exist many papers in the literatures about the response surface models. In contrast, 3-level fractional design has limited works. Thus, 3- level fractional design is an open research subject. Fractional Factorial Experiment Design for Factor at 3-Levels (Connor and Zelen, 1959) is a helpful resource conducting this kind of design. Many three- level fractional factorial designs and more importantly their alias tables can be found in their study. According to Myers et al., (1989), the important development of optimal design theory in the field of experimental design emerged following Word World II.

One of the important facts is whether the system contains a maximum or a minimum or a saddle point, which has a wide interest in industry. Therefore, RSM is being increasingly used in the industry. Also, in recent years more emphasis has been placed by the chemical and processing field for finding regions where there is an improvement in response instead of finding the optimum response (Myers et al., 1989). Response Surface Methods are designs and models for working with continuous treatments when finding the optima or describing the response is the goal (Oehlert 2000). The first goal for Response Surface Method is to find the optimum response. When there is more than one response then it is important to find the compromise optimum that does not optimize only one response (Oehlert 2000). When there are constraints on the design data, then the experimental design has to meet requirements of the constraints. The second goal is to understand how the response changes in a given direction by adjusting the design variables. In general, the response surface can be visualized graphically.

2.16.1. Box–Behnken Design (BBD)

In statistics, Box–Behnken designs are experimental designs for response surface methodology, devised by George E. P. Box and Behnken in 1960, to achieve the following goals:

- Each factor, or independent variable, is placed at one of three equally spaced values, usually coded as -1, 0, +1. (At least three levels are needed for the following goal.)
- The design should be sufficient to fit a quadratic model, that is, one containing squared terms and products of two factors.
- The ratio of the number of experimental points to the number of coefficients in the quadratic model should be reasonable (in fact, their designs kept it in the range of 1.5 to 2.6).
- The estimation variance should more or less depend only on the distance from the centre (this is achieved exactly for the designs with 4 and 7 factors), and should not vary too much inside the smallest (hyper) cube containing the experimental points.

The design with 7 factors was found first while looking for a design having the desired property concerning estimation variance, and then similar designs were found for other numbers of factors.

Each design can be thought of as a combination of a two-level (full or fractional) factorial design with an incomplete block design. In each block, a certain number of factors is put through all combinations for the factorial design, while the other factors are kept at the central values. For instance, the Box–Behnken design for 3 factors involves three blocks, in each of which 2 factors are varied through the 4 possible combinations of high and low. It is necessary to include centre points as well (in which all factors are at their central values).

2.16.2 Central Composite Design (CCD)

Central composite design is an experimental design, useful in response surface methodology, for building a second order (quadratic) model for the response variable without needing to use a complete three-level factorial experiment.

After the designed experiment is performed, linear regression is used, sometimes iteratively, to obtain results. Coded variables are often used when constructing this design. A central composite design is the most commonly used response surface designed experiment. Central composite designs is a factorial or fractional factorial design with center points, augmented with a group of axial points (also called star points) that let you estimate curvature.

A central composite design can be used to**:**

- Efficiently estimate first- and second-order terms.
- Model a response variable with curvature by adding center and axial points to a previously-done factorial design.

Central composite designs are especially useful in sequential experiments because you can often build on previous factorial experiments by adding axial and center points.

Implementation of central composite design

The design consists of three distinct sets of experimental runs:

1. A factorial (perhaps fractional) design in the factors studied, each having two levels;
2. A set of center points, experimental runs whose values of each factor are the medians of the values used in the factorial portion. This point is often replicated in order to improve the precision of the experiment;
3. A set of axial points, experimental runs identical to the centre points except for one factor, which will take on values both below and above the median of the two factorial levels, and typically both outside their range. All factors are varied in this way.

2.17. Review of previous works

Matoke et al., (2012) worked on effects of production methods on the physical and mechanical properties of recycled plastic – bamboo fiber boards. They used compression molding and open cast method for their production. They studied effect of fiber content and production method on the physical and mechanical properties of the composites. Their findings showed that molded composites showed better physical properties than open cast method, possibly due to reduced void space during polymerization.

Herrera – Franco and valadez – Gonzalez (2005) studied the degree of fiber – matrix adhesion and its effect on the mechanical reinforcement of short *henequen* fibers and a polyethylene matrix. The surface treatments studied were an alkali treatment, silane coupling agent and pre-impregnation process of the HDPE / Xylene solution. They established that the presence of Si-O-cellulose and Si-O-Si bonds on the lignocellulosic surface confirmed that the silane coupling agent was efficiently held on the fibers surface through both condensation with cellulose hydroxyl groups and self-condensation between silanal groups. They used fiber-matrix interface shear strength (IFSS) as an indicator of the fiber-matrix adhesion improvement and also to determine a suitable value of fiber length in order to process the composites with relative ease. They noticed that the IFSS observed for the different fiber surface treatments increased and such interface strength almost doubled only by changing the mechanical interaction and the chemical interactions between fibers and matrix.

Olusegun et al., (2001) worked on the mechanical properties of *Ukam*, banana, sisal, coconut, hemp and E-glass fiber reinforced laminates. The samples were fabricated by the hand lay-up process using 30:70 fiber and matrix ratio by weight. The results showed that glass laminate has the maximum tensile strength of 63MPa, bending strength of 0.5MPa, compressive strength of 37.75MPa and impact strength of 17.82J/m^2. The *Ukam* plant fibre laminate has the maximum tensile strength of 16.25MPa and impact strength of 9.8J/m among the natural fibres, the sisal laminate has the maximum compressive strength of 42MPa and maximum bending strength of 0.003MPa among natural fibres. Their results indicated that natural fibres are of interest for low cost engineering applications and can compete with artificial glass fibres (E-glass fibre) when a high stiffness per unit weight is desirable.

Nystrom et al., (2007) worked on natural fibre composites based on flax and polypropylene prepared by compounding and injection molding. They employed factorial trials and statistical analysis to investigate effects of processing parameters on microstructure and on mechanical performance. The tested parameters in the compounding step were screw speed, mass flow, number of dispersing elements and granule length. Their findings showed that all the parameters influenced the final structure and mechanical properties of the composites although the differences in the material properties were small. The most important parameter turned out to be screw speed and granule length.

Nagaraja and Rekha (2013) studied the potential applications of natural products which are commonly used in the preparation of natural fibre reinforced composites. Composite samples were prepared using untreated, uneven rice straw and chicken feather fibre using general purpose polyester of the resin matrix. They studied the tensile behaviour of the samples prepared with different volumetric proportion of the matrix and fibre. Their studies showed that an increase in the fibre loading decreased the tensile strength till the optimum fibre proportion. The tensile strength of the rice straw fiber reinforced composites showed a decreasing trend till 40% of fibre volume and then increased. Similar behaviour was observed in chicken feather fibre reinforced composites. The hybrid composites (rice straw and chicken feather combination) showed an increasing trend till 30% of fibre volume, then decreased for 40% and again increased.

Soma and Acharya (2014) worked on the effect of stacking sequence on tensile, flexural and inter – laminar shear properties of untreated wooden jute and glass fabric reinforced epoxy hybrid composite. In their study, new hybrid composites with epoxy as a resin and reinforcing both bio waste (jute) and traditional filler (glass) as continuous layered mat composites were considerably improved by incorporating the glass fibre at extreme glass piles.

Thakur and SIngha (2009) worked on the polymer biocomposites based on resorcinol formaldehyde resin matrix reinforced with pine needle fabricated by compression molding technique. Mechanical properties such as flexural strength, tensile strength, compressive strength and wear resistance of pine needle – reinforced phenolic resin matrix based composites were evaluated. They found out that addition of pine needles into the polymeric matrix promoted a significant improvement on the composite properties. They equally evaluated the effect of fibre dimension on mechanical properties of the composites. They observed that polymer composites obtained by particle reinforcement exhibited better mechanical properties compared to short and long fibre reinforcement. Morphological and thermal properties showed that when polymer resin was reinforced with fibres of different dimensions, morphological changes took place. Equally, the result indicated that the presence of lignocellulosic pine needles affects the thermal stability of polymer matrix.

Lu, et al., (2006) worked on the effect of fibre characteristics and polymer melt flow index (MFI) on the mechanical properties of sugar cane fiber/HDPE compostes. They discovered that fibre characteristics (fibre type, morphological and dimension) and polymer melt flow index (MFI) significantly affected the mechanical properties of sugar-cane fibre / HDPE composites. They observed that long fibre had a significant reduction on the dimension and aspect ratio during compounding, however, the short fibres had close values in these two properties before and after compounding. They observed that on sugarcane fibre / polymer composites, the HDPE resins with a low MFI value presented high tensile and impact strengths. Because of high sugar content, the pure rind fibre had a poor performance as filler in the HDPE resins with respect to the raw bagasse fibre and alkali – extracted bagasse fibre. On the other hand, the aspect ratio was found to be proportional to the mechanical performance of the fibres in the HDPE resins. They concluded that fibres with a large aspect ratio and low sucrose content improved the strength properties of the resultant composites.

Nystrom et al., (2003) studied injection molded short natural fibre composites (NFC). Micro-mechanical models were employed to investigate effects of fibre length distribution, fibre orientation, and fibre / matrix adhesion on mechanical performance of NFCs. Their study revealed that length / diameter ratio strongly affected the composites strength, where as fibre strength had very little influence on strength of the composites at poor fibre / matrix adhesion.

Puglia et al., (2007) worked on the analysis of fibre structure variation during the processing of a polypropylene matrix reinforced with cellulose flax pulp for different reinforcement concentrations. They found out that the final properties of composite materials were highly dependent on the residual geometrical parameters (length, diameter, aspect ratio), orientation and distribution of the fibres in the matrix which in turn are related to the processing conditions. They established that the fibre interfacial area after the process decreased as the fibre concentration increased and this evaluation explained how the adhesion methods that were used for fibre surface modification fail because of the decrement on the modifier interfacial density.

Ishak et al., (2010) compared the mechanical properties of short kenaf bast and core fibre reinforced unsaturated polyester composites. The composites were prepared using compression molding techniques. The results showed that the composites reinforced with kenaf bast fibre had higher mechanical properties than kenaf core fibre composites. The result equally showed that the optimum fibre content for achieving highest tensile strength for both bast and core fibre composites were 20% wt. They observed that the elongation at break for both composites decreased as the fibre content was increased. They observed for flexural strength, the optimum fibre content for both composites was 10% while for impact strength, it was 10% wt and 5% wt for bast and core fibre composites respectively.

Weager et al., (2010) researched on composite materials derived from natural renewable sources. They discovered that natural fibre reinforced composites had received significant interest in recent years due to increased environmental awareness, concern about the depletion of non renewable resources and instability in oil prices. They found out those natural fibres such as hem, flax and wood have already been used as composites reinforcements in particular in the automotive industry to reduce weight, cost and environmental impact. However, their findings suggested that the short and randomly oriented nature of the fibre limited the mechanical properties of the resultant composite parts.

Chang Geun (2012) worked on pretreatment and fractionation of lignocellulosic biomass for production of biofuel and value added product. He treated the biomass with zinc chloride and found out that it has swelling effect and selectivity for hemicelluloses, hence, most of the hemicelluloses was released into the liquid hydrolysates by zinc chloride treatment.

Sugumaran (2013) used fiber to enhance the physical and mechanical properties of wood composite. He treated the fiber with nitric acid and found out that the treatment increased the mechanical properties of the composite.

Oladele et. al., (2010) investigated the effect of chemical treatment on the constituents and tensile properties of sisal fiber. They treated the fiber with KOH, acetic acid, sodium chloride, and hydrochloric acid. They observed that the chemicals were successful in removal of lignin and hemicelluloses components, thereby increasing the tensile strength of the fiber.

Vautard et. al., (2012) studied the influence of an oxidation of the fiber surface by boiling nitric acid on the adhesion strength in carbon fiber-acrylate composite cured by electron beam. They observed that oxidation of the fiber surface in boiling nitric acid created a rough surface which significantly increased the specific surface area, and also generated a high density of hydroxyl groups, carboxylic acids and lactones in compariism to untreated fibers. This enhanced the mechanical properties of the composite.

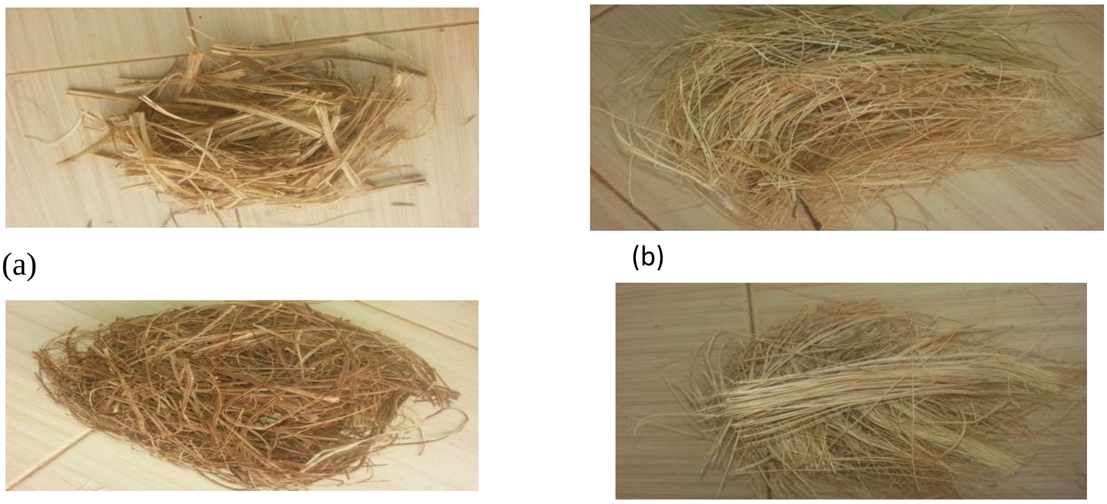
CHAPTER THREE

MATERIALS AND METHODS

3.1. Materials

3.1.1. Natural Fibers

Natural plant fibres (Fig. 3.1); Nwogbe (*Ampelocissus cavicaulis*), Usoro (*adenia lobata*), Ogbuebo (*morinda morindoides*) and Okpaowoko (*ampelocissus leonensis*) of the species of different plants were obtained from Olo, in Ezeagu L. G.A of Enugu State, Nigeria.



(a) (b)

(c) (d)

Fig. 3.1. Natural fibers (a) *Ampelocissus cavicaulis*, (b) *Adenia Lobata*, (c) *Morinda Morindoidies*, (d) *Ampelocissus Leonensis*

3.1.2 Thermoplastic (High Density Polyethylene)

High density polyethylene (HDPE) pellets were obtained from De Cliff Integrated Company, Enugu.

3.1.3. Chemicals

The chemicals; sodium hydroxide, acetic anhydride, acetic acid, nitric acid and zinc chloride used for chemical treatment were of analytical grade, and were purchased from De Cliff Integrated Company, at Ogbete main market, Enugu.

3.2. Extraction of fibers

These plant fibres were extracted mainly from the plant stems using water retting extraction process, giving fibres of different length and diameters. The obtained plant stems were cut and sliced longitudinally into four pieces and each was completely submerged in water for two weeks, after which the stems were removed from the water and loosened by smashing. Bundles of fibers were further placed in water for one week at room temperature to remove impurities and facilitate their separation. The extracted fibers were finally washed thoroughly in excess tap water and air dried at room temperature for three days. Before tensile testing, the fibres were visually selected in order to verify the absence of defects along the length of the fibres.

3.3. Chemcal compositions of fibers

3.3.1. Moisture Content Determination

The fibres were weighed and dried in an oven at the temperature of 103°C for 30 minutes, followed by cooling for 30 minutes and then weighed again. This step is repeated until the weight is constant. The moisture content of the test specimen was expressed as a percentage loss in weight of the final oven-dry weight using the equation;

$$\text{Moisture content} = \frac{W0 - \mathrm{W}1}{\mathrm{W}0} \text{ x } 100 \qquad (3.1)$$

Where W_0 is weight of fibre before drying in the oven and W_1 is weight of fibres after drying in the oven.

3.3.2. Ash content

The ash content determination was according to the work done by Thygesen et al. 2007. The ash content was determined by burning 5g of the fiber placed in a platinum crucible in a muffle furnace at 600^{o}C for 30minutes. The percentage of the ash content was determined using the equation (3.2).

$$\text{Ash content } = \frac{w_3 - w_2}{w_1} \qquad (3.2)$$

Where, w_2 is the weight of the porcelain crucible, w_3 is the weight of the porcelain crucible and fiber burnt to ashes and w_1 is the initial weight of the fiber

3.3.3. Wax content

The wax determination was done according to work done by Thygesen et al. (2007). 4g of each of the fibers was inserted into a thimble and put in a soxhlet reflux flask and mounted unto an oil extraction flask containing 300 millimeters of petroleum ether. The upper end of the reflux flask was connected to a condenser and was air tight. With the aid of non flame bearing heating mantle, the solvent in the flask was heated and it condensed into the reflux flask containing the thimble. The solvent was kept in contact with the fiber samples until the reflux flask was filled up and siphoned over, thus carrying extracted oil or fat down to the boiling flask. This process was repeated for about four hours with not less than fourteen refluxes. Then, the defatted fiber samples were carefully removed, dried in an oven at 80^{o}C for 30minutes, cooled in desiccators and reweighted. The weight loss due to fat extracted was obtained as the weight of the fat or oil or wax using the below equation

$$\text{Fat/wax content (\%)} = \frac{w_4 - w_5}{w_1} \text{ x } 100 \tag{3.3}$$

Where, w_4 is the weight of thimble and fiber before defatting, w_5 is the weight of thimble and fiber after defatting and w_1 is the initial weight of the fiber.

3.3.4 Determination of Lignin Content by Gravimetric Method

This procedure was in accordance with the work done by Onyeagoro (2012). 2.0g of the sample was weighed and placed inside a beaker. 72% H_2SO_4 was added and allowed to stand for 2 hours. 8% H_2SO_4 was later added and the solution refluxed for 3 hours. The residue was filtered with purpling cloth and washed severally with hot water. A crucible was weighed with the sample into it. The sample was oven dried at 110^0C for 1 hour and then cooled inside desiccators after which the weight was taken. The sample was ashed in a furnace at 500^0C for 3 hours. It was then cooled inside desiccators and finally weighed. The % lignin was calculated using equation (3.4)

$$\% \text{ Lignin} = \frac{W_2 - W_1}{W_s} \text{ x } 100 \tag{3.4}$$

Where,

W_1 = weight of ash sample + crucible

W_2 = weight of oven dried sample + crucible

Ws = initial weight of dried sample

3.3.5. Determination of Hemicellulose Content

Hemicelluloses content determination was according to work done by Thygesen et al. (2007). Hemicellulose was extracted by soaking 10g of the sample in 100mls of 8.5% sodium hydroxide solution for 24hours at room temperature and was filtered. The hemicellulose was precipitated by acidifying it with acid and ethanol. The precipitate was washed after filtration and dried over night and was weighed after cooling in a dessicator. The hemicelluloses content was calculated using the equation below.

$$\text{Hemicelluloses content (\%)} = \frac{w_3 - w_2}{w_3} \text{ x } 100 \tag{3.5}$$

Where, w_3 is the initial fiber weight before extraction, w_2 is the final fiber weight after extraction.

3.3.6 Determination of Cellulose Content

The remaining residue after hemicelluloses extraction was washed, dried, cooled in a dessicator and weighed as the cellulose fraction. The cellulose fraction was expressed as the percentage of the dry matter.

3.4. Methods of chemical treatment of the fibers

The fibers were subjected to chemical treatments such as mercerization with sodium hydroxide, acetylation with acetic anhydride, oxidation with nitric acid and zinc chloride treatment. This was done to improve fiber-matrix interaction for superior mechanical properties of the resulted composites. The experiments were done at Pymotech Research Center and Laboratories, number 6 convent avenue Abakpa Nike, Enugu State of Nigeria

3.4.1. Alkali Treatment

This procedure was in accordance with work done by Nural and Ishak (2012). The washed fibers were treated with 6% NaoH. The fibers were immersed in the alkali solution for 50 minutes, then neutralized with acetic acid and washed with distilled water repeatedly until all sodium hydroxide was eliminated. Finally, the fibers were washed with distilled water and dried at room temperature for 48h

3.4.2 Acetic Anhydride Treatment

The acetylation process was in accordance with work done by Bledzki, et al., (2008). The fibers were soaked in distilled water for an hour, filtered and placed in a round bottom flask containing 10% acetic acid solution for 30 minutes. After which it was placed on flask containing 14% acetic anhydride solution. The process temperature of acetylation was 30^0C and duration was 70 minutes. After modification, the fiber was washed periodically with distilled water until acid free. Finally, modified fibers were air dried for certain time before analysis.

3.4.3 Nitric Acid Treatment

The nitric acid treatment was according to Vautard et al., (2013). The size reduced fibers were oxidized with 6% nitric acid. The prepared oxidizing solution was boiled to a temperature of 60^0C and the fiber immersed in the solution at the said temperature for 50 minutes. After, they were neutralized with NaOH solution and washed with distilled water repeatedly until all the nitric acid was eliminated. Finally, the fibers were washed again with distilled water and dried to a constant weight at room temperature.

3.4.4 Zinc Chloride Treatment

Zinc chloride treatment was done in accordance with the work done by Nadanthangam et al., (2013). The fibers were soaked in 3% zinc chloride solution for 70 minutes after which it was washed with distilled water until the washing solution became chloride free. The fibers were washed with distilled water and dried at room temperature for 48 hours.

3.5. Determination of the physical and mechanical properties of the fibers

3.5.1. Diameter Determination

This test was performed as follows. Electronic digital caliper of linear capacity measuring system was used to measure the diameter of the fibres. At least twenty single fibre strands of all the different plant fibre species were measured and the average diameter was calculated using equation;

$$D = \frac{D1+\mathrm{D}2+\cdots\mathrm{D}20}{20}, \qquad (3.6)$$

where D is the fibre diameter.

3.5.2. Aspect ratio

The aspect ratios of the fibers were calculated from the average diameter using ASTM (D1037-12) equation below.

$$\text{Aspect ratio} = \frac{\text{Fiber length}}{\text{Fiber diameter}} \qquad (3.7)$$

3.5.3. Density of the fibers

This test was performed as follows. Archimedes principle was used to determine the density of the treated and untreated fibers accordin g to work done by Morton, 1980. In this method, the samples of the fibers were selected and bound into a bundle and its mass measured on a digital weighing balance with a resolution of 0.0001g. The volumes of these fixed mass of the fibers were measured using standard displacement technique (Gohil and Shaikh, 2010).

In this method, the weighted fiber bundles were immersed in 100mls measuring cylinder filled with water. The displaced volume was measured with a 10mls capacity measuring cylinder. The density was determined by dividing the mass of the fiber bundle by its displaced volume using equation (3.8) below. The process was observed for both the treated and untreated fibers.

$$\text{Density (g/cm}^3\text{)} = \frac{\mathrm{M}}{\mathrm{V}} \qquad (3.8)$$

Where M is the mass of the fiber bundle and V is the volume of the fiber

3.5.4. Water absorption

This test was performed according to ASTM (D1037-12). Humidity chambers (desiccators) were set up at 100% humidity using distilled water. The specimens were prepared from bundles of single fibers bound together. The specimens (untreated, alkaline treated, acetic anhydride treated, nitric acid treated and zinc chloride treated) were dried in a hot air oven at 60°C for 24h, weighed in a balance accurate to 4 decimal places and were then placed in a humidity chamber. The humidity chamber was held at room temperature. After 24h, each fiber bundle was taken out from the humidity chamber and excess water was carefully mopped with filter paper. Finally, the weight of the fibers was measured and the water absorption was calculated by weight difference. Then again the sample is immersed in water and the same process was repeated after every 24h.

$$\text{Water absorption (\%)} = \frac{\mathrm{ma} - \mathrm{md}}{\mathrm{md}} x100 \qquad (3.9)$$

Where ma is the mass of the sample after exposing to water and md is the mass of the dry sample.

3.5.5. Determination of tensile strength

The experiments were done at the Civil Engineering Department, University of Nigeria Nsukka, Enugu State Nigeria. Hounsfield universal testing machine (specifications; trade name: Tensometer limited 81 Morland Rad, Cryodon England, make: Hounsfield universal tensometer, maximum load, 2000KgF, precision of measurement: BSS 1610, grade AI, maximum tonnage: 0.025cm/min-100cm/min, maximum temperature: room temperature, model number: 8889) was used to determine the tensile strength, and elongation at break of the specimen according to

ASTM (D1037-12). After finding the average diameter (D) of the fibres, the cross sectional area of each individual fibre was determined using the relation

Cross sectional area, $A = \pi r^2$, where r = radius (3.10)

After this, they were glued in between two frames to assure a good gripping and straight position in the test clamps. The opening of the frame determines the gauge length; for this experiment it was set at 50 mm.

The single fibre tensile tests were performed on a mini tensile testing unit of the Hounsfield Tensometer machine (Fig. 3.2) with a load cell of 200N where the crosshead speed was set at 1 mm/min; the load and the displacement were registered during the complete test. A rotating drum on which special graph sheet was rapped was fixed to the side of the equipment. A manually operated rotating handle was used for loading the specimen under test. With the help of an adjustable mercury level and a manually operated marker, the load and extension values of the specimen were recorded on the graph sheet at a regular interval until failure occurred. The tensile strength was calculated from this relation

$$\text{Tensile Strength (Mpa)} = \frac{\text{Maximum load (N)}}{\text{Area } (\mathrm{mm}^2)} \qquad (3.11)$$

The tensile modulus was calculated from the slope of force vs. extension using below relation

$$\text{Tensile Modulus (Mpa)} = slope\ of\ PX\ \left(\frac{L_0}{A_0}\right) \qquad (3.12)$$

Where P is the Force (N), X is the extension (mm), L_o is the initial length of the sample, and A_o is the area of the sample

Fig. 3.2. Hounsfield Tensometer

3.5.6. Determination of Elongation at break

This test was performed according to ASTM (D1037-12). Elongation at break helps one to know how an object stretches before breaking. It is crucial for designing machine that operates under stressful conditions so that they do not elongate so much that they no longer have enough strength to withstand the forces while elongated.

$$\text{Elongation at break (\%)} = \frac{\text{Final length} - \text{Initial length}}{\text{Initial length}} \times 100 \qquad (3.13)$$

3.6. Preparation of fiber- polymer composite

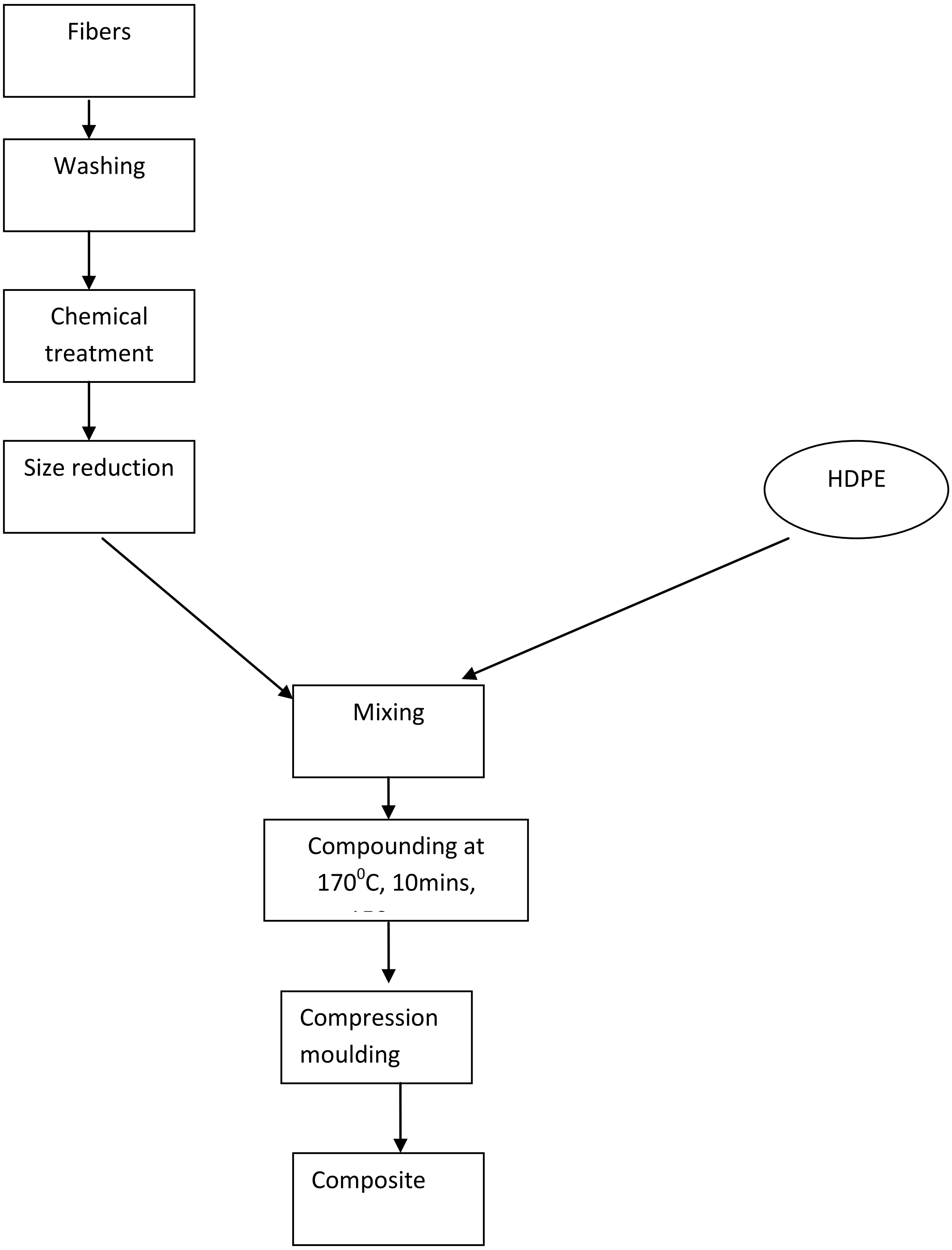


Fig. 3.3. Processing scheme of fiber-reinforced HDPE composites

The schematic overview of processing scheme of the fibers reinforced polymer composite is shown in Figure 3.3.

3.6.1 Compounding Process

The compounding process was done at Rubber Technology Department, Rubber Research Institute Benin, Nigeria. A method of Lu et. al., (2000) was used for the compounding process. The treated and untreated fibers were separately mixed with HDPE at a specified fiber loading ratio. The mixture of the fibers and HDPE were fed into two roll mill (Fig. 3.4) with specifications (mixing capacity: 0.2-5kg, roller speed ratio: 1:1.22, temperature of measurement: ± 3.5^{o}C, maximum temperature 260^{o}C, maximum speed: 960r/min, roll gap: 4mm, model: 35100, make: Aaronequipment company Literbven 1-7, 2740 Stovlunde Denmark) and were mixed with heating. This step was performed to avoid the separation of fiber from the polymer during the molding process. The rotor speed, compounding temperature and the compounding time were varied to establish the optimum conditions. After compounding, the resultant blend was introduced into the compression molding system for compression molding.

Fig. 3.4. Two roll mill for the compounding process

3.6.2 Compression Molding Process

3.6.2.1 Fiber reinforced composites

Compression molding process was done at Pymotech Research Center and Laboratories, number 6 convent avenue Abakpa Nike, Enugu State Nigeria using a locally fabricated compression molding machine (Fig. 3.5) with specifications; 32 tons hydraulic jack, pressure gauge range : 0-140 bar, temperatue range: 0-400^{o}C. This method used was in accordance with the work done by Vijay Kumar and Amar

Singh (2009). Blends from the compounding process were poured into specially made moulds. The surfaces of moulds were smeared on the inside with oleic acid to avoid adhesion of the mixture on the mould and to allow easy removal of the composites. The mixture was then spread equally on the surface of the moulds. Composite sheets where prepared by compression molding using locally fabricated compression molding machine. In compression molding the material charge was pressed between two halves of mould and allowed to transform into a solid product. The mold was pressed with the help of jack at 170^0C for 10mins and cooled down to room temperature. The pressure for heating and cooling were controlled between 10 to 50 bars and was monitored using a pressure guage attached to the machine.

Fig. 3.5. Compression molding machine

3.6.2.2 Effect of fiber loading

To study the effects of fiber loading, six various percentages of the fiber loading were used along with control sample (Virgin polymer) with 0.0wt% fiber content. Fiber loading used were; 5wt%, 10wt%, 20wt%, 30wt%, 40wt%, and 50wt% fiber content by mass to reinforce the thermoplastics composites. The procedure was as described above.

3.6.2.3 Effect of fiber lengths

To study the effect of fiber length, the fibers were cut into different lengths of 0.5mm, 1mm, 2mm, 3mm, 4mm, 5mm and 6mm and mixed with a known quantity of the polymer. Composite sheets were prepared as per method discussed above in a random oriented pattern.

3.6.2.4 Effect of molding temperatures

Six molding temperatures were selected for compression molding bearing in mind the melting point of the polymer, the recommended processing temperature and the decomposition temperature of the polymer. The molding temperatures selected were; 140^0C, 150^0C, 160^0C, 170^0C, 180^0C, 190^0C. Temperature effect was studied at two different constant times of 5minutes and 10minutes.

3.6.2.5 Effect of molding times

To study effect of moulding time, five different molding times were selected and studied at two different temperatures of 140^oC and 170^oC. They were 5mins, 10mins, 13mins, 16mins, and 20mins while cooling time was constant at 10mins. The molding process was as described on section 3.6.2.1.

3.6.2.6 Effect of molding pressure

Four molding pressures were selected based on preliminary work done. The moulding pressures were 10bar, 20bar, 30bar, 40bar and 50bar. The molding process was as described on section 3.6.2.1.

3.7. Determination of the mechanical properties of the composites

3.7.1 Determination of Tensile Strength

The Hounsfield universal Tensometer testing machine England (Fig. 3.2) (specifications; trade name: Tensometer limited 81 Morland Rad, Cryodon England, make: Hounsfield universal tensometer, maximum load, 2000KgF, precision of measurement: BSS 1610, grade AI, maximum tonnage: 0.025cm/min-100cm/min, maximum temperature: room temperature, model number: 8889) was used to determine the tensile strength, hardness test, Flexural strength, and elongation at break of the composites according to ASTM (D638-99). The samples were cut into (100x15x4) mm3 after which, they were clipped in between two frames to assure a good gripping and straight position in the test clamps. The tensile tests were performed on a mini tensile testing unit of the Hounsfield Tensometer machine with a load cell of 2500N where the crosshead speed was set at 1 mm/min; the load and the displacement were registered during the complete test. A rotating drum on which special graph sheet was rapped was fixed to the side of the equipment. A manually operated rotating handle was used for loading the specimen under test. With the help of an adjustable mercury level and a manually operated marker, the load and extension values of the specimen were recorded on the graph sheet at a regular interval until failure occurred. The tensile strength was calculated from this relation

$$\text{Tensile Strength (Mpa)} = \frac{\text{Maximum load (N)}}{\text{Area }(\mathrm{mm}^2)} \qquad (3.13)$$

The tensile modulus was calculated from the slope of force vs. extension using below relation

$$\text{Tensile Modulus (Mpa)} = \text{slope of PX } \left(\frac{L_0}{A_0}\right) \qquad (3.14)$$

Where P is the Force (N), X is the extension (mm), L_o is the initial length of the sample, and A_o is the area of the sample

3.7.2. Determination of Elongation at Break.

Elongation at break helps one to know how an object stretches before breaking. It is crucial for designing machine that operates under stressful conditions so that they do not elongate so much that they no longer have enough strength to withstand the forces while elongated.

$$\text{Elongation at break (\%)} = \frac{\text{Final length} - \text{Initial length}}{\text{Initial length}} \text{ x } 100 \qquad (3.15)$$

3.7.3. Flexural Tests

After the composite preparations, the composite samples were cut to dimensions of (300x19x3.2) mm^3 and were tested for flexural properties using universal testing machine (Hounsfield Tensometer 8889) (Fig. 3.6) according to ASTM (D790-02). The samples were centered on the supports with the long axis of the sample perpendicular to the loading nose and supports. The load was carefully applied to the sample and flexural strength and modulus were calculated from the maximum bending load using the relation below

Flexural Strength (Mpa) = $\frac{\text{Maximum bending force (N)}}{\text{Area of composite (mm}^3\text{)}}$ (3.16)

Flexural modulus (Mpa) = $\frac{\text{PL}}{\text{bd}^2}$ (3.17)

Where p is the maximum bending load, b is the width of the composites, L is the span length, and d is the depth of the composites.

Bending at break of the composites were obtained as the deflection at bending force

Fig. 3.6. Flexural testing Machine

3.7.4. Impact Test

This test was done at Mechanical Enginerring Department, University of Nigeria Nsukka. The test was done using an impact testing machine (Fig. 3.7) (specifications: trade name: Losenhausenwerk Dusseldorfer Germany, model: IT 406, make: Maschinenbau AG Dusseldorf, maximum load: 6.031Kg, precision: 0.01kgFm, maximum temperature: room temperature, maximum tonnage, 180kg) according to ASTM (D1822-93). The pendulum impact testing machine ascertains the notch impact strength of the material by shattering the V notched (158^{o}) specimen with a pendulum hammer, measuring the spent energy, and relating it to the cross section of the specimen. The machine was adjusted such that the blade on the free- hanging pendulum just barely contracts the specimen (zero position). The specimens were clamped in a square support and were struck at their central point by a hemispherical bolt.

The respective values of impact energy of different composites were recorded from the scale.

Fig. 3.7. Impact Testing Machine

3.7.5. Hardness Property

The brinnel hardness measurement was done using universal testing machine (Hounsfield Tensometer 8889) England (Fig. 3.8) according to ASTM (D2240-02). A 10mm diameter bulb indenter was placed on the samples of dimension 100x15x4 mm^3. A constant load of 72.45N was applied and the level of penetration recorded as depth of indentation. The hardness numbers of the samples were calculated using equation (3.18)

$$\text{Brinnel hardness number (HB)} = \frac{P}{\frac{2\pi}{D}(D^2 - \sqrt{D^2} - d^2)} \qquad (3.18)$$

Where P is the constant axial force, D is the brinnel bulb diameter, and d is the depth of indentation.

Fig. 3.8. Brinnel hardness testing Machine

3.8. Determination of the physical properties of the composites

3.8.1. Density

The densities of the fibers reinforced composites were determined according to ASTM D 792. The samples were weighed using digital weighing balance, while the dimensions of the samples were measured using a vernier caliper from which the volume were calculated. The density was then calculated from the following relationship.

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} \qquad (3.19)$$

3.8.2 Water Absorption

The water absorption test was carried out according to ASTM D570 -99. It involved total immersion of the samples in distilled water at room temperature. All the samples were previously dried in an oven at 50°C for 24hrs and then stored in a dessicator. Moisture uptake was determined by weighing the specimens after immersion for 24hrs. The moisture content was calculated according to the equation below

$$(\mathrm{Mt}) = \frac{\mathrm{W_w} - \mathrm{W_d}}{\mathrm{W_d}} \times 100 \qquad (3.20)$$

Where W_d and W_w are the weight of the samples when dry and with moisture respectively.

3.8.3 Thickness Swelling

The method used was in accordance with ASTM standard (D1037-03). This test, like water absorption was important in ascertaining dimensional changes. The thickness swelling samples were 75mm x 50mm x 20mm in dimension. The samples were soaked in distilled water for 24hours. The immersed samples were taken out and wiped with dry cloth to remove water from the surface. The thickness was measured using a vernier caliper to the nearest 0.01 along the length at room temperature and average results recorded. The thickness swellings of the samples were calculated using equation (3.21).

$$(\mathrm{Ts}) = \frac{\mathrm{T_F} - \mathrm{T_I}}{\mathrm{T_I}} \times 100 \qquad (3.21)$$

Where T_F is the final thickness after immersion and T_I is the initial thickness before immersion.

3.8.4 Chemical Resistance

The chemical resistance of the samples was studied using ASTM D543-87. The effects of seven chemicals, i.e. water, 0.5N NaOH, 20%ammonium hydroxide, 1N HCl, acetic acid, methanol, and benzene were studied on the fiber reinforced composite as well as the polymer. In each case, the pre-weighed sample was dipped in the respective chemicals for 24hour. They were then removed and immediately washed in distilled water and dried by pressing them on both sides with filter paper at room temperature. The samples were then weighed, and the percentage weight loss/gain was determined. The Percent weight loss/gain was determined using the following equation.

$$\text{Wight loss (\%)} = \frac{\text{Final Weight} - \text{Original Weight}}{\text{Original Weight}} \times 100 \qquad (3.21)$$

3.9. Surface characterization

SEM and FTIR were done at Chemical Enginnering Department, Ahmadu Bello University, Zaria Kaduna State Nigeria.

3.9.1. Scanning electron microscopy (SEM) analysis

Scanning Electron microscropy (SEM) of the fibers and the composites were carried out using Scanning Element Microscope, Fig. 3.9 shown below (specifications; magnification range: 20-120X, maximum temperature: 30^{o}C, maximum humidity: <80%RH, make: Phenom world, Dillenburgstraat 9E, 5652 AM Eindoven, The Netherlands, model: Phenom proX, line scan resolution range: 16-512 pixels, mapping resolution range, 16x16-512x512 pixels, maximum image resolution: 2048x2048 pixels). Analysis was carried out on the untreated, treated fibers, uncompounded blend, compounded blend and the composite to study their surface textures.

Fig.3.9. Phenom ProX Scanning Element Microscope

3.9.2. Fourier Transform Infrared Spectroscopy

The surface functional groups and structure were studied by fourier transform infrared spectroscopy, (specifications; make: Shimadzu Corporation, International Marketing division, 3, Kanda-Nishikicho, 1-Chrime, Chiyoda-ku Japan, model: FTIR-8400S, signal to noise ratio: 20,000:1, wave number range: 7800 to 350cm^{-1}, wave number resolution: 0.85cm^{-1}, peak to peak resolution: 4cm^{-1} per minutes, temperature range: 15-30^{o}C, humidity: <70%). The FTIR spectra of the untreated fibers, treated fibers, and the composites were scanned at a wavelength of 500–4000nm to obtain its spectra lines.

3.10. Statistical analysis and optimization

Central composite design was used to optimize the chemical treatment conditions for chemical treatments of natural fiber. Box Benhken design (BBD) was used to optimize the compounding conditions and molding conditions. CCD is characterized by three operations namely; 2n axial runs, 2^n factorial runs and six center runs. The total number of experiments is 2^n + 2n+ nc where n is the number of factors, nc is the number of center points (Ejikeme et al, 2013). CCD involved three numeric factors and one categoric factor. The addition of categoric factor multiplied the total number of the experiments by the level of the factor; in this case we have 13x4 = 52 experiments each for each fiber. The factors and levels used for the CCD are shown on Table 3.1 while the design matrix is shown on Table 4.3.

Table 3.1 Factors and levels for chemical treatments of the fibers using CCD

Factors	Units	Levels				
		-α	-1	0	+1	+α
Chemical strength	%	2.0	6.0	10.0	14.0	18.0
Treatment time	Minutes					
Type of chemicals 1. Sodium hydroxide 2. Acetic anhydride 3. Nitric acid 4. Zinc chloride						

Box Behnken Design (BBD) was used for the optimization of the compounding and molding conditions. This design was used because relatively few experimental combinations of the variables are adequate to estimate potentially complex response functions (Annadurai and Sheeja, 1998). The compounding process has three numeric factors giving a total of 17 experiments for each fiber treated with four different chemicals. The factors and levels for the design are shown on Table 3.2 while the design matrix is shown on Table 4.12.

Table 3.2 Factors and levels for compounding process using BBD

Factors	Units	Levels		
		-1	0	+1
Rotation Speed	Rpm	60.0	80.0	100.0
Temperature	oC	160.0	170.0	180.0
Time	Minutes	10.0	15.0	20.0

BBD was equally used to optimize the molding conditions. It involved three numeric factors and one categoric factor. The categoric factor has five levels which multiplied the 17 experiments from the three factors giving a total of eighty-five (85) experiments for each fiber. The factors and levels for the BBD are shown on table 3.3 below.

Table 3.3 Factors and levels for the molding process using BBD

Factors	Units	Levels		
		-1	0	+1
Temperature	^{o}C	150.0	170.0	190.0
Time	Minutes	5.0	10.0	15.0
Pressure	Bar	10.0	30.0	50.0

Fiber conditions

1. Untreated
2. Sodium hydroxide treated
3. Acetic anhydride treated
4. Nitric acid treated
5. Zinc chloride treated

CHAPTER FOUR

RESULTS AND DISCUSSION

4.1 Chemical composition of natural fibers

To better understand the properties of natural fibre-reinforced composite materials, the chemical and mechanical properties of the natural fibres used were analysed. Natural fibre consists of cellulose, hemicelluloses, lignin, wax, water content and water soluble substances. These compositions differed with the different species of the plant.

The chemical structure of cellulose is the same for all natural fibres, but the variations in the cellulose content as seen on Table 4.1 was as a result of the degree of polymerization or length of the polymer chains of the cellulose. The degree of polymerization of cellulose has a significant effect on the mechanical properties of a fibre as it is directly related to the molecular weight (Mohanty et al., 2000). When the cellulose has a higher degree of polymerization and molecular weight, the fibre will have greater strength properties (Mohanty et al., 2000).

Table 4.1. Chemical Compositions of natural fibres:

S/N	Sample fibre	Common name	Celluose (%)	Hemi cellulose (%)	Lignin (%)	Ash content (%)	Wax (%)	Moisture content (%)
1	*Ampelocissus leonensis*	Okpaowoko	43.22	28.221	34.910	0.570	0.01	5.50
2	*Ampelocissus cavicaulis*	Nwogbe	48.967	21.221	31.33	2.43	0.21	0.514
3	*Adenia lobata*	Usoro	55.20	9.321	28.22	2.572	0.08	3.21
4	*Morinda morindoides*	Ogbuebo	55.76	8.621	31.22	2.457	0.5	0.48

This statement was confirmed in Table 4.2, where *morinda morindoides* that had highest cellulose content had highest tensile strength.

From Table 4.1, it can be seen that *Morinda morindoides* had highest cellulose content, followed by *Adenia lobata*, *Ampelocissus cavicaulis*, and lastly *Ampelocissus leonensis*. The order of their cellulose content shows the order of their degree of polymerization, thus explaining the order of their effectiveness when used in composite production.

Hemicelluloses is a fully armorphous branched polymer with a selection of sugar molecules as monomeric unit (Cutter, 2008). They have significantly lower molecular weight with low degree of polymerization making it weak polymer when compared to cellulose (Pott, 2004). From Table 4.1, it can be seen that fibres with high cellulose content had low hemicellulose content and high tensile strength while those fibres with high hemicellulose contents, had low cellulose content with attendant low tensile strength. This was linked to the fact that hemicellulose has low degree of polymerization which is directly related to the tensile strength.

Lignin is the most complex polymer among naturally occurring high molecular weight material. It functions as a structural support material in plant by filling the spaces between the cellulose and hemicelluloses region cementing them together and decreasing the permeation of water across the cell walls (Cutter, 2008).

The aromatic nature of lignin, high carbon and high hydrogen content makes it hydrophobic (Mohanty et al., 2000; Pott, 2004). Fiber with high lignin content, had low moisture content and that with low lignin content, had high moisture content.

4.2. Mechanical properties of natural fibers

The ultimate tensile strength of natural fibres is the maximum stress that the fiber can withstand while being stretched or pulled before failing or breaking (Wikipedia, 2012). From Table 4.2, it was observed that the fibres had high tensile strength in decreasing order of *Morinda morindoides* → *Adenia lobata,* → *Ampelocissus cavicaulis*→ *Ampelocissus leonensis*. Though their tensile strengths are lower than that of glass fibre, their low densities gave them comparable value of specific strength. Their high tensile strength was as a result of high degree of polymerization of the cellulose content of the fibre. This means that the fibers are ductile and can experience plastic deformation and possible necking before fracture.

The elastic modulus was used to ascertain the stiffness of the fibers. The elastic moduli of the fibres in Table 4.2 were high, except that of *Ampelocissus leonoesis*. Ampleocissus cavcaulis was nine (9) times stiffer than Ampelocissus leonensis, while *morinda morindoides* was twenty eight (28) times stiffer than *Ampelocissus leonensis*.

Elongation at break is the maximum possible extension of the fibre until it breaks. Elastic elongation is of decisive importance because composite products without elasticity would hardly be usable. The result obtained showed that the fibres must be able to deform in order to withstand high loading and must also return to shape. *Ampelocissus leonensis* had lowest tensile strength and highest Elongation at break.

Table 4.2. Mechanical Properties of Natural Fibres:

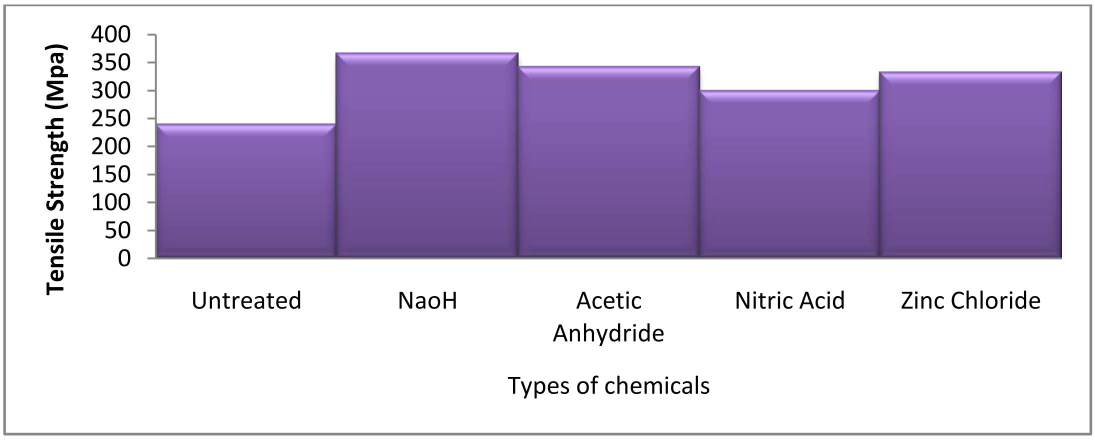
S/N	Fibre type	Density (g/cm^3)	Tensile Strength (Mpa)	Elastic Modulus (Mpa)	Ultimate Elongation at Break (%)
1	*Ampelocissus leonensis*	1.14	67.65	422.81	8.0
2	*Ampelocissus cavicaulis*	1.28	238.28	3,971.33	3.0
3	*Adenia lobata*	1.41	588.94	2,030.82	6.75
4	*Morinda morindoides*	1.58	1202.79	11946.22	5.03

4.3. Effect of chemical treatment on tensile properties of the fibers

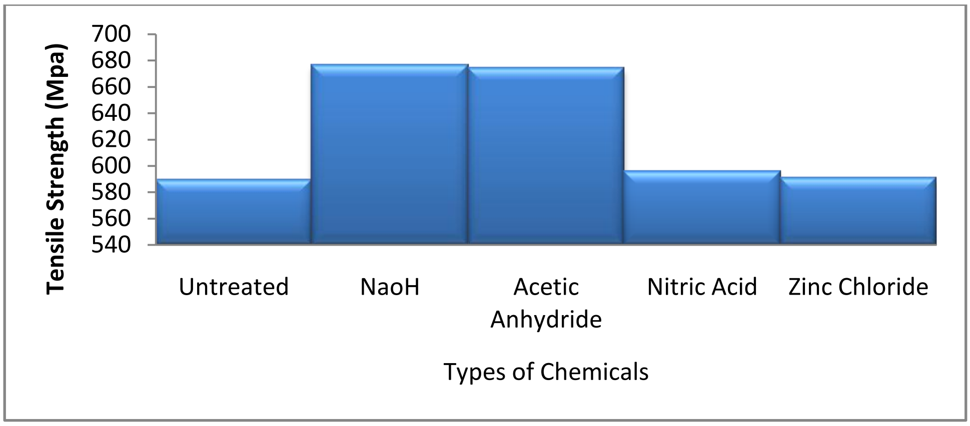
Natural fibre-reinforced composites could offer specific properties comparable to those of conventional fibre composites, however low interfacial properties between fiber and polymer matrix often reduce the potential of natural fibers as reinforcing agents (Mohanty et al., 2001). Interfaces play an important role in the physical and mechanical properties of composites (Joseph et al., 2000). In order to improve natural fiber-matrix adhesion, the fiber should be commonly modified to better fibre surface properties (Wang, 2004). In this study, chemical treatments were applied to the fiber with the aim of changing surface tension and polarity through modification of fiber surface (Scandola et al., 2000). Some of the shortcoming and limitations of natural fibers when used as reinforcement for composites are related to the lower strength properties, lower interfacial adhesion and poor resistance to moisture absorption, lower durability and dimensional stability (shrinkage and swelling). To overcome the shortcomings, various chemicals like NaOH, acetic anhydride, nitric acid and zinc chloride were used to treat the fibers before using them as reinforcement. The chemicals type used were of different efficiencies for improving the mechanical properties of the fibers with resultant improvement on various properties of the final composite products. From this study, it has been seen that the efficiency of the treatment depends not only on the type of the chemicals used, but equally on the concentration of the chemicals as well as the length of treatment time. Effect of different processing variables like chemical types, chemical concentration and time of treatment has been seen to induce variations in the physical properties of the fibers.

4.3.1. Effect of chemical types on the tensile strength of the fibers

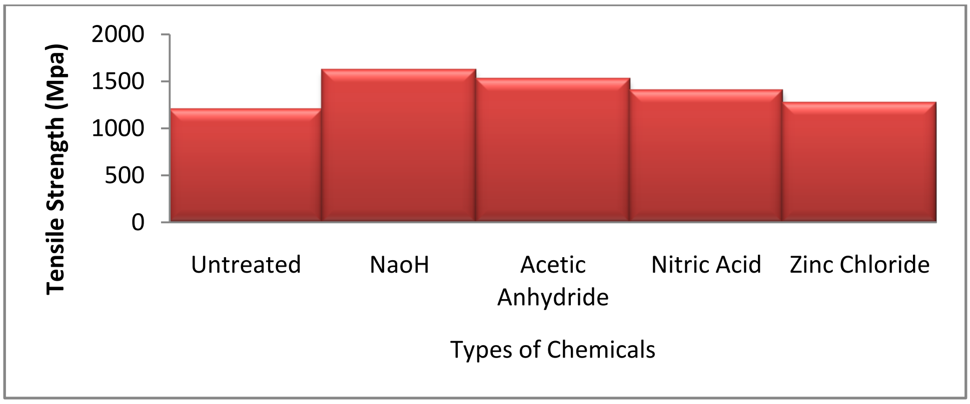
The effect of different types of chemical used on the tensile strength of the fibers was studied to understand the magnitude of variation resulted due to chemical types used. The chemical effects were studied at optimum chemical concentrations and 50 minutes pretreatment time. Fig. 4.1 shows the tensile strength of all the fibers treated with different chemicals with the untreated sample serving as control.



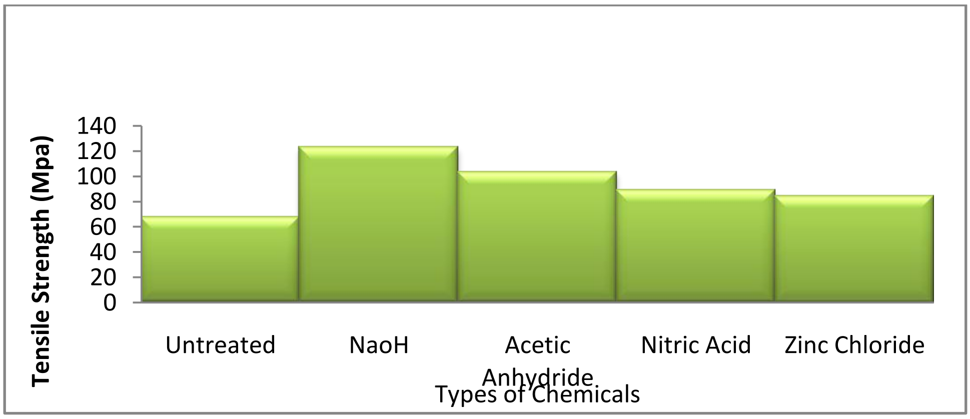
(a)



(b)



(c)



(d)

Fig. 4.1**.** Variations of tensile strength with different chemical types; (a) *Ampelocissus cavicaulis,* (b) *Adenia lobata* (c) *Morinda morindiodes* (d) *Ampelocissus leonensis*.

It is clear from the plots that sodium hydroxide had highest effect on the tensile strength of all the fibers studied. This was as a result of improved fiber –matrix adhesion which resulted from the removal of cementing substances like Lignin and hemicellulose during the treatment. NaOH treated fibers has its attendant problems like fibrillation which causes the breaking down of the composite fiber bundle into smaller fibers resulting in reduction in fiber diameters and overall weight loss (Bei Wang 2004). All the chemical treated fibers had improved tensile strength compared to the untreated fibers. Natural fibers composed of cellulose, hemicelluloses, lignin and other extractives. Cellulose gives the strength, stiffness and structured stability of the fiber, and is the major framework components of the fiber. Lignin and hemicelluloses produce the adhesive to hold the cellulose framework structure of the fiber together, reducing the permeation of substances to the cellulose. The chemical treatment used resulted in the removal of certain portions of hemicellulose, lignin and other extractives covering materials. As a result, fiber surface became cleaner as evidenced in the SEM analysis (4.17). In other words, finer surfaces become more uniform due to the elimination of micro voids and thus stress transfer capacity between the ultimate cells improved. This resulted to higher tensile strength when compared with the untreated fibers.

4.3.2. Effect of sodium hydroxide treatment on the tensile strength of the fibers

Sodium hydroxide (NaOH) is the most commonly used chemical for bleaching and/or cleaning the surface of plant fiber (Mwaikambo and Ansell 1999).

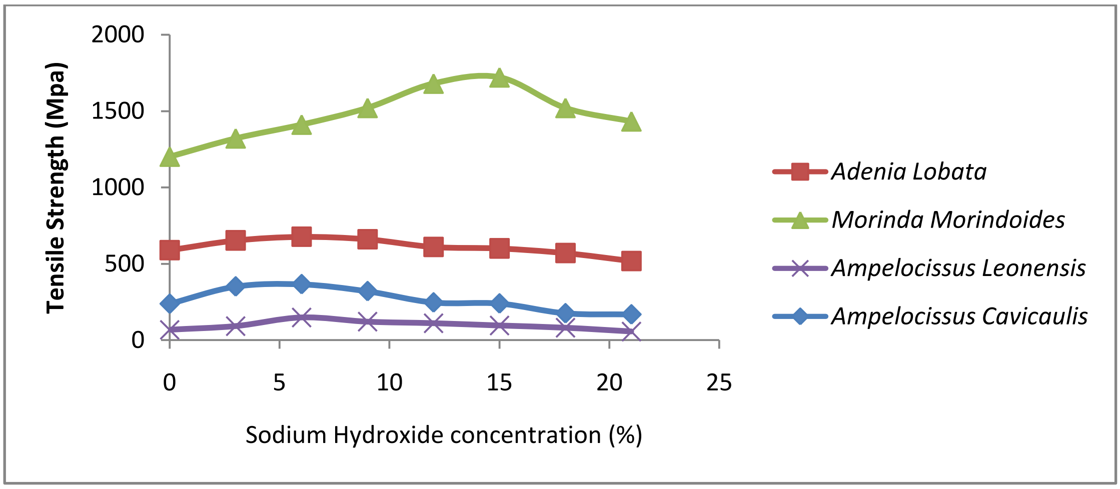


Fig. 4.2. Effect of NaOH concentration on the tensile strength of the fibers.

The standard definition of mercerization proposed by ASTM D1695 is the process of subjecting a vegetable fiber to the action of a fairly concentrated aqueous solution of a strong base so as to produce greater swelling with resultant changes in the fine structure, dimension, morphology and mechanical properties.

Zeronian (1985) proposed another definition of mercerization which is suitable for basic research and is more specific. Mercerized cellulose is a sample of cellulose which has been treated with a solution of an alkali metalhydroxide of sufficient strength to cause essentially complete conversation of the crystal structure from cellulose I to II. He reported that residual traces of cellulose I are found even when the strength of the alkali used in the mercerization treatment are considered optimum for conversation. In that case, no matter the strength of condition of mercerization used there cannot be total conversion. The resultant increase in the tensile strength of the alkali treated fibres was as a result of the delignification of the fiber.

Alkali treatment increased the surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on fiber surface (Wang 2004). Thus alkali treatment increases the number of possible reaction sites and allows better fiber wetting. Reaction which took place during this treatment is as follows:

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{fiber} - \text{ONa}^{+} + H_2O \qquad (4.1)$$

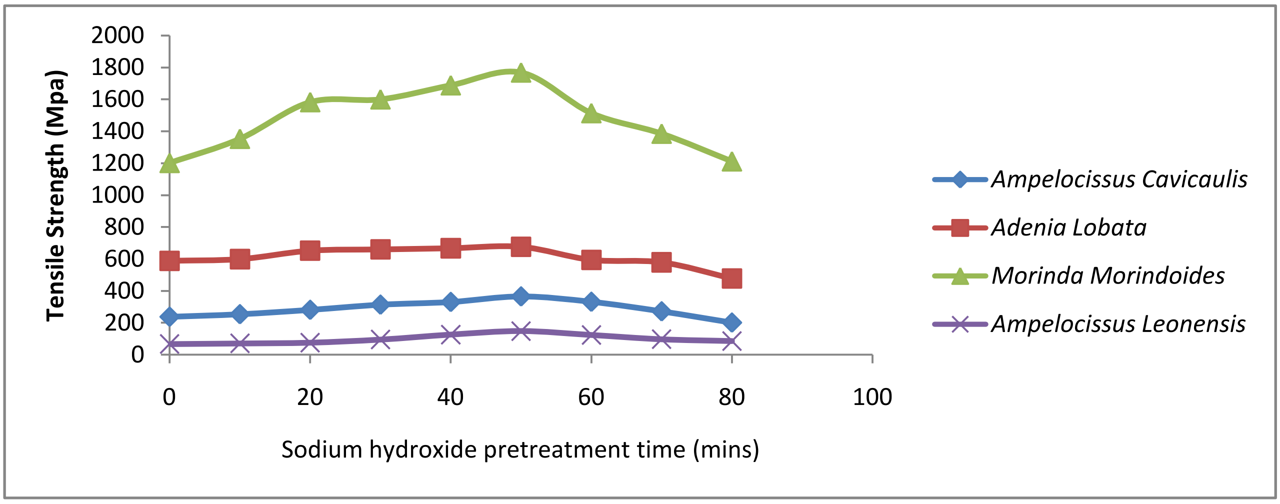


Fig. 4.3. Effect of pretreatment time on the tensile strength of the fibers at optimum concentrations

The type of alkali treatment, its concentration and time of pretreatment influence the degree of swelling and degree of lattice transformation into cellulose, there by affecting the final mechanical properties of the resultant composite.

When the alkali treatment was higher than the optimum conditions, excess delignification of the fibre took place and resulted in the weakness or damage of the fibre which resulted to reduction on the tensile strength of the treated fiber. Fig. 4.4 shows the relative effects of NaOH on the tensile strength of the fibers. It showed that the percentage effect on the tensile strenght was more on *Ampelocissus leonensis* followed by *Ampelocissus cavicaulis*, *Morinda morindoides* and lastly on *Adenia lobata* fiber. This was as a result of different physical and chemical compositions of the fibers

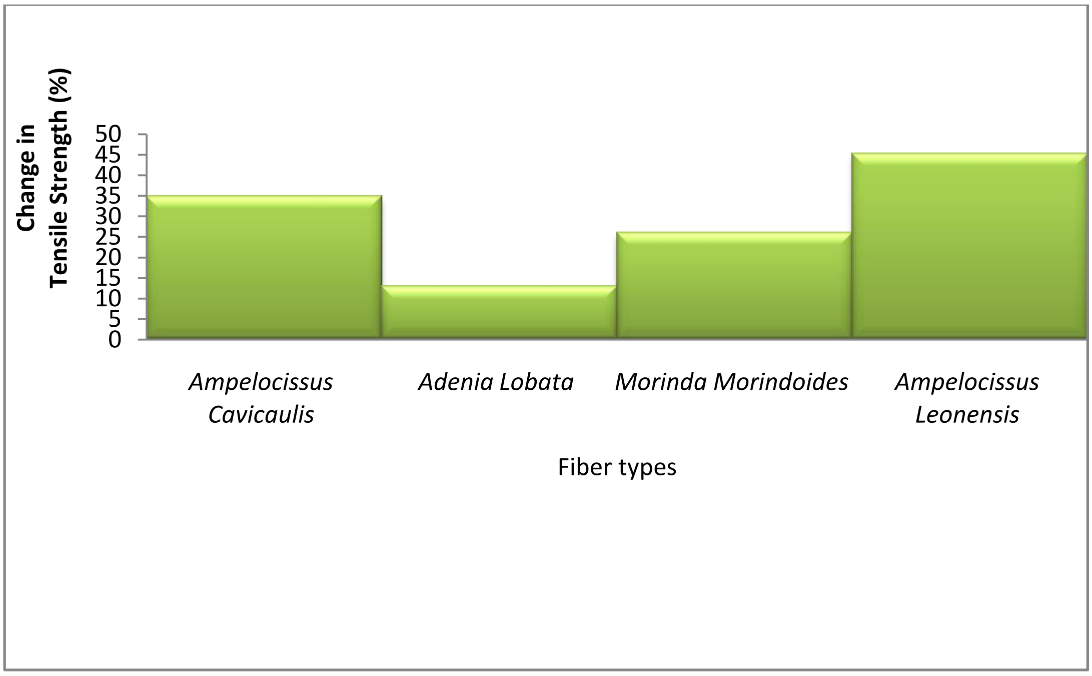


Fig 4.4. Percentage effect of NaOH treatment on the tensile strength of the fibres

It was evidenced that the tensile strength of the treated fiber increased to a certain level, and decreased drastically. The optimum conditions for the treatment were different for the four types of fibers used as a result of their different physico-chemical compositions. This was in line with different works done by different researchers. Jacob etal (2004) examined the effect of NaOH concentration (0.5, 1, 2, 4 and 10%) for treating sisal fiber reinforced HDPE composite and concluded that maximum tensile strength resulted from the 4% NaOH treatment at room temperature.

4.3.3. Effect of acetic anhydride treatment on the tensile strength of the fibers

Acetylation treatment on natural fiber is known as esterification method for plasticizing of cellulose fibers. During acetylation treatment there is an introduction of acetyl functional group (CH_3C00^{-1}) into the fiber. Chemical modification with acetic anhydride substituted the polymer hydroxyl groups of the cell wall with acetyl groups, modifying the properties of these polymers so that they become hydrophobic (LI et al., 2007). This reaction generated acetic acid as by product which was removed from the fibres before their use.

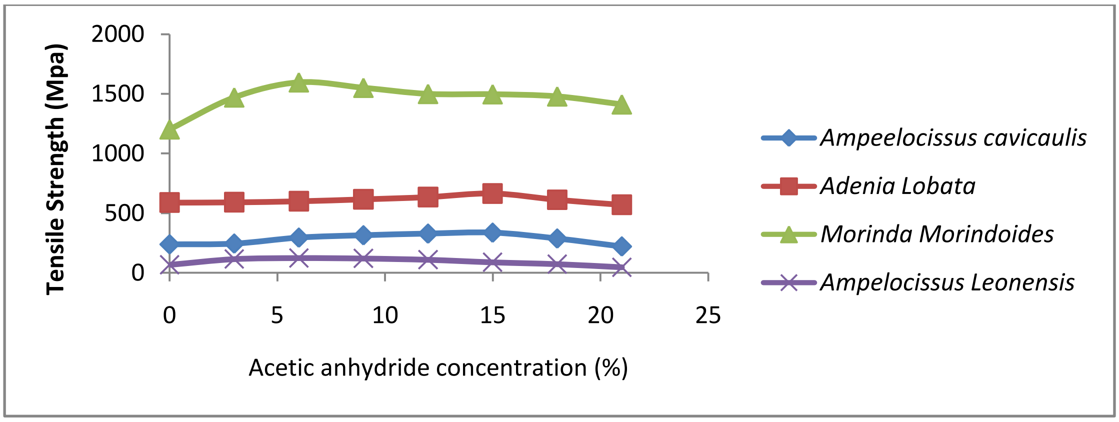


Fig. 4.5. Effect of concentration of acetic anhydride on the tensile strength of the fibers treated for 50mins.

The reaction of acetic anhydride with fiber is as shown below;

Fiber – OH + CH_3 – C (=0) –O – C(= O) CH_3 ➔ fiber – $OCOCH_3$ + CH_3COOH (4.2)

The preliminary treatment done confirmed that acetic anhydride alone cannot sufficiently treat fibres, therefore the fibers were treated with 10% acetic acid for 30mins before treatment with different concentrations of acetic anhydride for different time intervals. It can be seen from Figure 4.5 that as the concentration of acetic anhydride was increased, the tensile strength also increased. The same trend was observed in Figure 4.6. This is because the reaction has succeeded in replacing the hydroxyl groups of the cell wall with acethyl groups thereby making the fiber more hydrophobic. This also enhances stronger interfacial bonding and ultimately increases the tensile strength.An increase above the optimum brings about decrease in the tensile strength .This is because at very high concentration, there is bulking action of the acethyl groups on the fiber cell walls causing a smoother fiber surface (Rana et al 1997) which discourages strong interfacial bonding. In Figure 4.6 as the treatment time is increased the tensile strength is increased. This is because of the fact that at long treatment time all available polymer hydroxyl groups are reached and able to be replaced with acethyl groups.Prolonged exposure brings about bulking action.

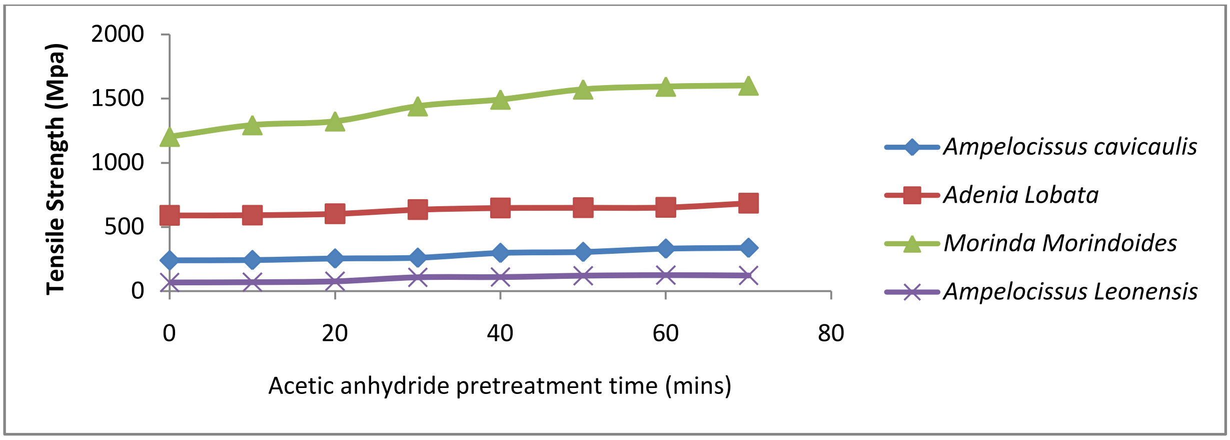


Fig. 4.6. Effect of pretreatment time on the tensile strength of the fibers treated at their optimum concentrations.

Fig. 4.7 shows the relative percentage effect of acetic anhydride on the tensile strength of the fibers. It can be seen that acetic anhydride had 34.79% increases on the tensile strength of *Ampelocissus leonensis*, followed by *Ampelocissus cavicaulis* with 30.32%. Increase, *Morinda Morindodies* with 21.08% increase and lastly *Adenia lobata*, with 12.46% increase.

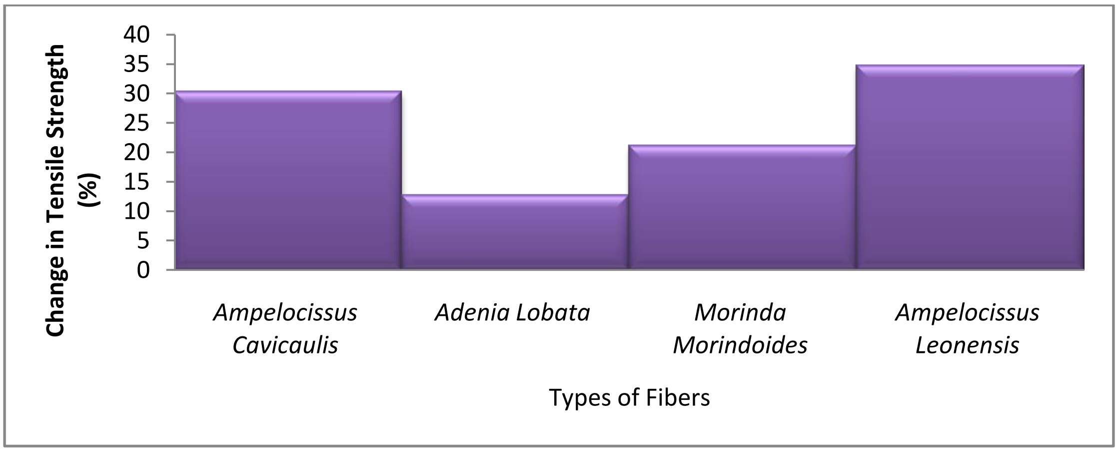


Fig. 4.7. Effect of acetic anhydride treatment on the fibers

4.3.4. Effect of nitric acid treatment on the tensile strength of the fibers

The natural fibers were treated with nitric acid and subsequently analyzed for the tensile strength to develop an understanding of the property changes and how it will affect the composite. The preliminary work done showed no appreciable increase in the tensile strength of the fiber when treated at room temperature; therefore the treatment was done at higher temperature of 60^{0}C. Treatment of fiber with nitric acid is called oxidation method and it introduces an oxidizing group to the surface of the natural cellulose fiber.

According to Vautard et al. (2013), the oxidation of fiber surface in a boiling nitric acid created a rough surface which significantly increased a specific surface area, and also generated a high density of hydroxyl groups, carboxylic acids and lactones in comparison to the untreated fibers. These changes resulted in increase in tensile strength of the treated fibers. Figures 4.8 and 4.9 show the effect of concentration of nitric acid and pretreatment time on the tensile strength of the fibers, respectively.

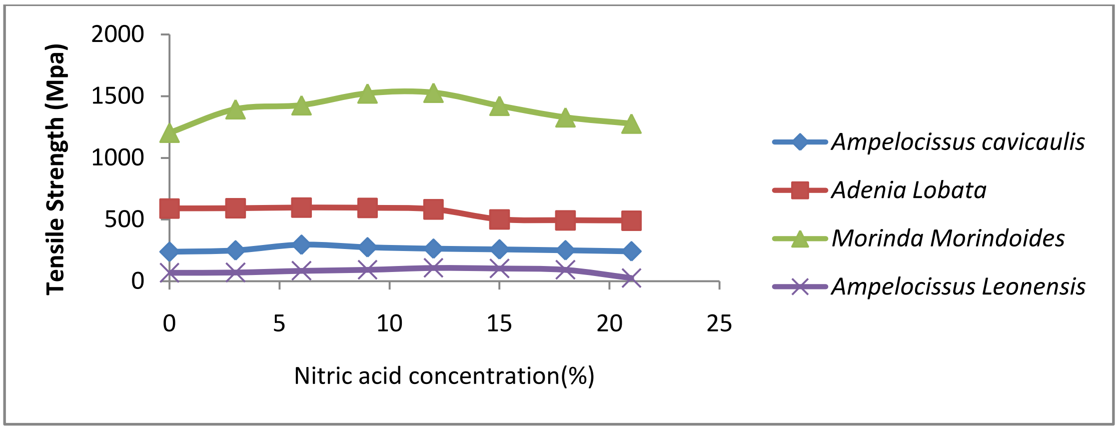


Fig. 4.8. Effect of nitric acid concentrations on the tensile strength of the fibers.

From the result, there was increase in the tensile strength of the fiber as the concentration and pretreatment time was increased till the optimum conditions before it started decreasing. This was attributed to the increase on the quantity of surface functional groups on the surface of the fibers which thus enhanced the ability of the fibers to establish strong interaction between their molecules.

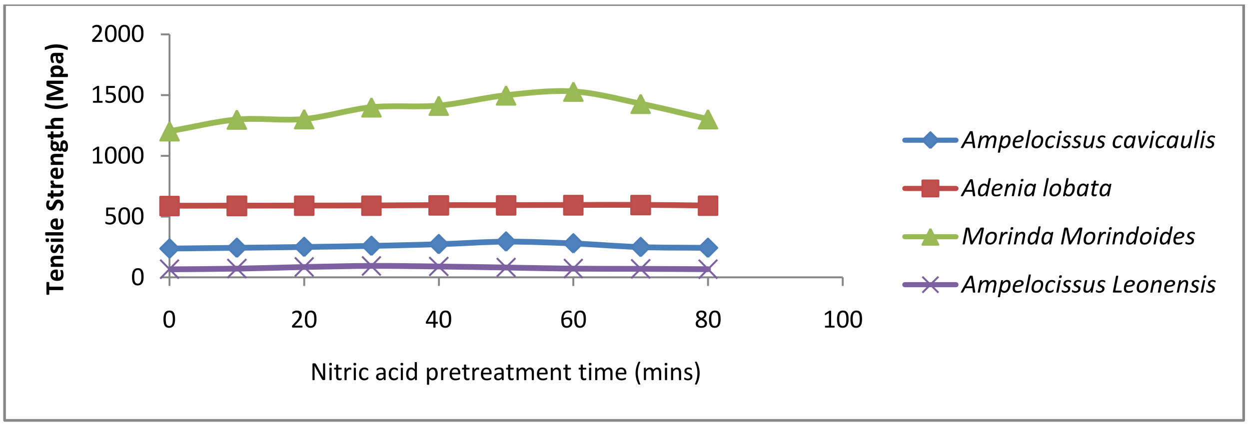


Fig. 4.9. Effect of Nitric Acid pretreatment time on the tensile strength of the fibers

Figure 4.10 shows the percentage effect of nitric acid on all the fibers. It was seen that nitric acid had intense effect on *Ampelocissus leonensis* with 25.881% effect, followed by *Ampelcossus cavicaulis*, *Morinda morindoides* and lastly on *Adenia lobata* with 1.16% effect.

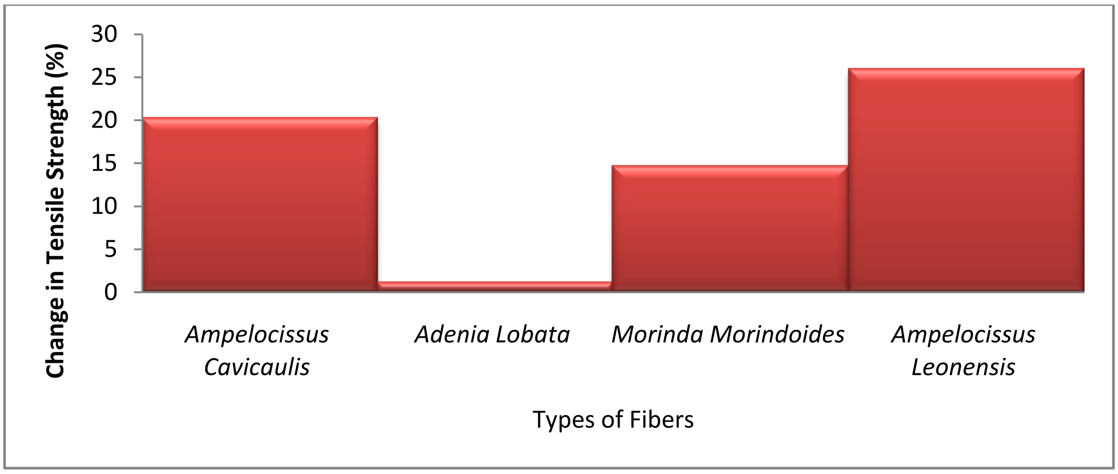


Fig. 4.10**.** Percentage effect of nitric acid on the fibers

The discrepancies found were as a result of the variations on the physiochemical composition of the fiber.

The value range of measurement uncertainties found for the samples were due to the heterogeneous chemical nature of the fibers which caused more profound changes on some fibers better than others. Zheng et al., (2007) attributed increase in the tensile strength of the oxidized fiber to number of acidic functional groups introduced to the fiber aiming to improve the fiber strength through a combination of increased acid base interactions, chemical bonding and / or enhanced mechanical interlocking on the fiber surfaces.

4.3.5. Effect of zinc chloride treatment on the tensile strength of the fibers

Zinc chloride treatment on natural fibers removes natural and human impurities that are non cellulosic constituents and other unwanted substances and increases the tensile strength and the affinity of the cellulose for finishes (Sunita and Shanhnaz, 2013). Zinc chloride equally act as a swelling agent there by increasing the accessibility of the cellulose to the secondary layer (Nadanthangam et al., 2012).

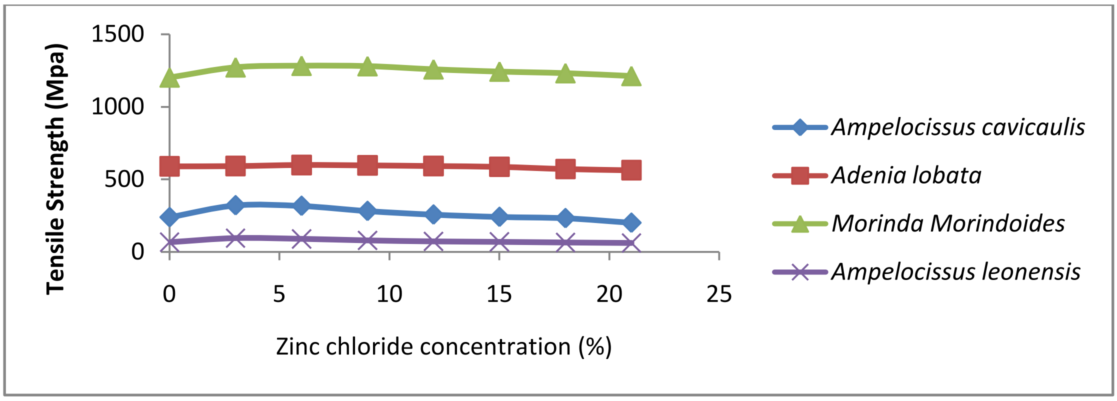


Fig. 4.11. Effect of Zinc Chloride Concentrations on the tensile strength of the fibers

When fiber was treated with zinc chloride, the cellulose became partially gelatinous and bounds the fibers together within and between the pies, thereby increasing the tensile strength of the fibers.

Zinc chloride is known to have destructive effect on fiber especially when used at high concentration and longer pretreatment time (Nadanthangam et al 2012).This was evident on the results of the analysis shown on Figures 4.11 and 4.12. It was noticed from Figures 4.11 and 4.12 that shorter treatment time and lower concentration increased the tensile strength of the treated fiber because of the cleansing and swelling effect of zinc chloride. But deviation from these optimum conditions led to decrease in tensile strength. This can be attributed to the destructive effect of zinc chloride on the fiber due to high concentration and long pretreatment time. Equally, excess delignification can reduce the tensile strength of the fibers.

Fig. 4.13 shows the percentage effect of zinc chloride on all the fibers. It was seen from the bar plot that *Ampelocissus cavicaulis* fiber had 27.98% increase on the tensile strength, 20.08% increase for *Ampelocissus leonensis* fiber, 5.89% increase on *Morinda morindoides* fiber and lastly, 0.3% increase on *Adenia lobata* fiber. The variations on the result were as a result of the fiber compositions.

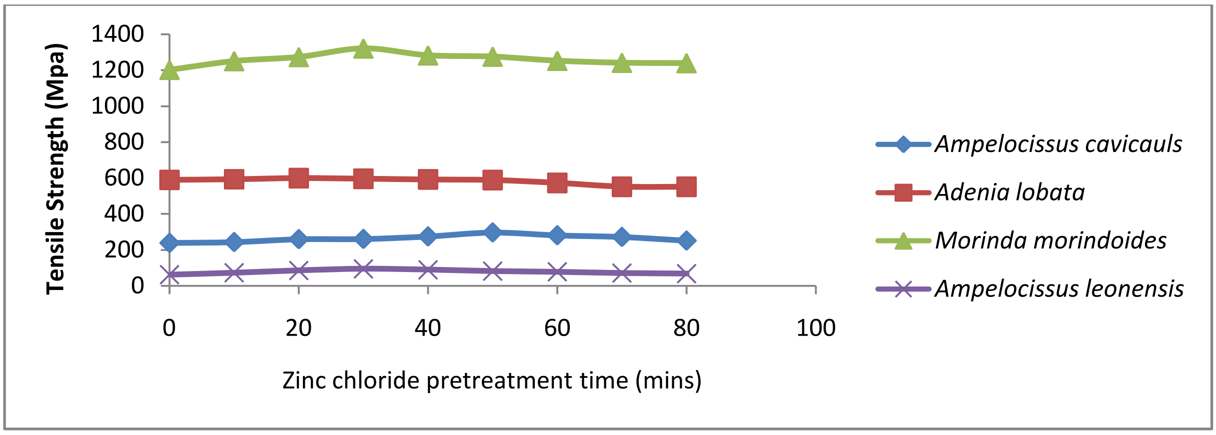


Fig. 4.12. Effect of Zinc chloride pretreatment time on the tensile strength of the fibers.

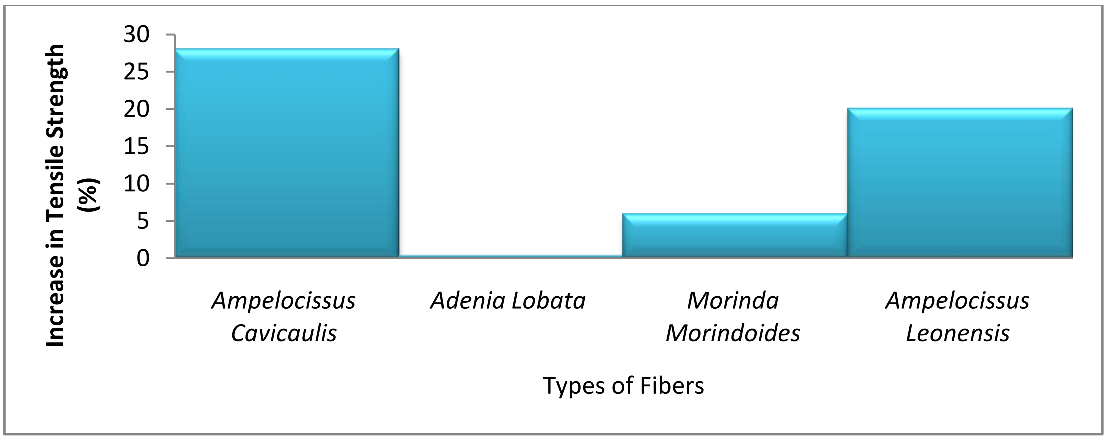


Fig. 4.13. Percentage effect of Zinc Chloride on the Fibers

4.4 Optimization of Chemical Treatment of Fiber using RSM

The optimization was done using central composite design (CCD) with full factorial core encompassing two numerical factors (chemical concentration and time) and one categorical factor (chemical type). With the categorical factor added, the experimental runs were multiplied by the number of the categoric factor making the experiment fifty-two runs for each fibre type.

Table 4.3. Design matrix with the experimental values for all the chemical treatment process

Std Order	Run Order	Chemical concentration (%)	Time (Mins)	Chemical type	Tensile Strength *Ampelo Cavicaulis*	Tensile Strength (Mpa) *Adenia lobata*	Tensile Strength (MPa) *Morinda Morindorides*	Tensile Strength (MPa) *Ampelo Leonensis.*
11	1	10.00	70.00	NaOH	330.176	750	1740.88	95.385
27	2	6.00	50.00	Acetic Anhydride	318.4	609.39	1402.4	100.225
8	3	10.00	110.00	NaOH	315.936	590	1200	70.16
38	4	10.00	70.00	Acetic Anhydride	329.52	639	1200	94.21
7	5	10.00	30.00	NaOH	328.088	690.39	1720.8	107.65
32	6	18.00	70.00	Acetic Anhydride	340.8	647.27	970.48	83.765
5	7	2.00	70.00	NaOH	321.576	630	1113.56	150
35	8	10.00	70.00	Acetic Anhydride	328.296	647.33	1001.52	90.165
46	9	10.00	30.00	Zncl	319.912	509	1195.88	73.66
4	10	14.00	90.00	NaOH	416	580.85	1800	80.16
40	11	6.00	50.00	Zncl	319.016	612.72	1128	84.36
9	12	10.00	70.00	NaOH	305.6	751	1593.28	84.625
3	13	6.00	90.00	NaOH	320.208	652.56	1596.8	125.055
37	14	10.00	70.00	Acetic Anhydride	328.312	600.22	1120.44	92.605
28	15	14.00	50.00	Acetic Anhydride	328.632	632.44	920.8	81.71
48	16	10.00	70.00	Zncl	316.176	461.33	1048.44	63.66
36	17	10.00	70.00	Acetic Anhydride	306.04	640.44	1072.8	96.16
47	18	10.00	110.00	Zncl	202.168	400.72	488	39.11
52	19	10.00	70.00	Zncl	330.224	498.22	912	56.165
16	20	6.00	90.00	Nitric Acid	316.576	600	1193.2	87.86

33	21	10.00	30.00	Acetic Anhydride	327.496	620.3	1074.08	95.055
41	22	14.00	50.00	Zncl	240.416	450.87	879.92	56.26
34	23	10.00	110.00	Acetic Anhydride	329.6	540	801.56	79.195
17	24	14.00	90.00	Nitric Acid	205.848	568.33	958.48	79.385
6	25	18.00	70.00	NaOH	378.288	570	1780	78.5
49	26	10.00	70.00	Zncl	336	465.22	1250.12	50.39
29	27	6.00	90.00	Acetic Anhydride	320.632	614.98	1400.84	99.16
45	28	18.00	70.00	Zncl	220.712	422.78	803.16	49.385
19	29	18.00	70.00	Nitric Acid	193.864	560.39	920	77.5
30	30	14.00	90.00	Acetic Anhydride	336.256	650	960.44	82.56
13	31	10.00	70.00	NaOH	331.464	700	1040	85.11
24	32	10.00	70.00	Nitric Acid	240.176	584.01	1082	89.915
1	33	6.00	50.00	NaOH	316.264	640.32	1400.88	144.66
12	34	10.00	70.00	NaOH	320.784	732	1760	75.19
39	35	10.00	70.00	Acetic Anhydride	318.976	649.22	1170.4	86.165
10	36	10.00	70.00	NaOH	240	600	1600	84.4
15	37	14.00	50.00	Nitric Acid	208.576	570.36	967.12	81.165
43	38	14.00	90.00	Zncl	208.096	411.32	674.88	41.165
2	39	14.00	50.00	NaOH	408	600	1797.28	83.665
20	40	10.00	30.00	Nitric Acid	320	580.32	1200	85.11
14	41	6.00	50.00	Nitric Acid	314.024	595.12	1180.44	90.155
31	42	2.00	70.00	Acetic Anhydride	312.8	590	1600	110
23	43	10.00	70.00	Nitric Acid	244.616	582.66	1128	86
25	44	10.00	70.00	Nitric Acid	216.72	592.31	840.44	49.26
44	45	2.00	70.00	Zncl	314.304	590.72	960	95.16

42	46	6.00	90.00	Zncl	34`.048	607.34	1420	72.61
18	47	2.00	70.00	Nitric Acid	312.576	590.07	1161.28	87.5
50	48	10.00	70.00	Zncl	270.488	467.33	1200	61.005
22	49	10.00	70.00	Nitric Acid	238.664	580.92	1161.28	87.64
26	50	10.00	70.00	Nitric Acid	249.376	587	1082.24	85.26
51	51	10.00	70.00	Zncl	320	432.78	1240.96	70.5
21	52	10.00	110.00	Nitric Acid	200.576	569.99	962.8	75.16

These numeric factors had factorial, axial and center points as indicated in chapter three, but categoric factor had only four levels but nothing in between as center point. The location of the axial points from the factorial points was “two” making the design rotatable. The experiments were randomized to protect against an unknown bias distorting the result of the experiment. The design matrix used for the experiment with the experimental values for the four fibers are shown in Table 4.3.

4.4.1 Selection of a good predictive model for chemical treatments of the fibers

Like the ANOVA, the sequential model sum of square was used to compare different models (appendix D). It shows the statistical significance of adding new model terms step by step in increasing order. It provided accounts of variation and associated P-values (Prob>F) so that one can see how far it is worth going in degree of polynomial. The objective was to add a higher level source of term only if it explains a significant amount of variation beyond what was already accounted for.

The model was selected based on the highest order model that was significant (P-value small) and not aliased, lack of fit (P-value > 0.10) and reasonable agreement between Adjusted R-squared and predicted R-squared (within 0.2 of each other). For the four fibres, the predicted R-squared were in close range to the adjusted R-squared for all the sources of the model, but the models were suggested based on the model p-value and lack of fit p-values. The suggested model for all the fibers were quadratic models. Although the linear model had low p-value for the three fibres, it was discarded due to significant lack of fit. The linear model being insignificant in the sequential model sum of square means that the error term at that stage still contains variation that can be explained by higher order terms, in this case quadratic model.

4.4.2. Analysis of variance for the chemical treatment process

It is important to examine the suggested model if it provides an adequate approximation of the true response surface. The analysis of variance (ANOVA) was used for this purpose. ANOVA is statistical method based on the F-test that assessed the significance of experimental results. It involves sub-dividing the total variations of the set of data into component parts. It tests the linear terms, interaction terms and the quadratic terms included in the model. Any of the terms was removed from the model only when it had insignificant p-value or was retained in the model to support model hierarchy.

ANOVA was used to interpret the relative contribution of each factor to the total variations. R-squared, predicted R-squared and adjusted R-squared values were equally used to ascertain if the model selected will produce good prediction for average outcome.

Attention was focused on predicted R-square and adjusted R-square because the regular R-square can be artificially inflated by simply continuing to add terms to the model, even when the terms were not statistically significant. The adjusted R-squared plateaus when insignificant terms are added to the model, and the predicted R-square will decrease when there are too many insignificant terms. A role of thumb is that the adjusted and predicted R-square values should be within 0.2 of each other. There is no “cut off” value for R-square (Anderson and Whitcomb, 2005).

The models were deemed appropriate in this study based on the significancy of the model p-value, insignificant lack of fit test, good agreement between adjusted and predicted R-squared, adequate precision over four and well behaved residuals. Adequate precision measures the signal-to- noise ratio. It compares the range of the predicated values at the design points to the average prediction error. Ratio greater than four indicated adequate model discrimination (Anderson and WhitComb, 2005). The ANOVA for the four fibres was shown in Tables 4.4– 4.7

For *ampelocissus cavicaulis*, the model F-value of 12.20 implied that the selected model was significant. There was only 0.01% chance that a model F-value” this large could occur due to noise. Values prob< F less than 0.0500 indicated that the model terms were significant. Values greater than 0.1000 indicated the model terms were not significant. For *Ampelocissus cavicaulis* fiber; linear effect of chemical concentration (A), linear effect of time (B), linear effect of chemical type (C), quadratic effect of chemical concentration and interaction effects of chemical concentration and chemical types (AC) were significant.

The lack of fit F-value of 1.3 implied it was not significant and there was a 6.94% chance that a lack of fit F-value" this large could occur due to noise. Insignificant lack of fit was desirable because it is a measure of risk. The predicted R-squared of 0.7315 was in agreement with the Adj R-squared of 0.8575. 90.2% of the variability of the response data around its mean was explained by the model. Adequate precision of 16.133 indicated an adequate signal.

Table 4.4. ANOVA Table for *Ampelocissus cavicaulis* fiber

Source	Sum of Square	df	Mean Square	F Value	P-value prob>F
Model	102586.78	9	11398.53	12.20	<0.0001 suggested
A.Chemical Concentration	4247.45	1	4247.45	4.55	0.0436
B-Time	4862.12	1	4862.12	5.20	0.0314
C-Chemical type	55258.35	3	18419.45	19.72	<0.0001
AC	35183.73	3	11727.91	12.55	<0.0001
A^2	3035.13	1	3035.13	3.25	0.049
Residual	39233.46	42	934.13		
Lack of Fit	30716.25	26	1181.39	1.3	0.1694 not significant
Pure error	8517.21	16	532.33		

R^2 = 0.9020, Adj-R^2 = 0.8575, pred-R^2 = 0.7315 adeq – precision = 16.1333

For *Adenia lobata* fiber, the F – value of 18.77 implied that the selected model was significant and can explain the process well. There was only 0.01% chance that this large value of F-value can occur due to error. Values of prob>F less than 0.0500 indicated significant model terms. Values greater than 0.100 indicated insignificant model terms. Analysis of *Adenia lobata* fiber showed that the linear effects of chemical concentration, time, quadratic effect of chemical concentration and the interaction effect of the chemical concentration and chemical type were significant, with prob>F values less than 0.05.

The lack of fit test came out insignificant with F-value of 1.3. There was 16.27% chance that this large value of lack of fit will occur due to error. The predicted R-squared of 0.8247 was in close range with the adjusted R-squared of 0.9233. R-square of 0.97793 was high which showed that 97.8% of the total variation of the outcome will be explained by the model. Adequate precision of 18.650 was adequate and indicated a good signal

Table 4.5. ANOVA Table for *Adenia lobata* fiber

Source	Sum of Square	df	Mean Square	F Value	P-Value Prob>F
Model	2.658E+005	9	29536.957	18.77	<0.0001 significant
A-Chemical Concentration	15731.07	1	15731.07	9.99	0.0040
B-Time	8123.44	1	8123.44	5.16	0.0345
C-Chemical Type	2.024E+005	3	67450.19	42.85	<0.0001
AC	32462.97	3	10820.99	6.88	0.0012
A^2	7132.61	1	7132.61	4.53	0.0261
Residual	66105.97	42	1573.95		
Lack of fit	53481.73	26	2056.99	1.30	0.1627 not significant
Pure error	12624.24	16	789.02		

R^2 = 0.97793, Adj. R^2 = 0.9233, Pred.R^2 = 0.8247, Adeq precision = 18.650

For *Morinda morindoidies*, the F- value of 17.3 implied that the selected model was significant. There was only 0.01% chance that this large F-value can occur due to noise. Values of prob>F less than 0.0500 indicated significant model terms, values greater than 0.100 indicated insignificant model terms. ANOVA of *Morinda morindoides* showed that the linear effect of chemical concentration, time, chemical types, quadratic effect of chemical concentration and the interaction effects of chemical concentration and chemical types were significant with their prob>F value less than 0.05. Lack of fit F-value of 1.09 implied insignificant lack of fit. There was 55.48% chance that a lack of fit F-value this large could occur due to noise.

The predicated R-squared of 0.6911 was in good agreement with the adjusted R-square of 0.8494. Adequate precision of 14.938 was above four, indicating an adequate signal.

Table 4.6. ANOVA Table for *Morinda morindoides* fiber

Source	Sum of Square	Df	Mean Square	F Value	P-Value Prob>F
Model	4.610E+005	9	5.122E+005	17.3	<0.0001 significant
A-Chemical Concentration	1.288E+005	1	1.288E+005	4.35	0.0040
B-Time	2.066E+005	1	2.066 E+005	6.98	0.0045
C-Chemical Type	2.327E+005	3	7.757 E+005	26.19	<0.0001
AC	2.94E+005	3	2.64 E+005	8.91	0.0012
A^2	1.154E+005	1	1.154E+005	3.90	0.0413
Residual	1.244E+006	42	29619.048		
Lack of fit	8.387E+005	26	32257.69	1.09	0.5548 not significant
Pure error	4.053E+005	16	25331.25		

R^2 = 0.9178, Adjusted R^2 = 0.8494, Predicated R^2 = 0.6911, Adeq precision = 14.938

For *Ampelocissus leonensis*, the F-value of 23.15 indicated significant model. There was only 0.01% chance that this large F- value can occur due to noise. Values of prob>F less than 0.0500 indicated significant model terms. Values greater than 0.100

indicated insignificant model terms. Analysis on *Ampelocissus leonensis* revealed that linear terms of chemical concentration (A), time (B), chemical type (C), interaction effect of chemical concentration and chemical type (AC) and the quadratic effect of chemical concentration were significant with prob>F values less than 0.05. The insignificant lack of fit was desirable with 68.327% chance of getting the high F-value due to noise. There was a very good agreement between predicated value of 0.8518 and adjusted value of 0.9461. Adequate precision was high enough (24.609) indicating an adequate signal.

The ANOVA of the four fibers confirmed that the models selected were adequate to predict the response well.

Table 4.7. ANOVA Table for *Ampelocissus leonensis* fiber

Source	Sum of Square	df	Mean Square	F Value	P-Value Prob>F
Model	19205.22	9	2133.91	23.15	<0.0001 significant
A-Chemical Concentration	5743.61	1	5743.61	62.32	<0.0001
B-Time	1301.82	1	1301.82	14.13	0.0005
C-Chemical Type	8057.05	3	3019.03	32.76	<0.0001
AC	2077.72	3	692.57	7.51	0.0004
A^2	1025.02	1	1025.02	11.12	0.0033
Residual	3870.86	42	92.16		
Lack of fit	2210.73	26	85.03	0.82	0.6832 not significant
Pure error	1660.13	16	103.76		

R^2 squared = 0.97793, Adjusted R-squared = 0.9461, Predicated R-squared=0.8518, Adequate precision = 24.609.

4.4.3. Predictive Models in Coded Versus Actual Units

Predictive models equations generated are mathematical representation of the chemical treatment process using the selected model after removing the insignificant factors. The model equations were presented in both coded and actual values. The coded values works only if the factors are converted to the standard coding scale – 1 to + 1 for the low versus high values, respectively of the factorial ranges. Due to dependency of the actual values on units, their coefficients did not tell anything, coding factors removes their units of measure. The intercept in coded value represents the center of the design of experiments, and the regression coefficients tell us how the response changes relative to this point of reference. Thus, the coded model facilitates knowledge of the process.

Regardless of the form of the model, it is only an approximation, not the real truth. It is good enough to help you move in the proper direction, but not to make exact prediction particularly outside the actual experimental region.

Typically, a categoric factor's level are represented by indicator "dummy" variables in regression. The value of the dummy variables are "0" if that types is not present in that treatment/run, and "I" if it is present, therefore, the four chemical types were represented by 100, 010, 001, and -1-1-1 respectively. The "100" meant that the first chemical type was present while the other ones were absent. "010" meant the second type of chemical was present while others were absent. "001" meant the third chemical was present, while other ones were absent and "-1-1-1" meant that none of chemicals were present. These codes were assigned randomly to different chemicals as illustrated on Table 4.35. The coded equation involving categoric factor can be seen as being four equation one comprising C[I] with its interactions, two comprising C[2] with its interaction, three comprising C[3] with all its interactions, and C[4] was seen as the reference level of the categorized factors. The Equation for C [4] was one with all the C terms and interaction terms were eliminated. Each chemical type adjusts the intercept by the amount of its coefficient, while its effect on interaction with other factors affects the slope due to the factor.

Equation involving C[1] was used with the chemical type "100" for response prediction, C[2] equation used for chemical type "010" for respond predication, C[3] equation was used for chemical type “001” while C[4] being the reference level was used for chemical type “1-1-1- “ for response prediction.

- Model equation for *Ampelocissus cavicaulis* (Nwogbe) fiber;

Final Equation in terms of coded factors:

Tensile strength (Mpa) = 2.99.02 – 9.41A^2 – 10.06B+ 33.54 C[1] – 48.13 C[2] +26.03 C[3] + 35.99 AC[1] – 28.39AC[2] +16.23AC[3] (4.2)

Final equation in terms of actual factors:

NaOH :

Tensile strength (Mpa) = +301.34542+6.644.83 $(CC)^2$– 0.50323 Time (4.3)

Nitric acid:

Tensile strength (Mpa) = + 380.61744 -9.45000 $(CC)^2$ - 0.50323 Time (4.4)

Acetic anhydride:

Tensile strength (Mpa)= + 343.23.23688+1.70533$(CC)^2$–0.50323 Time (4.5)

Zinc Chloride:

Tensile strength (Mpa) = + 405.87729 – 8.30700 $(CC)^2$ – 0.50323 Time (4.6)

Where “CC” is chemical concentration.

Using model equation involving categoric factors in predicating responses is always a complex issue, the final equation in terms of coded value in equation 4.2 can be seen as being four equations, one for each type of chemical as thus;

Tensile strength (Mpa) - +299.02 – 9.41A^2 – 10.06B +33.54C[1] + 35.99AC[1] (4.7)

Tensile strength (Mpa) = +299.02 – 9.41A^2 – 10.06B +48.13 C[2] - 28.39 AC[2] (4.8)

Tensile strength (Mpa) = +299.02 – 9.41A^2 – 10.06B +26.03 C[3] + 16.23AC[3] (4.9)

Tensile strength (Mpa) = +299.02 – 9.41A^2 – 10.06B (4.10)

In equation 4.7, the presence of the chemical type “100” adjusted the intercept positively by 33.54 and also, its effect affected the slope due to chemical concentration (A). It increased the sensitivity of tensile strength by 35.99 due to chemical concentration.

The presence of chemical type “010” adjusted the intercept positively by 48.13 and increased the sensitivity of tensile strength due to chemical concentration by 28.39 as indicated in equation (4.8).

The presence of chemical type “001” adjusted the intercept positively by 26.03 and increased the sensitivity of tensile strength due to chemical concentration by 16.23. Equation 4.10 is the reference level and was used when all the chemical types and their interaction with other factors were eliminated, in other words “-1-1-1 was used in the design matrix.

- Model equations for *Adenia lobata* (Usoro) fiber

Final equation in terms of coded factors:

Tensile strength (Mpa) = $+585.76 - 18.10A^2 - 13.01B + 67.10\,C[1] - 4.1\,C[2] + 35.82C[3] - 1.23AC[1] + 8.45AC[2] + 32.49AC[3]$ (4.11)

Final equation in terms of actual factors:

NaOH:

Tensile strength (Mpa) = $+746.72705 - 4.83396\,(CC)^2 - 0.65048$ Time (4.12)

Nitric acid:

Tensile strength (MPa) = $+651.30731 - 2.41229\,(CC)^2 - 0.650$ Time (4.13)

Acetic Anhydride:

Tensile strength (Mpa) = $+631.5551 + 3.59604\,(CC)^2 - 0.65046$ Time (4.14)

Zinc chloride:

Tensile strength (Mpa) = $+ 677.01333 - 14.45313\,(CC)^2 - 0.65046$ time (4.15)

where “CC” represents chemical composition.

The four components of the coded equation;

Tensile strength (Mpa) = $+ 585.76 - 18.10A^2 - 13.01B + 67.10\,C\,[1] - 1.23AC\,[1]$ (4.16)

Tensile strength (Mpa) = $+ 585.76 - 18.10A^2 - 13.01B + 4.11\,C\,[2] + 8.45\,AC\,[2]$ (4.17)

Tensile strength (Mpa) = $+ 585.76 - 18.10A^2 - 13.01B + 35.82\,C\,[3] + 32.49A\,C[3]$ (4.18)

Tensile strength (Mpa) = $+ 585.76 - 18.10A^2 - 13.01B$ (4.19)

For *Adenia lobata* fiber, the effect of chemical type affected the tensile strength of the fiber and the sensitivity of the fiber due to chemical concentration.

Effect of sodium hydroxide increased the overall average tensile strength by 67.10 and decreased the sensitivity due to chemical concentration by 1.23. Effect due to nitric acid, decreased the intercept by 4.11 and increased the sensitivity due to chemical concentration by 8.45. Effect due to acetic anhydride increased the overall mean by 35.82 and increased the slope due to chemical concentration by 32.49. Zinc chloride was used as a reference chemical and was assumed as having zero effect when appeared in the treatment for response prediction only.

- Model equation for *Morinda morindoides* (Ogbuebo) fiber

Final equation in terms of coded factors:

Tensile strength (Mpa) = $+1189.96 - 51.79A^2 - 65.60B + 359.54\,C[1] - 12555\,C[2] - 59.52\,C[3] + 212.83AC[1] - 25.76\,AC[2] - 129.96\,AC\,[3]$ (4.20)

Final equation in terms of actual factor

NaOH:

Tensile strength (Mpa) $=+1376.51304+40.26000\,(CC)^2- 3.28021$ Time (4.21)

Nitric acid:

Tensile strength (Mpa) $=+1487.89574-19.38750\,(CC)^2- 3.28021$ Time (4.22)

Acetic Anhydride:

Tensile strength (Mpa) $=+1814.44099-45,\,43833\,(CC)^2- 3.28021$ Time (4.23)

Zinc chloride:

Tensile strength (Mpa) $=+1577.37048-27.2267\,(CC)^2-3.28021$ Time (4.24)
where CC represents chemical concentration.

The equation (4.20) can be split into;

Tensile strength (Mpa) $=+1189.96-51.79A^2-65.60B+359.54\,C[1]+212.83\,AC[1]$ (4.25)

Tensile strength (Mpa) $=+1189.96-51.79A^2-6560B-1215.55C\,[2]-25.76AC\,[2]$ (4.26)

Tensile strength (Mpa) $=+1189.96-51.79A^2-65.60B-59.52\,C[3]-129.96\,AC[3]$ (4.27)

Tensile strength (Mpa) $=+1189.96-51.79A^2-65.60B$ (4.28)

Effect of "100" in the treatment increased the overall average tensile strength by 359.54 and equally increased the sensitivity due to chemical concentration by 212.831. The effect due to "010" in the run decreased the overall average of tensile strength by 125.55 and equally decreased the sensitivity due to chemical concentration by 25.76. The effect due to "001" in the treatment decreased the intercept by 59.52 and decreased the slope due to chemical concentration by 129.96.

"-1-1-1" in the run was regarded as reference level in which all the effect due to chemical type and its interaction were eliminated.

The intercept of the equation varies depending on the chemical type used and their effect on the sensitivity by other factors.

- Model equation for *Ampelocissus leonensis* (Okpaowoko) fiber

Final equation in terms of coded factors:

$$\text{Tensile strength (Mpa)} = +80.33 - 10.94A - 5.21B + 13.99\,C[1] - 1.60C[2] + 8.33C[3] - 9.80AC[1] + 7.82\,AC[2] + 3.64\,AC[3] + 3.21A^2 \quad (4.29)$$

Final equation in terms of Actual factors:

NaOH:

$$\text{Tensile strength (Mpa)} = +184.43422 - 9.1933\,CC - 0.26039\,Time + 0.20041\,(CC)^2 \quad (4.30)$$

Nitric Acid:

$$\text{Tensile strength (Mpa)} = +124.79888 - 4.78869\,CC - 0.26039\,Time + 0.20041\,(CC)^2 \quad (4.31)$$

Acetic anhydride:

$$\text{Tensile strength (Mpa)} = +145.17719 - 5.83348\,CC - 0.26039\,Time + 0.20041\,(CC)^2 \quad (4.32)$$

Zinc chloride:

$$\text{Tensile strength (Mpa)} = +129.35795 - 7.15598\,CC - 0.26039\,Time + 0.20041\,(CC)^2 \quad (4.33)$$

Where "CC" represents chemical concentration.

The coded equation can be seen as comprised of these four equations;

$$\text{Tensile strength (Mpa)} = +80.33 - 10.94A - 5.21B + 13.99\,C[1] - 9.80AC[1] + 3.21A^2 \quad (4.34)$$

$$\text{Tensile strength (Mpa)} = +80.33 - 10.94A - 5.21B - 1.60C\,[2] - 2.82AC\,[2] + 3.21A^2 \quad (4.35)$$

Tensile strength (Mpa) + 80.33 – 10.94A – 5.21B +8.33C [3] + 3.64AC [3] + $3.21A^2$ (4.36)

Tensile strength (Mpa) =+80.33–10.94A–5.21B + $3.21A^2$ (4.37)

4.4.4. Optimal conditions for the chemical treatment process

Numerical optimization was used to search the design space using the model created during analysis to find factor settings that met the defined goal. Maximization of tensile strength was set as goal to be met for the optimization. The software automatically generated a list of potential factor settings that met the specific criteria based on the desirability. The factor setting used for the optimization was selected based on the highest desirability.

- The Optimum conditions based on the categoric factor involved for *Ampelocissus cavicaulis* fiber is as follows:

(a) 6% Na0H for 50minutes with predicted ultimate tensile strength of 369.21 2Mpa

(b) 14% acetic anhydride for 70minute with predicted ultimate tensile strength of 341.944 Mpa.

(c) 6% Nitric acid for 50 minutes with predicted ultimate strength of 298.25% Mpa.

(d) 3% zinc chloride for 70 minutes with predicted ultimate strength of 330.84 Mpa.

- Optimum conditions based on the categoric factor for *Adenia lobata* fiber

(a) 6% NaOH for 50minutes with predicated tensile strength of 685.2 Mpa

(b) 14% acetic anhydride for 70minutes with predicated tensile strength of 684.75 Mpa

(c) 6% nitric acid for 70munites with predicated tensile strength of 604.311 Mpa

(d) 6% zinc chloride for 20minutes with predicated tensile strength of 600.772 Mpa.

- Optimum conditions based on the categoric factor for *Morinda morindoidies* fiber

(a) 14% NaOH for 50minute with predicated tensile strength of 1631.22 Mpa

(b) 6% Acetic anhydride for 70minutes with predicated tensile of 1542.2 Mpa

(c) 10% Nitric acid for 60minutes with predicated tensile strength of 1411.30 Mpa

(d) 6% zinc chloride for 30minutes with predicated tensile strength of 1289.91 Mpa.

- Optimum Conditions based on the Categoric factor for *Ampelocissus leonensis* fibre

(a) 6% NaOH for 50minute with predicated tensile strength of 123.469 Mpa.

(b) 6% Acetic anhydride for 62minutes with predicated tensile of 104.371 Mpa.

(c) 14% Nitric acid for 50minutes with predicated tensile strength of 90.2619 Mpa.

(d) 3% zinc chloride for 30minutes with predicated tensile strength of 85.6173 Mpa.

4.4.5. Validation of optimum conditions for the chemical treatment process

The optimum conditions obtained based on the predicted models were validated to confirm the predicted response and obtain the percentage deviation (error) from the predicted ultimate conditions. Tables 4.8 to 4.11, show the model desirability, the optimum conditions, and the predicted and experimented ultimate strength with their percentage errors for the four fibers.

Table 4.8. Validation of optimum conditions for *Ampelocissus cavicaulis* fiber

Model desirability	Chemical Strength (%)	Chemical Type	Time (minutes)	Tensile Strength (Mpa)		Error (%)
				Predicted value	Experimental values	
1.00	6.0	NaOH	50.0	369.212	365.62	0.97
0.928	14.0	Acetic anhydride	70.0	341.944	338.52	1.0
0.829	6.0	Nitric acid	50.0	298.756	295.111	1.22
0.723	3.0	Zinc chloride	70.0	330.874	326.407	1.35

Table 4.9. Validation of optimum conditions for *Adenia lobata* fiber

Model desirability	Chemical Strength (%)	Chemical Type	Time (minutes)	Tensile Strength (Mpa)		Error (%)
				Predicted value	Experimental values	
1.00	6.0	NaOH	50.0	685.2	676.292	1.3
0.958	14.0	Acetic anhydride	70.0	684.565	674.16	1.52
0.911	6.0	Nitric acid	70.0	604.311	595.851	1.4
0.852	6.0	Zinc chloride	70.0	600.772	600.100	1.11

Table 4.10. Validation of optimum conditions for *Morinda morindodies* fiber

Model desirability	Chemical Strength (%)	Chemical Type	Time (minutes)	Tensile Strength (Mpa)		Error (%)
				Predicted value	Experimental values	
0.982	14.0	NaOH	50.0	1631.22	1623.074	0.5
0.912	6.0	Acetic anhydride	70.0	1542.77	1522.714	1.3
0.820	10.0	Nitric acid	60.0	1411.30	1408.48	0.2
0.781	6.0	Zinc chloride	30.0	1289.91	1277.011	1.0

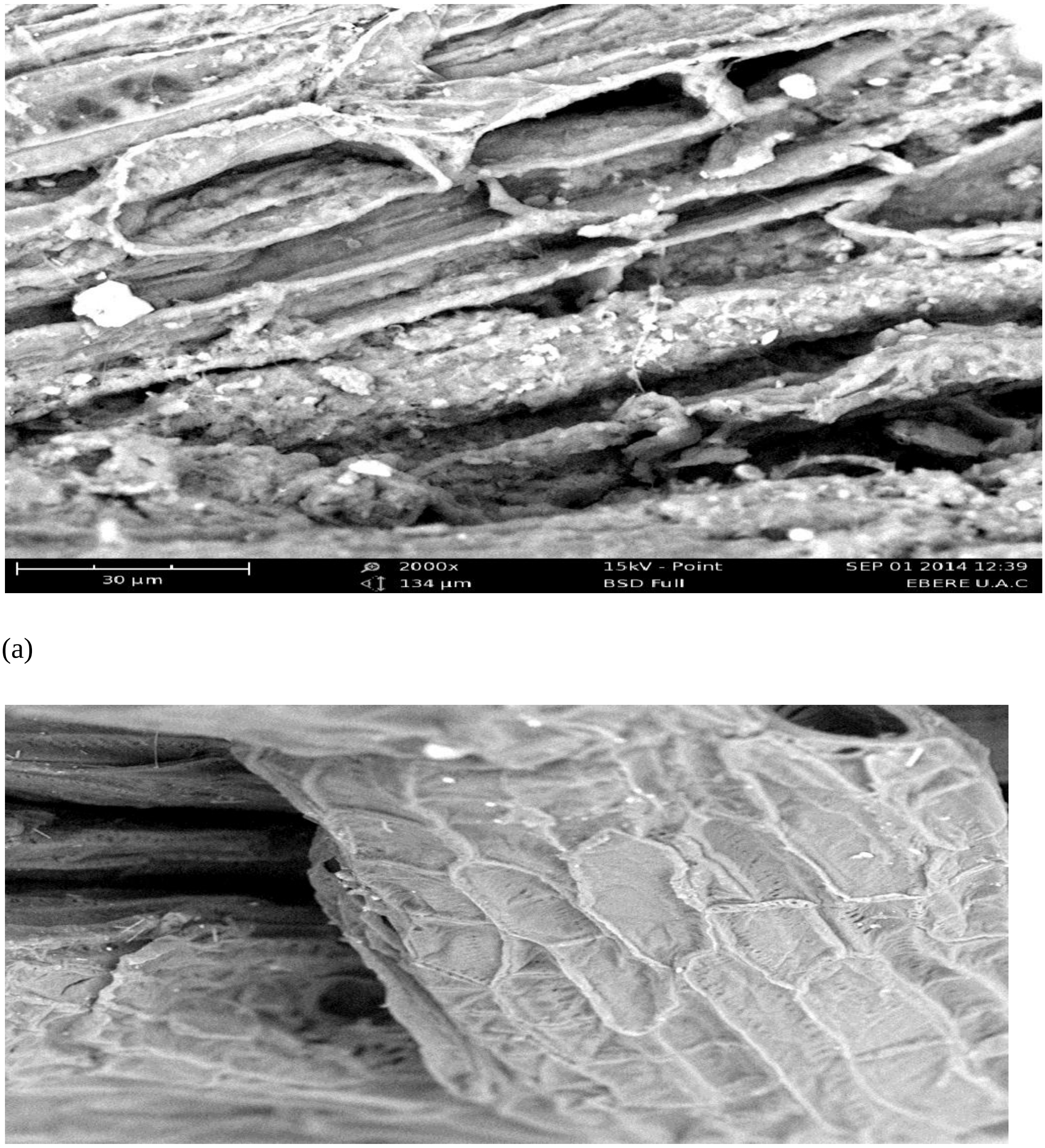
Table 4.11. Validation of optimum conditions for *Ampelocissus leonensis* fiber

Model desirability	Chemical Strength (%)	Chemical Type	Time (minutes)	Tensile Strength (Mpa)		Error (%)
				Predicted value	Experimental values	
1.0	6.0	NaOH	50.0	123.469	123.346	0.1
1.0	6.0	Acetic anhydride	62.0	104.371	103.745	0.6
0.925	11.4	Nitric acid	50.0	90.2619	89.540	0.8
0.781	3.0	Zinc chloride	30.0	85.6173	84.590	1.0

The results shown on Tables 4.8 to 4.11 confirmed the optimum conditions obtained with different chemicals with little errors of less than 2.0%.

4.4.6. Scanning Electron Microscopy (SEM) Analysis of Untreated and Treated Fibers

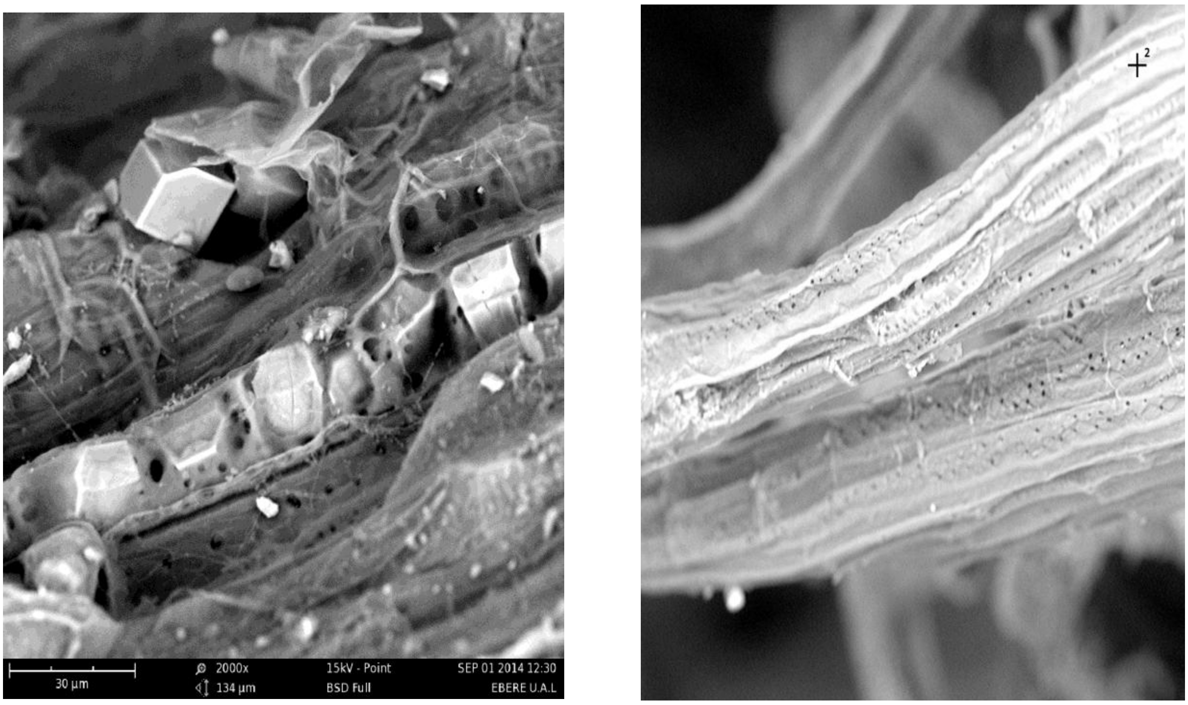
The effects of chemical treatments on the surface of the fibers are shown as SEM photomicrographs in Figures 4.14 to 4.17 for the untreated fibers and fibers treated with sodium hydroxide. The SEM micrograph of the surfaces of untreated fibers shows the presence of wax, oil, and surface impurities. Waxes and oils provide protective layers to the surface of the fibers (Kumar et al., 2011). The chemical treatments on the fiber were successful in removing the surface contaminants as evidenced in the Figures 4.14 to 4.17. The micrographs show very clean surfaces which confirmed the removal of the wax, oil and other surface contaminants from the fiber surface.



(a)

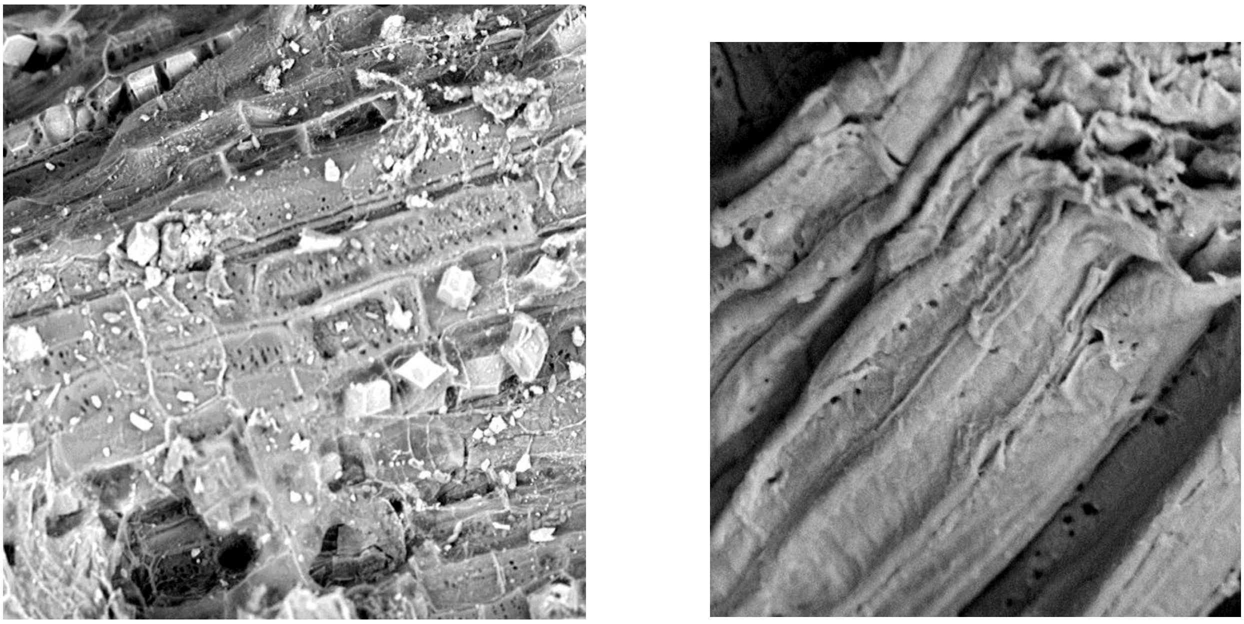
(b)

Figure 4.14. SEM micrographs of *Ampelossisus cavicaulis* fiber (a)untreated (b) sodium hydroxide treated



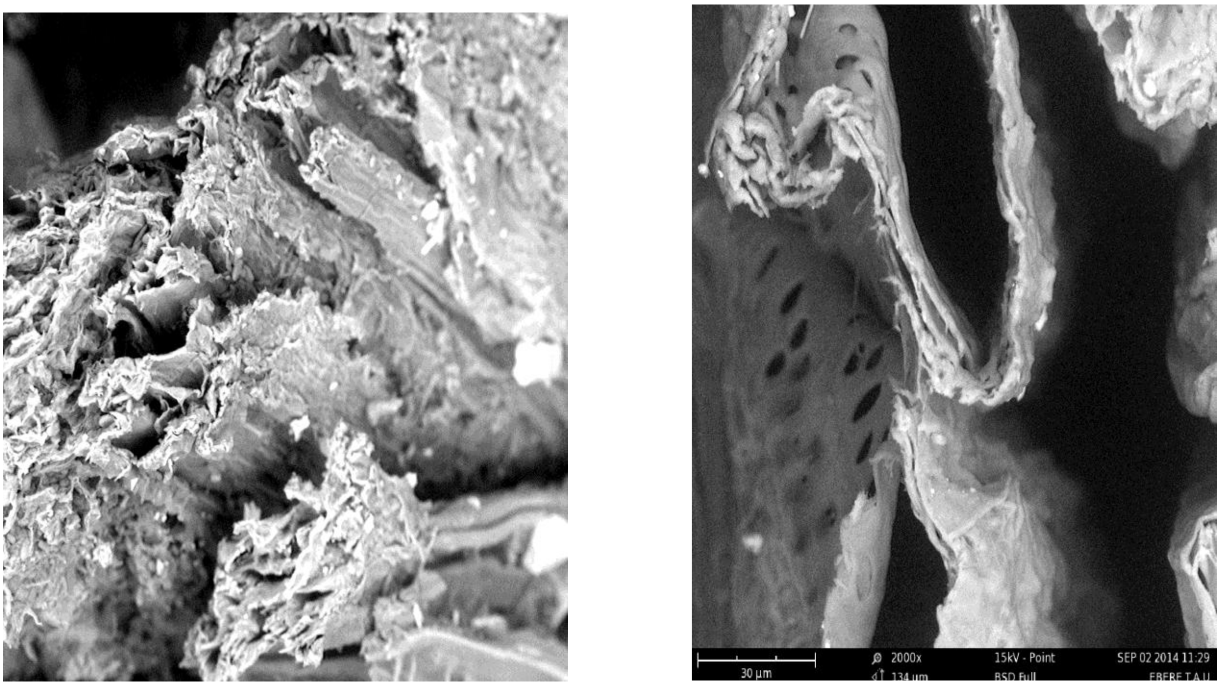
(a) (b)

Figure 4.15. SEM micrographs of *Adenia lobata* fiber (a) untreated (b) Sodium hydroxide treated



(a) (b)

Figure 4.16. SEM micrographs of *Morinda morindoides* fiber (a) untreated (b) sodium hydroxide treated.



(a)

(b)

Figure 4.17. SEM micrographs of *Ampelossisus leonensis* fiber (a) untreated (b) sodium hydroxide treated.

4.5. Effect of chemical treatments on the physical properties of the fibers

Effect of the fiber modifications using different chemicals were studied on some physical properties of the fibers. These physical properties were water absorption and aspect ratio.

4.5.1. Water absorption of the fibers

From Fig. 4.18 it was shown that, the study on water absorption of the untreated fiber showed that the untreated fiber absorbs more water than the treated fibers. The water absorption by the untreated fiber is due to the fact that the untreated fiber contains very high percentage of hemicellulose in its structure. Also, in natural cellulose fibers, a hollow cavity called lumen exists in the unit cell of the fibers which generates more path ways for water to diffuse into the fiber (Reddy and Yang, 2005). The effect of chemical treatment on the water absorption showed that the chemical treatment process decreased the dissemination of water into the fiber and the fibers became more hydrophobic. This reduction is likely due to the reduction of the hemicellulose components from the raw fibers, as stated in the literature, chemical treatments result in the partial dissolution of hemicellulose (Ring et al., 2001). Hemicellulose, however is responsible for the water absorption of the fiber (Nabi and Jog, 1999). It was observed that the mercerized fibers absorbed the least water compared to the other chemical treated fibers. This can be attributed to the removal of the non-cellulose constituents' pectin, lignin, hemicellulose and impurities from the fibers surface by mercerization (Mbod et al., 2003). This removed the polar group and prevented the formation of hydrogen bonds with water molecules (Ishidi et al., 2011). This inevitably reduced the water uptake by the treated fiber. The results equally pointed out that acetylation process decreased the dissemination of water into the fiber and fiber became more hydrophobic. This is due to the replacement of hydroxyl groups by hydrophobic acetyl groups. Chemical modification takes place as the acetic anhydride substitute the cell wall hydroxyl groups with acetyl groups making the surface more hydrophobic (Bessadok et al., 2007; Paul et al., 1977). The same trend was observed on the nitric acid and zinc chloride treated fibers. The result of the treatment was the reduction of the hemicellulose component of the fiber which resulted to the hydrophobic nature of the treated fibers.

From the Fig. 4.18, it was equally observed that the water absorption capacity of *Ampelocisssus leonensis* fiber was greater than other fiber. This observation can be attributed to the high hemicellulose content of the fiber.

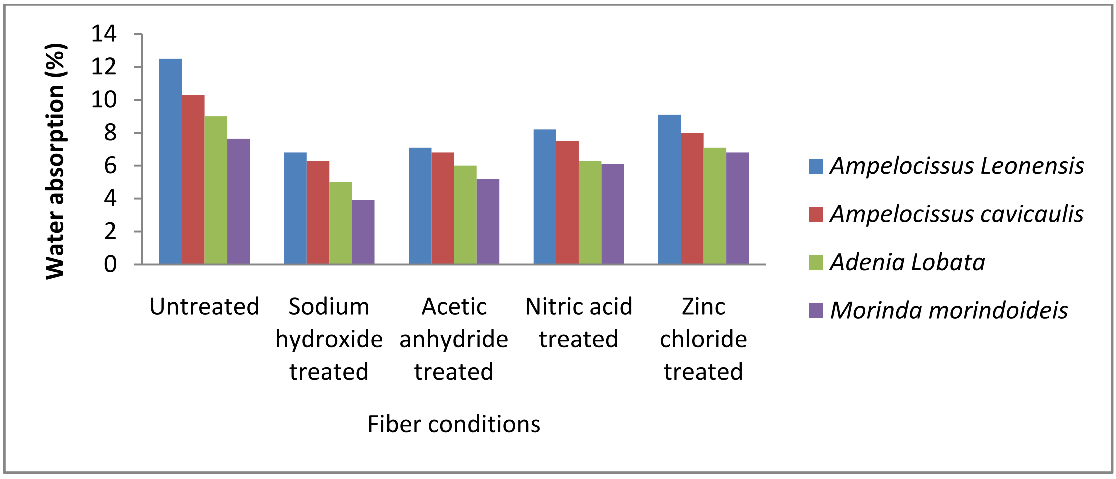


Fig. 4.18. Water absorption of untreated and treated fibers.

4.5.2. Aspect ratio of the fibers

Aspect ratio is one of the factors that determine the quality of fibers as reinforcement in composite materials. The fiber lengths, diameter and aspect ratio of the untreated and chemically treated fibers are shown in Table 4.12 and aspect ratio depicted in Figure 4.19. From the results, it can be seen that the aspect ratio of the treated fibers are higher than the untreated fibers. This is as a result of the effect of the chemical treatment on the fibers which resulted to reduction of the diameters of the fibers. It is the usual method to produce high quality fibers by removing natural and artificial impurities. Chemical treatment increases surface roughness and lead to fibrillation of the fiber bundle, thus, reducing the fiber diameter and thereby increasing aspect ratio. The development of a rough surface and the enhanced aspect ratio resulted in a better mechanical interlocking and induced an improved fiber /matrix interfacial adhesion in composite. This can be attributed to the increase in fiber diameter as a result of the coupling of acetyl group with the hydroxyl group of lignin, hemicellulose and amorphous cellulose leading to expansion of fiber cell wall which swells the fiber (Zafeiropoulos et al., 2002).

It was equally observed from the results that there were no changes on the length of the fibers as a result of the chemical treatments. This is likely because of the optimum treatment conditions used for the chemical treatments which in-turn avoided the breaking of the fibers as result of excess fibrillation. According to Molid et al. (2013), for optimum effectiveness, the aspect ratio of fiber must be in the range of 100 – 200. From this present study, the aspect ratio of the fiber were within the range, thus, they can serve as a good reinforcement for composites.

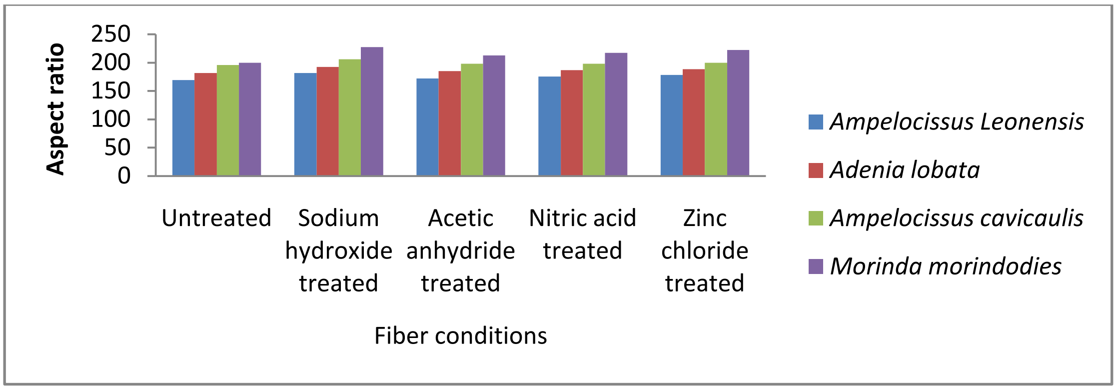


Fig. 4.19. Effect of chemical treatments on fibers' aspect ratio.

Table 4.12. Effect of chemical treatments on fiber length, diameter and aspect ratio

S/N	Samples	Average Length (mm)	Average diameter (mm)	Aspect ratio (L/D)
1	*Ampelocissus leonensis*			
	Untreated	100.00	0.58 – 0.60	169.6
	NaOH Treated	100.00	0.54 – 0.56	181.8
	Acetic Anhydride Treated	100.00	0.57 – 0.59	172.4
	Nitric Acid Treated	100.00	0.56 – 0.58	175.4
	Zinc Chloride Treated	100.00	0.55 – 0.57	178.6
2	*Adenia Lobata*			
	Untreated	100.00	0.54 – 0.56	181.8
	NaOH Treated	100.00	0.51 – 0.53	192.3
	Acetic Anhydride Treated	100.00	0.53 – 0.55	185.1
	Nitric Acid Treated	100.00	0.53 – 0.54	186.9
	Zinc Chloride Treated	100.00	0.52 – 0.54	188.7

3	*Ampelocissus Cavicaulis*			
	Untreated	100.00	0.50 – 0.52	196.1
	NaOH Treated	100.00	0.48 – 0.49	206.1
	Acetic Anhydride Treated	100.00	0.50 – 0.51	198.0
	Nitric Acid Treated	100.00	0.50 – 0.51	198.0
	Zinc Chloride Treated	100.00	0.49 0.51	200.0
4	*Morinda Morindoidies*			
	Untreated	100.00	0.49 – 0.51	200
	NaOH Treated	100.00	0.43 – 0.45	227.3
	Acetic Anhydride Treated	100.00	0.46 – 0.48	212.8
	Nitric Acid Treated	100.00	0.44 – 0.48	217.4
	Zinc Chloride Treated	100.00	0.43 – 0.47	222.2

4.6. Effect of Chemical treatments of Fibers on the Mechanical Properties of the Fibers

The fibers were treated using the optimum conditions established and the effects of the treatments on the mechanical properties of the fibers were determined. Table 4.13 shows the results of the tensile strength, tensile modulus, and elongation at break.

Table 4.13. Effect of chemical treatments on the mechanical properties of the fibers

	AMPELOCISSUS CAVICAULIS			*ADENIA LOBATA*			*MORINDA MORINDODIES*			*AMPELOCISSUS LEONENSIS*		
	Tensile Streng. (Mpa))	Tensile Modu. (Mpa)	Elong. At Break (%)	Tensile Streng. (Mpa))	Tensile Modulus (Mpa)	Elongat. At Break (%)	Tensile Strength (Mpa)	Tensile Modulus (Mpa)	Elonga At Break (%)	Tensile Streng. (Mpa))	Tensi. Modulus (Mpa)	Elongat. At Break (%)
Untreat	238.28	3971.3	3.0	588.94	2030.82	6.75	1202.79	11946.2	5.03	67.67	422.8	8.0
NaOH Treated	365.02	6083.6	4.2	676.2	2331.7	7.71	1623.07	16119.8	5.2	123.34	770.6	9.0
Acetic Anhydride Treated	338.52	5642.2	4.0	674.16	2424.0	7.5	1522.7	15122.9	5.5	103.75	648.2	8.5
Nitric Acid Treated	295.11	4918.7	3.6	596.85	2054.5	6.2	1408.48	13988	4.8	89.54	559.4	7.3
Zinc Chloride Treated	326.40	5440.2	3.8	601.00	2072.2	7.0	1277.07	1275.2	4.2	84.59	528.5	7.0

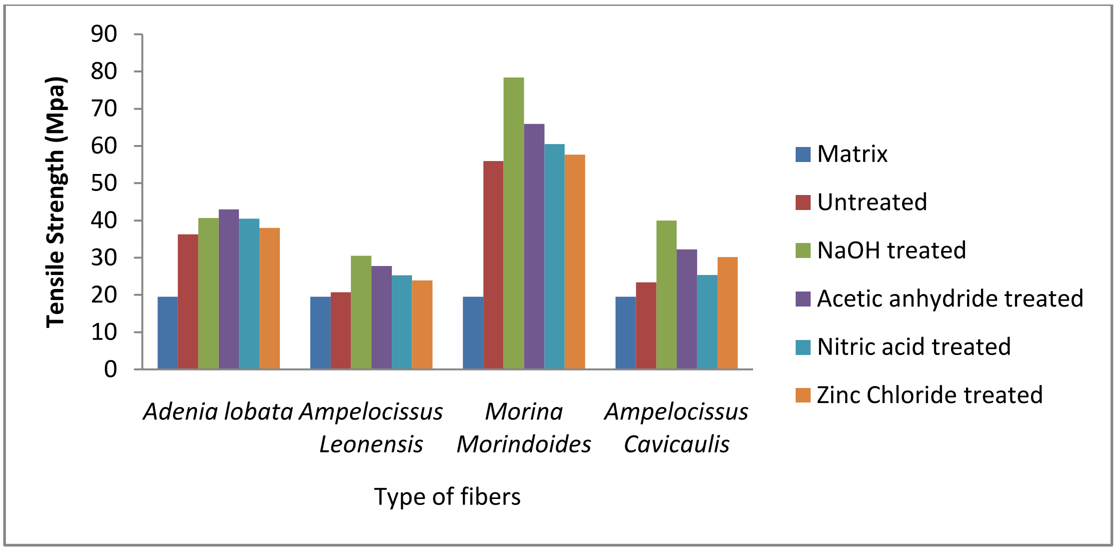
4.7. Effect of chemical treatments of fibers on mechanical properties of the composites

The investigation of mechanical properties of composites is one of the most important techniques in studying the behaviour of composites materials. Mechanical properties of fiber-reinforced composites depend on the nature of matrix material and the distribution and orientation of the reinforcing fibers, the nature of the fiber-matrix interfaces and of the interphase region (Srinivas and Bharath, 2011). Even a small change in the physical nature of the fiber for a given matrix may result in prominent changes in the overall mechanical properties of the composites. It is well known fact that different degrees of reinforcement effects are achieved by the addition of hydrophilic fibers to different polymers. This may be due to the different adhesion strength between matrix and fibers. Mechanical properties were investigated on the matrix, untreated fiber reinforced composites, NaOH treated fiber reinforced composites, acetic anhydride treated fiber reinforced composites, nitric acid treated fiber reinforced composites and zinc chloride treated fiber reinforced composites.

4.7.1. Tensile Properties

From Fig.4.20, it was observed that the tensile strengths and modulus of the fiber reinforced composites were higher than the tensile strength of the matrix. This was possible because when the composites were stressed, the load was transferred between the fiber and the polymer since both were bonded together. Both fiber and the matrix will stretch by the same amount; the fiber being stiffer carries the larger stress. As can equally be seen from the results (Fig.4.20), that the tensile strength of the composite with treated reinforcement was higher than the untreated one. The increase was more significant in the case of alkali treatment. Some of the shortcoming and limitations of natural fibers when used as reinforcement for composites are related to the lower strength properties, and lower interfacial adhesion. To overcome the shortcomings, various chemical like NaOH, acetic anhydride, nitric acid and zinc chloride were used to treat the fibers before using as reinforcement. During treatments, waxes, hemicelluloses and part of the lignin present on the fiber surface which acted as cement covering the cellulose were removed. The removal of these compounds enhanced the surface roughness, which allows mechanical interlocking. The strong interface region can transfer the maximum load from the matrix to fiber surface. The formation of strong covalent bonds between the polymer and the hydroxyl groups of cellulose lead to significant improvement on the tensile strength. Improvements in tensile strength of composites help the composite to withstand more tensile forces when they are in service. Also, the materials with more strain are likely to fail safe in service.

The tensile strength and modulus of *Morinda morindoides* fiber reinforced composites were far higher than others. This observation can be attributed to the inherent nature of the fiber. *Morinda morindoides* fiber has high tensile strength than other fiber, thereby withstanding more stress when used as reinforcement on a matrix.



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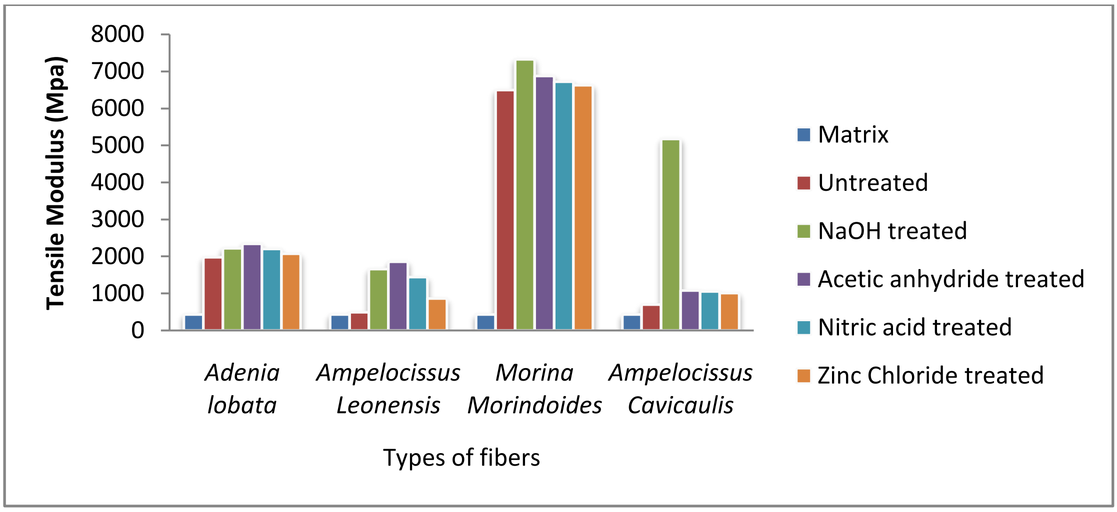


(b)

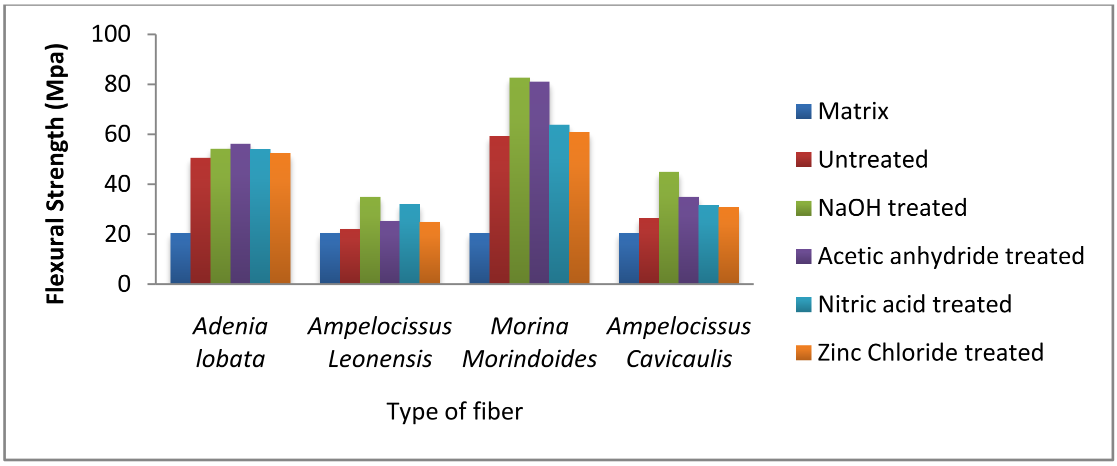
Fig.4.20. Tensile test of the composites (a) Strength (b) Modulus.

4.7.2. Flexural Properties

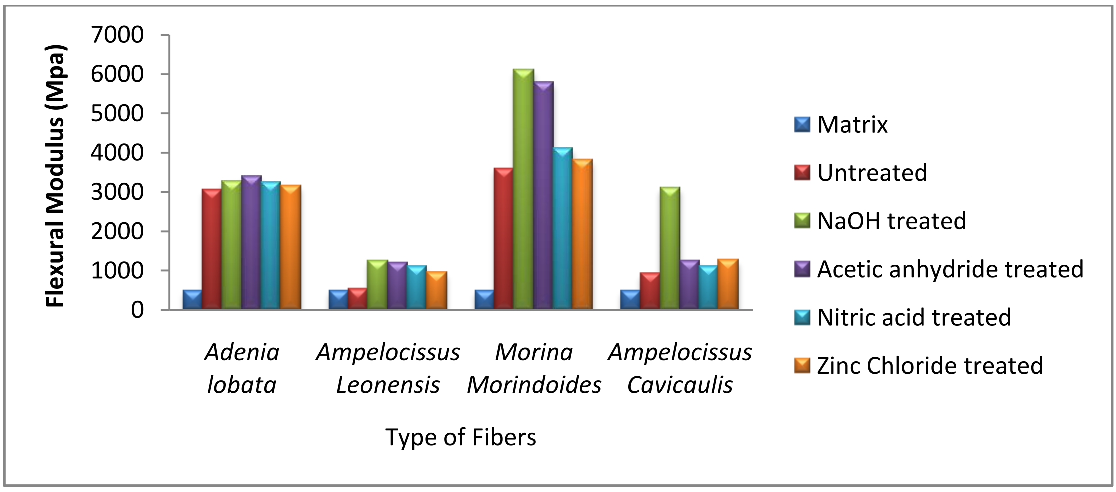
It was observed from Fig.4.21 that the flexural strength and modulus of the fiber reinforced composites were greater than the flexural strength and modulus of the polymer.

Impregnations of natural fiber help in the interface bonding which improved the structural properties of the composites. Chemically treated fiber reinforced composite presented high flexural strength and modulus before failure compared to the untreated fiber reinforced composites prepared from different fibers. The failure indicates the separation between fiber and matrix. This observation can be linked to the fact that chemically treated fibers exhibited better fiber matrix interface bonding which resulted to better fiber matrix adhesion. It was equally observed that with untreated fiber as reinforcement, there was decline in the flexural strength and modulus of the composite as a result of poor adhesion observed between the fiber and matrix.

Under flexural loading, the upper and lower surfaces of the specimen are subjected to higher deformation than the mid plane. Therefore, flexural strength and stiffness depends on the properties of the surface layers (Ishaya and Ohaeri, 2014). *Morinda morindaides* has greater flexural strength than other fibers as evidenced from the results.



(a)



(b)

Fig.4.21. Flexural test of composites (a) Strength (b) Modulus.

4.7.3. Hardness test

The results show that the incorporation of fibers inside the matrix did not reduce the hardness of the matrix but slightly increased it (Fig.4.22). From this investigation, it was clear that the fiber reinforced composite gained huge mechanical properties over the matrix material and thus indicated good fiber matrix adhesion.

High hardness numbers that were obtained from hard composites indicated a shallow indentation while low numbers found with soft composites indicated deep penetration.

From the result (Fig. 4.22), it was observed that the hardness of treated fiber reinforced composites were higher than the hardness of the untreated fiber reinforced composite. The increase in hardness recorded was due to stronger interface bonding of the fiber with the polymer as a result of the chemical treatment. This significant enhancement in hardness can equally be attributed to the distribution of the test load on the fiber, which decreased the penetration of the test ball to the surface of the composite material (Abbasi, 2003). The improvement in the hardness of the composite material could lead to the reduction in wear rate when in service. Comparatively, low hardness was observed for untreated fiber reinforced composite. This can be attributed to the poor dispersion of fibers with the matrix. A careful examination of the result shows that *morinda morindoides* fiber reinforced composite had highest hardness number compared to other fibers reinforced composites. This behaviour can be attributed to the inherent nature of the fiber.

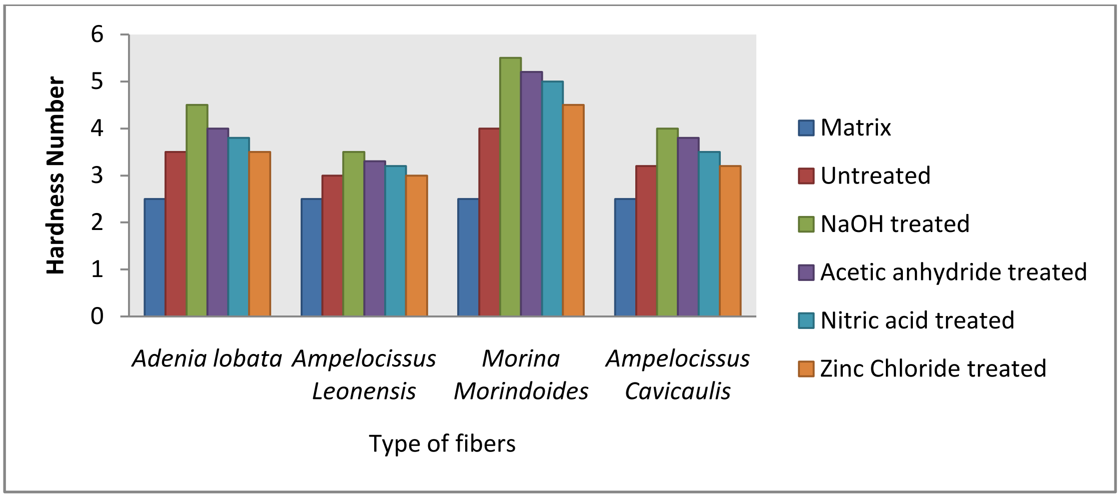


Fig. 4.22. Hardness number of composites

4.7.4. Impact strength

From the results on Figure 4.23 below, it can be seen that the impact strength of the fiber reinforced composites were higher than the polymer itself. It was equally observed that the impact strengths of the treated fiber reinforced composites were higher than the untreated reinforced composites prepared from all the fibers. The enhancement in impact strength as a result of chemical treatment may be ascribed to the good adhesion at the matrix / fiber interface as a result of treatment. Hence, this permits the optimum operation of stress transfer from the matrix to the fibers thus resulting in an improvement of impact strength property. The reduction in impact strength recorded with untreated fiber reinforced composite can be attributed to the formation of fiber agglomerates and voids as a result of impurities on the untreated fibers. This reduces the fiber - matrix adhesion with consequent decrease in impact strength.

It was equally observed that the impact strength of *morinda morindaides* fiber reinforced composites were highest followed by *Adenia lobata* fiber, *Ampelocissus cavicaulis* fiber and lastly *Ampelocissus leonensis* fiber reinforced composite. This outcome was as a result of the inherent nature of the fiber, *morinda morindoides* been the toughest of all.

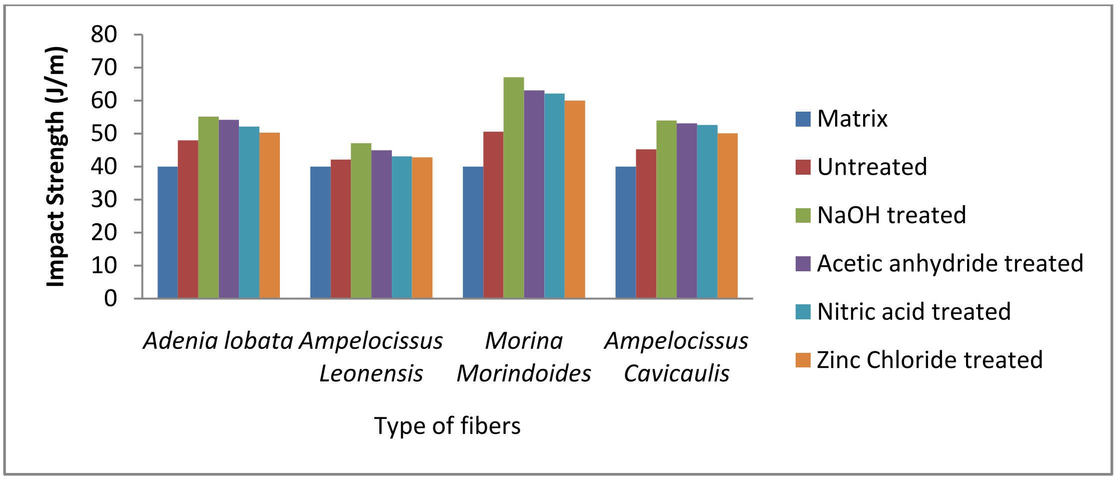


Fig.4.23. Impact strength of composites

4.7.5. Elongation at Break

From the results (Fig.4.24) it was observed that the elongation at break of the polymer was higher than that of composite. This is because of the low elongation at break of the fibers compared to the polymer. This response is characteristics of the materials reinforced with stiff inorganic materials and particularly noteworthy for its interrelated morphology. The reduction in elongation at break as a result of incorporation of fiber as a reinforcement is line with findings of Onuegbu and Igwe, 2011; Fuad et al., 1995 and Basuki et al., 2004. The experimental results show the reduction in the ductility of the composite due to the deformation of a rigid interfacial interaction between the fiber and matrix. This indicates that the composite is tending towards brittle nature. It was equally observed that elongation at break of the untreated reinforced composites were lower than the treated fiber reinforced composites.

The reduction of elongation at break with incorporation of untreated fibers as reinforcement can be attributed to the absence of interfacial adhesion between the fibers and matrix, and also the stiffening effect contributed by the fibers resulted in the reduction of the elongation at break. It was due to the poor interaction between the components stemming from the hydrophilicity of the fibers in the hydrophobic environment. Furthermore, there was aggregation of the fiber particles leading to stress concentration points. It was very interesting to note that in the present investigation, *morinda morindoides* fiber reinforced composite did not exhibit significant elongation at break compared to other composites. This is an indication that *morinda morindoides* reinforced composite is brittle and rigid. The fiber was not flexible enough to significantly restrain the polymer molecules. Consequently, high localized strains might have occurred causing dewetting between the polymer and the fiber, and thus, leaving essentially a matrix that is not ductile. In essence, the stretching resistance of the fiber in the matrix contributed to the observed trend.

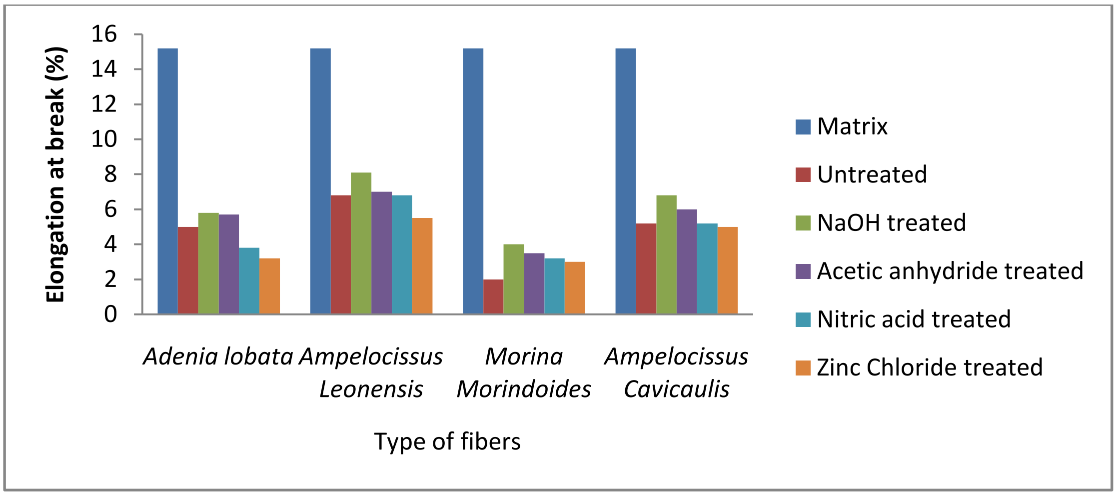


Fig. 4.24. Elongation at break of the composites.

4.7.6. Bending at Break

From the results (Fig.4.25) it was observed that the bending at break of the polymer was higher than the fiber reinforced composites. This was as a result of the ductile nature of the polymer compared to the fibers. This low bending at break of the fiber reinforced composite shows that they are brittle and resists stretching. The lower value of the bending at break of the untreated fiber reinforced composites can be attributed to the poor adhesion between the fibers and polymer that resulted to the fragile nature of the composites. In order words, the composite can easily be damaged without deflection. It is noteworthy to mention that *Ampelocissus cavicaulis* fiber reinforced composite exhibited highest bending at break. This development can be attributed to the inherent nature of the fiber which resulted to its flexible interfacial interaction with the polymer. As a result, it had maximum deflection before bending.

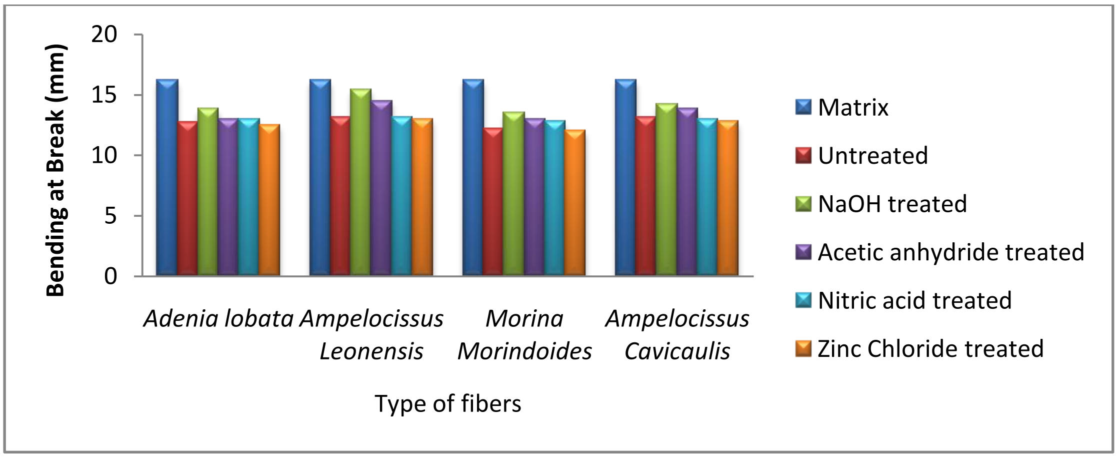


Fig.4.25. Bending at break of the Composites.

4.8. Effect of compounding conditions on the mechanical property of the composites

4.8.1. Effect of Rotation Speed on the tensile strength of the composites

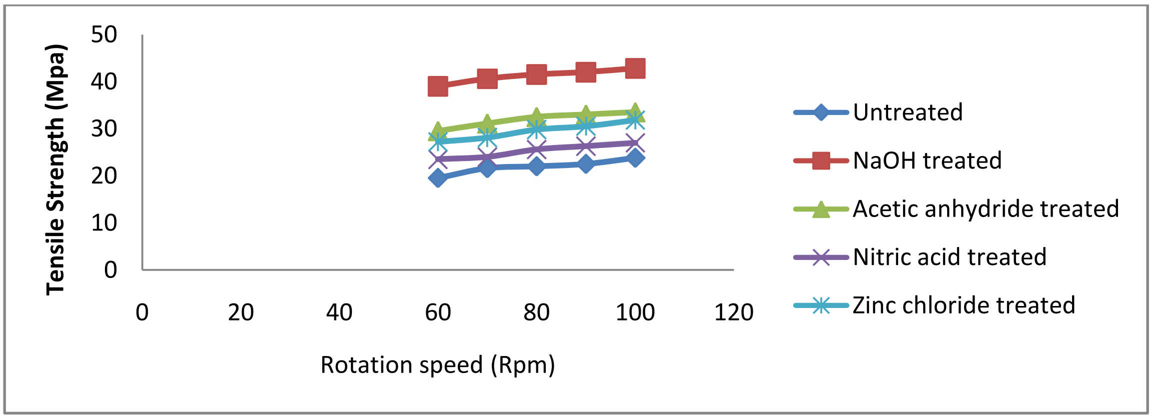
Rotation of the roll mill helps to disperse the fiber uniformly with the matrix. The effect of the rotation speed on the dispersion of the fiber was studied at the midpoints of other factors, temperature of 170^0C and dispersion time of 15mins.

The effect of rotation speed on the compounding of all the untreated fibers and fibers treated with different chemicals are shown in Fig. 4.26.

From the plots, it was seen that increase in rotation speed increased the tensile strength of the resulted composite for the entire untreated blend and the blends treated with different chemicals. All the graphs had similar trends, though with variations on the degree of the effect due to the physio-chemical composition of the fibers and effect of different chemical used in the pretreatments.

The observed trend was attributed to the fact that increase in rotation speed increased the dispersion of the fibers on the polymer.

These findings were in line with the work done by some authors like Takase and shiraishi (1989) and Lu et al., (2004). They established that increasing the rotation speed for the blending increases the mechanical property of the resultant composite due to increased dispersion of the fibers on the polymer.



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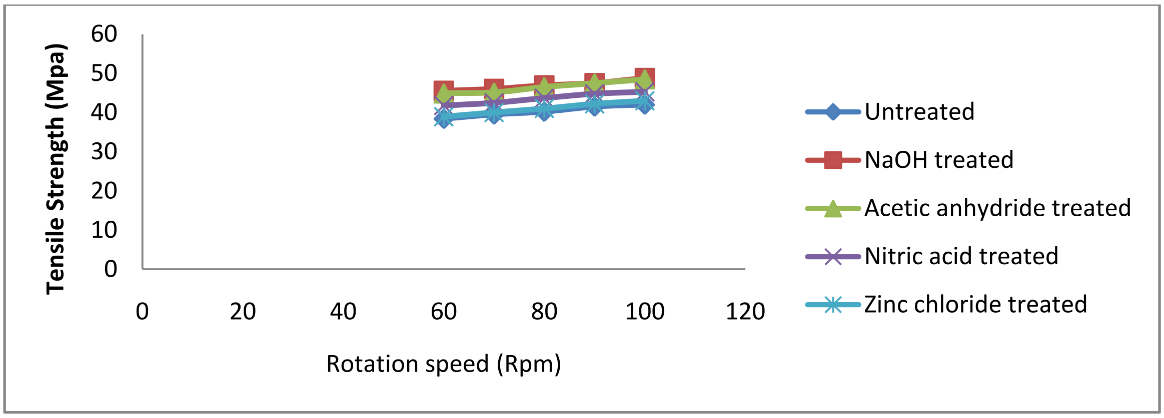

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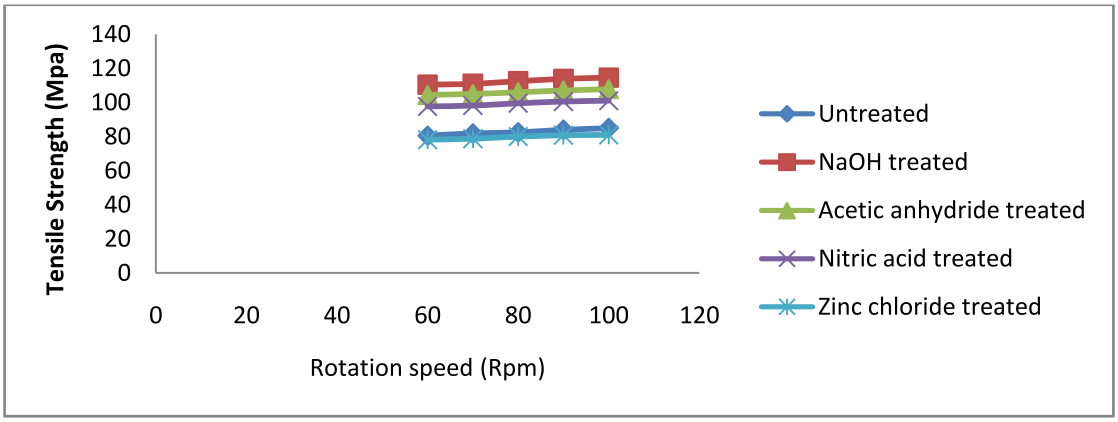

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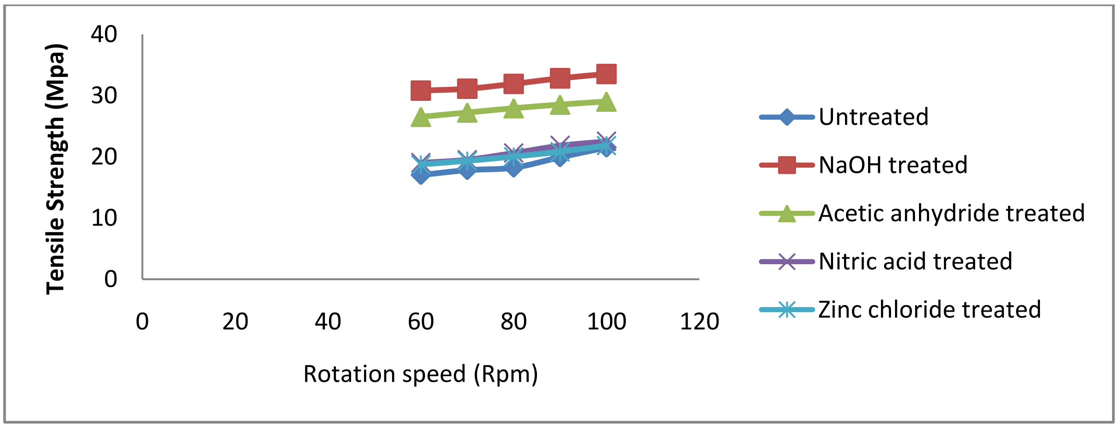


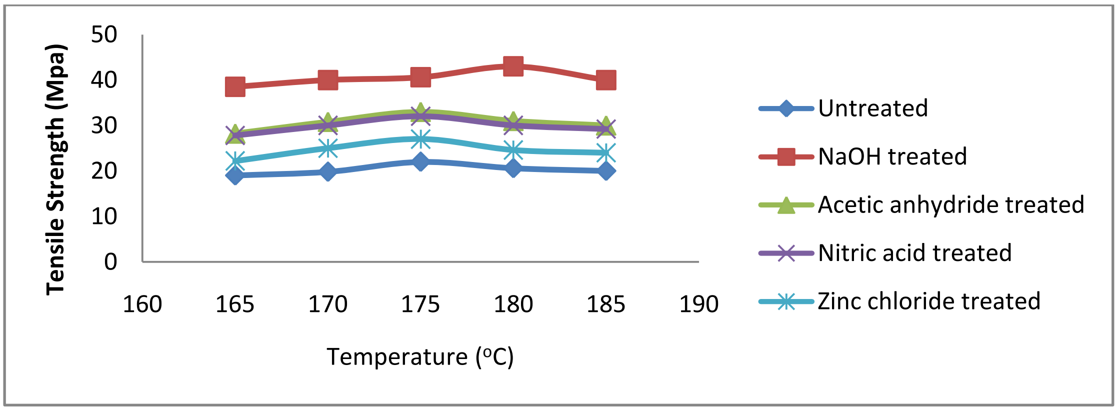
(d)

Fig 4.26**.** Effect of rotation speed on the tensile strength of the composite (a) *Ampelocissus cavicaulis* (b) *Adenia lobata* (c) *Morinda morindoides* (d) *Ampelocissus leonensis*

4.8.2. Effect of Temperature on the tensile strength of the composite

The temperature helps to melt the polymers for easier mixing with the fibers. Temperature effect was studied at the midpoint of other factors, rotation speed of 80rpm and dispersion time of 15mins. Temperature effect on the compounding of fibers both untreated and treated is shown in Fig. 4.27. The plots showed a curvature. Increase in temperature between 165^{0}C and 175^{0}C increased the tensile strength of the composite after which further increase decreased the tensile strength. Similar trends were observed on all the fibers studied, both untreated and treated fibers but the degree of the effect varied due to the differences on the physical and chemical composition of the fibers and on the effects of the chemical used for the pretreatments. Increasing the temperature from 165^{0}C to 175^{0}C helped to melt the polymer and facilitated the dispersion, but the decreasing trend observed at higher temperature was due to decomposition and degradation of the fibers and the polymer under high temperature, which resulted to lower tensile strength.

This was in conformity with the work done by maldas et al., (1989) and Takase and shiraishi (1989). High temperature may cause chain scission of cellulose and polymer molecules (Lu et al., 2004).



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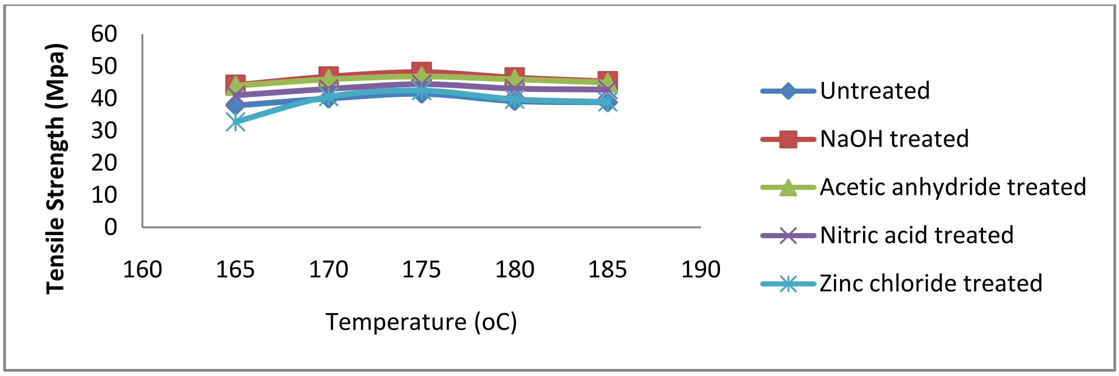

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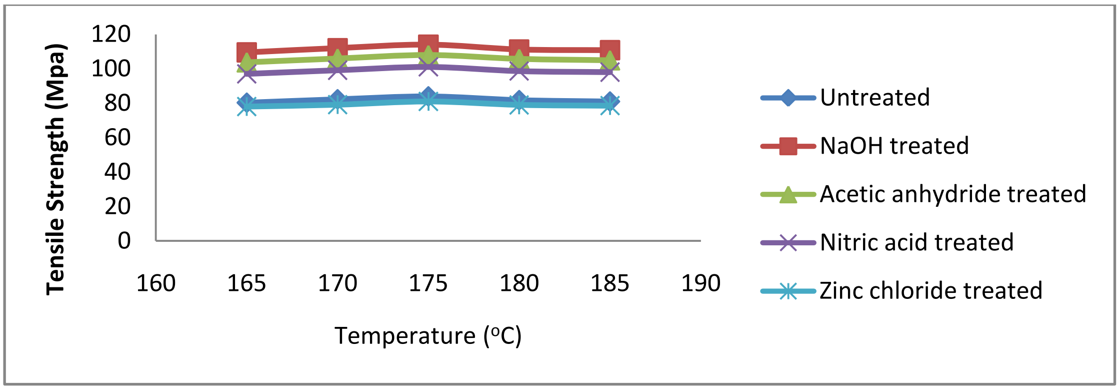

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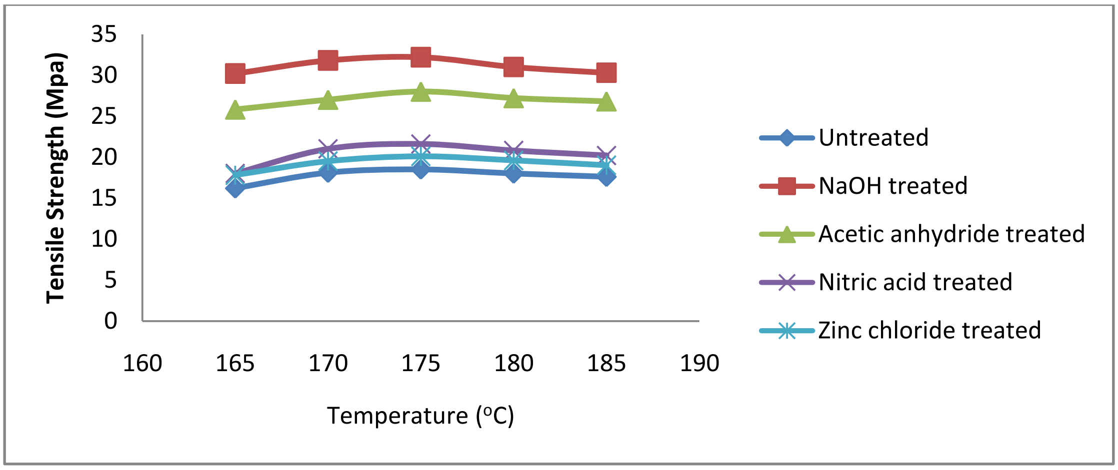
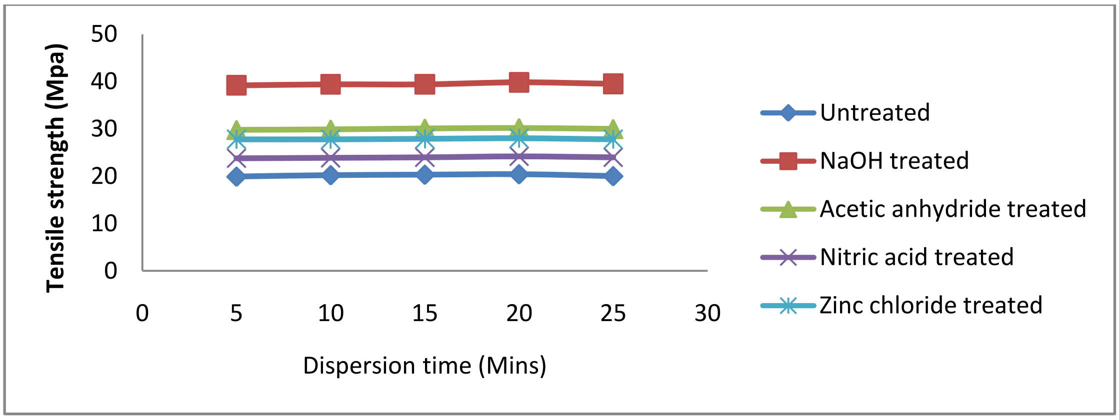


(d)

Fig. 4.27**.** Temperature effect on the tensile strength of the composite (a) *Ampelocissus cavicaulis* (b) *Adenia lobata* (c) *Morinda morindoides* (d) *Ampelocissus leonensis*

4.8.3. Effect of Dispersion Time on the tensile strength of the composites

The effect of dispersion time on the tensile strength of the resultant composites was studied. This study was embarked on to understand how dispersion time will affect the tensile strength of the resultant composites. Effect of dispersion time was studied at the midpoints of other factors; temperature of 170^{0}C and rotation speed of 80rpm. The effects of dispersion time on all the fibers both treated and untreated are shown in Fig. 4.28. All the plots showed the same trend for different types of fiber, but the magnitude of the effect varied. This was as a result of the differences in the chemical composition of the fibers and in the chemicals used for the pretreatment. *Adenia lobata* treated with sodium hydroxide and zinc chloride had marginal time effect. It can be recalled that chemical pretreatment had lowest effect on *Adenia lobata* due to the nature of the fiber, as a result did not effectively modify the fiber surface. High dispersion time probably disrupted the fiber surface due to poor modification.



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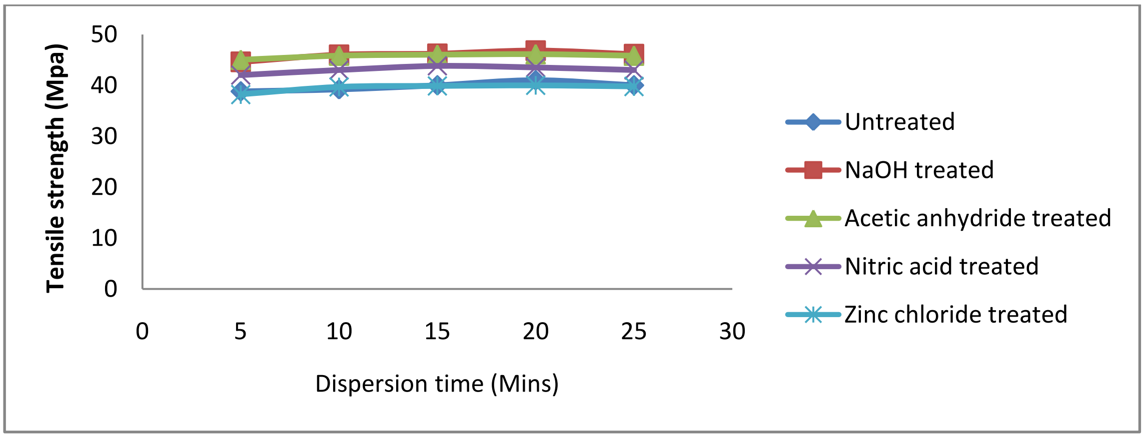

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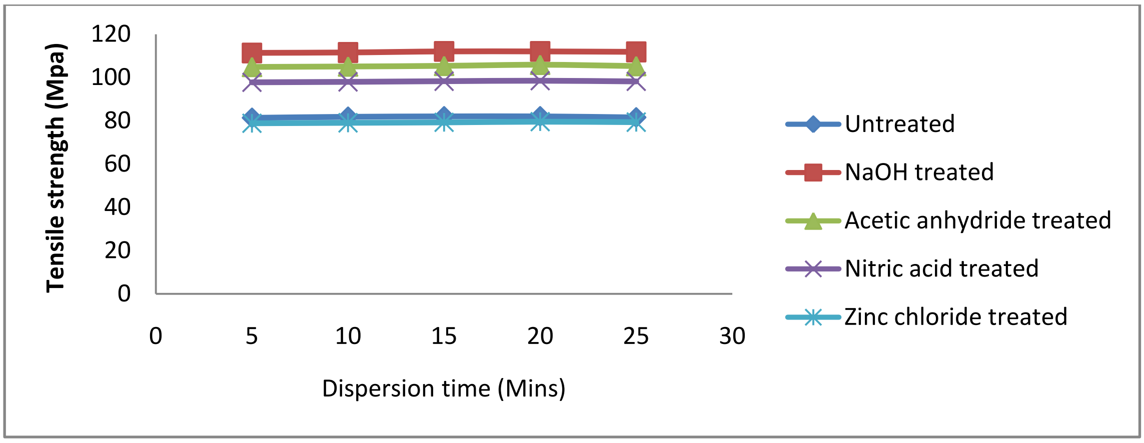

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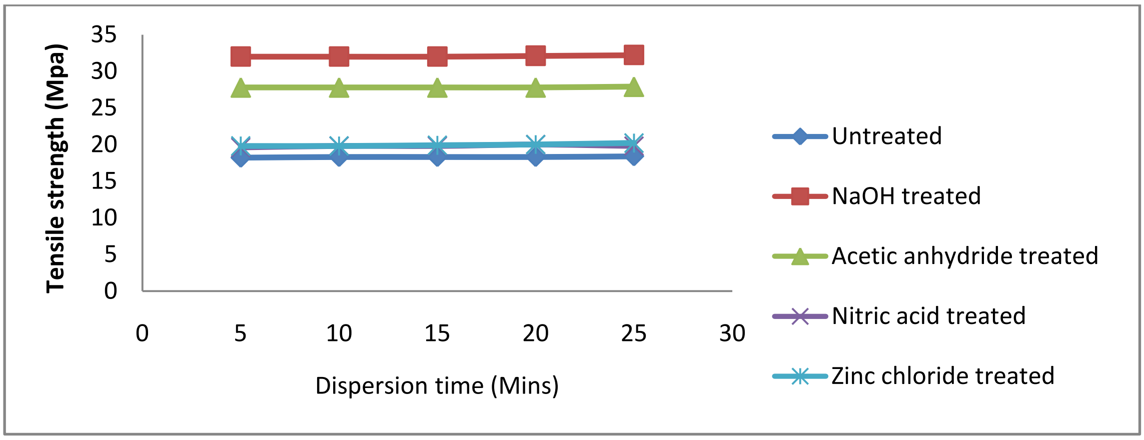


(d)

Fig 4.28. Effect of dispersion time on the tensile strength on the composite (a) *Ampelocissus cavicaulis* (b) *Adenia lobata* (c) *Morinda morindoides* (d) *Ampelocissus leonensis*

4.9. Statistical and Optimization of Composites Compounding Process Based on Optimal Fiber Modifications

Compounding process directly influences compounded quality of fiber-polymer blend and its resultant composites. Blending fiber with a polymer is the key step of composite production, because compounding process helps to uniformly distribute the fiber in the thermoplastic matrix, decrease pore ratio, and stabilizes the filler/matrix interaction (Berlin et al., 1986). There has been no criterion to determine optimum compounding conditions (LU et al., 2004). Usually, compounding condition vary with mixing machine type, compounding steps, weight ratio of fiber and the polymeric matrix, moisture content of fiber, and species of thermoplastic and fiber.

In this process, two roll mill was used for the compounding process.

The optimization of the compounding conditions was done using Box-Behnken design. Three compounding conditions were studied at three levels each with five center points giving 17 experiments each for each fiber with four different chemicals. The experimental runs were randomized to protect against an unknown bias distorting the outcome of the experiment. The design matrix for the compounding process is shown on appendix F. The experimental response was limited to the tensile strength (Mpa) of the composite. In order to identify the effect of the compounding conditions, the resultant blends were separated and used to produce a composite at constant conditions of 180^0C molding temperature, molding time of 10mins, pressure of 15bar and fiber loading of 40% with fiber length of 3mm.

4.9.1. Selection of a good predictive model

A good predictive model is first step toward optimization. Once a good model is established, it can be optimized. Box Behnken design can fit linear, 2FI, and quadratic model. Because BBD has only three levels, it cannot predict cubic model. The best model selected was based on the lack of fit test, P-value of adjusted and predicted R-squared. The sequential model sum of squares was used to select the best model based on the highest order model that was significant (small P-value) and not aliased, no lack of fit (P-value > 0.10) and reasonable agreement between adjusted R-squared and predicted R-squared (within 0.2 of each other). Lack of fit which is the measure of risk was included because some points were replicated (center points) to produce estimate of pure error.

The summary table with the sequential model sum of square for the four fibers treated with four chemicals and the untreated ones are shown on the appendix H.

From the tables on appendix H, it can be seen that the suggested model for all the fibers were quadratic models. Addition of cubic terms to the models did not improve the model but, if it does, Box Behnken design lacks the design points needed to fit all terms required for the cubic model, to that effect, they were labeled as being aliased.

The linear and two factor interaction model (2FI) were not significant (P-value>0.05) and there were large difference between the predicted and adjusted R-squared.

The suggested quadratic models had non significant lack of fit (P-value >01), and good agreement between the predicted and adjusted R-square (within 0.2 of each other).

4.9.2. Inspection of selected model

Models selected using sequential sum of square were inspected for adequacy using analysis of variance (ANOVA). ANOVA was used to test the suggested models, the linear terms, interaction terms and the quadratic terms included in the model. Any term was removed from the model only when it had insignificant P-value (>0.1) or was retained in the model to support model hierarchy. From the ANOVA Tables below, it can be seen that time was insignificant, but was included in the final model to maintain model hierarchy since interaction of time and temperature was significant. Equally, the model was inspected based on its R-Squared, predicted R-squared and adjusted R-squared. Attention was more on the predicted and adjusted R-squared because they can give more estimate of how well the model will give good prediction for the average response. The regular R-squared can artificially rise when any model term is added whether statistically significant or not and as such, can not estimate how well the model predicts the response. There is no cut off value for R-square, the rule of the thumb is that the predicted and adjusted R-square must be within 0.2 of each other (Anderson and Whitecomb 2005).

Another parameter that was used to inspect the model was adequate precision. Adequate precision measures the signal to noise ratio. It compares the range of predicted values at the design point to the average predicated error. A ratio greater than 4 is always desired for a good model (Design expert software 8.0.7.1 2010)

- Analysis of variance for *Ampelocissus cavicaulis* fiber

The analysis of variance (ANOVA) Tables for *Ampelocissus cavicalis* are shown on Tables 4.14 to 4.18 for untreated, acetic anhydride, sodium hydroxide, nitric acid and zinc chloride treated fibers respectively. From the results, it showed that the selected quadratic models were significant based on their low p-values.

For the untreated *ampelocissus cavicaulis* fiber (Table 4.14), the model F-value of 70.5 implied that the selected model was significant. There was only 0.01% chance that a “Model F-value” this large could occur due to noise. Values of F Prob>F less than 0.0500 indicated model terms were significant. In this case single effect of Rotation speed (A), temperature (B), and time (C), interaction of rotation speed and temperature (AB), interaction of temperature and time (BC), quadratic effect of temperature (B^2) and quadratic effect of time (C^2) were significant. Addition of quadratic terms improved the model. Values greater than 0.100 indicated the model terms were not significant and as such, were removed from the model to improve the model. The lack of fit F-values of 1.22 implied the lack of fit was not significant relative to the pure error. There is 73.41% chance that a “lack of fit F-value” this large could occur due to noise. The pred. R-squared of 0.9427 was in reasonable agreement with the Adj R-squared of 0.9630 with high R-squared of 0.9792. Adequate precision of 24.339 was high, which indicated an adequate signal. Having met the conditions stipulated by ANOVA, the model can be said to be good enough to navigate the design space.

Table 4.14. ANOVA Table for untreated *Ampelocissus cavicaulis* for compounding process

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	135.36	8	16.92	70.5	<0.0001
A-Rotation Speed					
B-Temperature	24.68	1	24.68	102.83	<0.0001
C-Time	32.12	1	32.12	133.83	<0.0001
AB	1.5	1	1.5	6.25	0.0221
BC	17.81	1	17.81	74.21	<0.0001
B^2	20.03	1	20.03	83.46	<0.0001
C^2	31.43	1	31.43	130.96	<0.0001
Residual	6.14	1	6.14	25.58	0.0017
Lack of fit	1.92	8	0.24		
Pure error	1.17	4	0.29	1.22	0.7341
Cor Total	0.75	4	0.19		
	137.28	16			

R^2 = 0.9792, Adj-R-squared = 0.9630, Pred. R-squared = 0.9427, Adeq Precision = 24.339

For *ampelocissus cavicaulis* treated with acetic anhydride (Table 4.15), the model F-value of 69.76 implied that the selected model was significant. There was only 0.01% chance that a "model F-value" this large could occur due to noise. Values of "Prob>F" less than 0.0500 indicated model terms were significant. In this case single effect of rotation speed (A), single effect of temperature(B), interaction effect of rotation speed and temperature (AB), interaction effect of temperature and time(BC), quadratic effect of temperature (B^2) and quadratic effect of time (C^2) were significant. The insignificant model terms were removed from the model for model improvement. The lack of fit value of 1.06 implied the lack of fit was not significant relative to the pure error. There is only a 67.67% chance that a lack of fat F-value this large could occur due to noise. Since our target is for the model to fit, Insignificant lack of fit is desirable (0.9790) indicated high adequacy of the model. The pred R-square of 0.9413 was in reasonable range with Adj. R-squared of 0.9626. Adeq. precision which measured the signal to noise ratio is high enough indicating adequate signal. The analysis led to the conclusion that the selected model can be used to predict the response.

Table 4.15. ANOVA Table for compounding of *Ampelocissus cavicaulis* treated with Acetic anhydride.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	141.20	8	17.65	69.76	<0.0001
A-Rotation Speed					
B-Temperature	25.85	1	25.85	102.17	<0.0001
C-Time	32.64	1	32.64	129.01	<0.0001
AB	2.11	1	2.11	8.34	0.0102
BC	18.92	1	18.92	74.78	<0.0001
B^2	18.84	1	18.84	74.47	<0.0001
C^2	32.82	1	32.82	129.72	<0.0001
Residual	8.08	1	8.08	31.94	0.0052
Lack of fit	2.02	8	0.253		
Pure error	1.36	4	0.27	1.06	0.6767
Cor Total	0.66	4	0.17		
	143.22	16			

R-squared = 0.9790, Adj R-squared = 0.9626, Pred. R-squared = 0.9413, Adeq precision = 24.109.

For *ampelocissus cavicaulis* treated with sodium hydroxide (table 4.16), the model F-value of 79.27 implied that the selected model was significant. There was only 0.01% chance that a “model F-value” this large could occur due to noise, values of ‘Prob>F less than 0.0500 indicate that the model terms were significant. In this case, single effect of rotation speed (A), temperature (B), time (C), interaction effect of rotation speed and temperature (AB), interaction effect of temperature and time (BC), quadratic effect of temperature (B^2) and the quadratic effect of time (C^2) were significant. The non significant terms based on the prob>F were removed to improve the mode. The lack of fit F-value of 1.05 implied the lack of fit was not significant relative to the pure error. There was only a 73.67% chance that a “lack of fit F-value” this large could occur due to noise since lack of fit is a measure of risk, insignificant lack of fit was desired. The predicted R-squared of 0.9375 was in good agreement with Adjusted R-squared of 0.96.50 with high R-square value of 0.9803. Adequate precision which measures the signal to noise ratio was high (24.888). A value greater than 4 is desirable. The value obtained confirmed that the selected model was adequate to predict the response.

Table 4.16. ANOVA Table for compounding *Ampelosicuss cavicaulis* treated with NaOH

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	139.51	8	17.44	79.27	<0.0001
A-Rotation Speed					
B-Temperature	24.89	1	24.89	113.14	<0.0001
C-Time	32.89	1	32.89	149.5	<0.0001
AB	1.62	1	1.62	7.36	0.0410
BC	17.06	1	17.06	77.55	<0.0001
B^2	22.28	1	22.28	101.27	<0.0001
C^2	32.83	1	32.83	149.23	<0.0001
Residual	6.24	1	6.24	28.36	0.0015
Lack of fit	1.79	8	0.22		
Pure error	1.14	4	0.23	1.05	0.7367
Cor Total	0.65	4	0.16		
	141.30	16			

R-squared = 0.9803, Adj R-squared = 0.9650, pred. R-squared = 0.9375, Adq. precision = 24.888.

For *ampelocissus cavicaulis* treated with nitric acid (Table 4.17), the model F-value of 60.82 implied that the model is significant. There is only a 0.01% chance that a "model F-value: this large could occur due to noise. Values of "prob>F" less than 0.0500 indicate model terms are significant. In this case, the single effect of rotation speed (A), temperature (B), time (C), the interaction effect of temperature and time (BC), the interaction effect of rotation speed and temperature (AB), the quadratic effect of temperature (B^2) and the quadratic effect of time (C^2) were significant. The insignificant effects were removed to improve the model. The lack of fit F-value of 1.10 implied that the lack of fit was not significant relative to the pure error. There is only 76.36% chance that a lack of fit F-value this large could occur due to noise. Non significant lack of fit is desirable. The predicted R-squared of 0.9438 was in good agreement with the adjusted R-squared of 0.9589 with high correlation coefficient of 0.9769. Adequate precision of 23.198 was adequate. The analysis showed that the selected model can be used to navigate the design space.

Table 4.17. ANOVA Table for compounding *Ampelosicus cavicaulis* treated with nitric Acid

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	136.27	8	17.03	60.82	<0.0001
A-Rotation Speed					
B-Temperature	25.06	1	25.06	89.5	<0.0001
C-Time	35.03	1	35.03	125.11	<0.0001
AB	1.94	1	1.94	6.93	0.0438
BC	15.72	1	15.72	56.14	<0.0001
B^2	21.02	1	21.02	75.07	<0.0001
C^2	29.53	1	29.53	105.46	<0.0001
Residual	6.33	1	6.33	22.61	0.0022
Lack of fit	2.20	8	0.28		
Pure error	1.24	4	0.31	1.10	0.7636
Cor Total	0.96	4	0.24		
	138.46	16			

R-squared = 0.9769, Adj. R-squared = 0.9589, pred. R-squared = 0.9438, Adeq. Precisiion = 23.198.

For *ampelocissus cavicaulis* treated with zinc chloride (Table 4.18), the model F-value of 81.34 implied the model was significant. There is a 0.01% chance that a "model F-value" this large could occur due to noise. Values of 'prob>F" less than 0.0500 indicated model terms are significant. In the case the single effect of rotation speed (A), temperature (B) and time (C), the interaction effect of rotation speed and temperature (AB), the interaction effect of temperature and time (BC), the quadratic effect of temperature (B^2) and the quadratic effect of time (C^2) were significant. The lack of fit F-value of 0.6 implied the lack of fit was not significant relative to the pure error. There is a 72.93% chance that a lack of "fit F value" this large could occur due to noise. Since lack of fit is a measure of risk, low F-value was desired. 97.34% of the total variation of the response will be well explained by the model as indicated by the R-squared value. The predicted R-squared was within 0.2 ranges with adjusted R-squared as prescribed by statistics. The adequate precision that measured signal to noise ratio was high enough (21.305), this indicated adequate signal. The analysis of variation done showed that the selected mode can be used to navigate the design space.

The ANOVA Tables for other fibers are shown on appendix I and the models were selected in a similar way as described above.

Table 4.18. ANOVA Table for compounding of *Ampelocessus cavicaulis* treated with zinc chloride

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	136.65	8	17.08	81.34	<0.0001
A-Rotation Speed	26.06	1	26.06	124.10	<0.0001
B-Temperature	30.81	1	30.81	146.71	<0.0001
C-Time	2.56	1	2.56	12.19	0.0270
AB	15.92	1	15.92	75.81	<0.0001
BC	20.88	1	20.88	99.43	<0.0001
B^2	32.14	1	32.14	153.05	<0.0001
C^2	6.53	1	6.53	31.10	0.0031
Residual	1.67	8	0.21		
Lack of fit	0.5	4	0.13	0.6	0.7288
Pure error	1.17	4	0.29		

R-Squared = 0.9734, Adjusted R-squared = 0.9527 predicated R squared = 0.9174, Adeq. Precision = 21.305.

4.9.3. Model equations for the compounding process

The model equations generated are mathematical representation of the compounding process after removing the non significant factors. It was used to predict the response which was the tensile strength of the composite. It was presented in both actual and coded values. The coded values can only be used in predicting the response only when the factors are converted to the standard codes of -1, + 1 and “0” on the design matrix. Where “-1” represents the low value of the factorial range, “+1” represents the high value of the factorial range and “0” represents the center point. The two types of model equations can give an approximation that leads to the proper direction. The coefficients of the coded equations give the relative contribution of each factor to the response because their units of measure have been removed, but actual equations cannot give such contributions because their coefficients depend on their unit of measure.

- Model equations for *compounding* of *Ampelocissus cavicaulis* fiber

The equations are shown on equations 4.38 to 4.47 after removing insignificant factors.

Untreated *Ampelocissus cavicaulis* fiber**,** the final equation in terms of coded factors;

$\text{Tensile strength (Mpa)} = +21.22 + 1.76\text{A} + 2\text{B} - 0.26\text{C} - 2.11\text{AB} - 2.24\text{BC} - 2.73\text{B}^2 - 1.21\text{C}^2$
(4.38)

Final equation in terms of actual factors;

Tensile strength (Mpa) = - 1077.53257 + 1.88131Rotation speed +10.99136 Temperature +19.10679Time – 0.010550Rotation speed x temperature – 0.044750 temperature x Time – 0.027282Temperature^2 – 0.048226Time^2 (4.39)

From equation 4.38, the quadratic effect of temperature (B^2) had highest effect on the tensile, though negative effect. The inclusion of the quadratic term decreased the overall intercept by 2.73 and equally increased the overall intercept by 2.00 due to its linear effect. Temperature decreased the tensile strength due to its interaction with time by 2.24. The inclusion of temperature equally decreased the slope due to its interaction with rotation speed by 2.11. The overall intercept was increased by 1.76 with the inclusion of rotation speed in the model. The quadratic term of time included in the model decreased the overall intercept by 1.21 and its linear effect equally increased the overall intercept by 0.26.

The final equation in terms of coded factors for *Ampelocissus cavicaulis* treated with sodium hydroxide:

Tensile (Mpa) = + 40.86+ 1.76A + 2.03B + 0.28C – 2.07AB – 2.36BC – 2.79B^2 - 1.22C^2 (4.40)

Final equation in terms of Actual factors;

Tensile (Mpa) = - 1079.03283 + 1.84344Rotation speed + 11.21693Temperature + 9.53870Time – 0.010325Rotation speed x Temperature – 0.047200Temperature xTime – 0.02788Temperature^2 – 0.048632Time^2 (4.41)

The final equation in terms of coded factors for *Ampelocissus Cavicaulis* treated with acetic anhydride;

Tensile strength (Mpa) = $+31.43 + 1.80A + 2.02B + 0.37C - 2.18AB - 2.17BC - 2.79B^2 - 1.38C^2$ (4.42)

The final equation in terms of actual factor Tensile strength (Mpa) = $-1088.00882 + 1.93862$Rotation speed $+ 11.20274$ Temperature $+ 9.11229$ Time $- 0.010875$ rotation speed x Temperature $- 0.043400$Temperature x Time$-$ 0.027882 Temperature^2 $- 0.055326$Time^2 (4.43)

The final equation in terms of coded factors for *Ampelocissus cavicaulis* treated with nitric acid;

Tensile strength (Mpa) = $+25.26 + 1.77A + 2.09B + 0.34C - 1.98AB - 2.29BC - 2.64B^2 - 1.22C^2$ (4.44)

The final equation in terms of actual factors;

$- 1045.41645 + 1.77362$ Rotation speed $+10.68121$Temperature $+ 9.33237$ Time $- 9.91250$E-003Rotation speed X Temperature $- 0.045850$ Temperature X Time $- 0.026445$ Temperature^2 $- 0.048979$ Time^2 (4.45)

The final equation in terms of coded factors for *Ampelocissus cavicaulis* treated with Zinc Chloride

Tensile strength (Mpa) = $+ 28.97 + 1.81A + 1.96B + 0.26C - 2.00AB - 2.29BC - 2.76B^2 - 1.24C^2$ (4.46)

The final equation in terms of actual values;

Tensile strength (Mpa) = $-1073.13539 + 1.78600$Rotation speed $+11.06017$ Temperature $+ 9.31474$ Time $- 9.9500$E-003 Rotation speed x Temperature $- 0.045700$ Temperature x Time $- 0.027589$ Temperature^2 $- 0.049758$ Time^2 (4.47)

- Model equations for compounding of *Adenia lobata* fiber

The model equations are shown on equations 4.48 to 4.57 for both the untreated and treated fibers after removing insignificant factors.

The Final equation in terms of coded factors for Untreated *Adenia lobata*;

Tensile strength (Mpa) = $+40.21 +1.79A + 2.04B + 0.24C - 2.11 AB -2.42 BC - 2.78B^2 - 1.21C^2$ (4.48)

Final equation in terms of Actual factors;

Tensile strength (Mpa) = - 1084.78947 + 1.88087 Rotation speed + 11.24136 Temperature + 9.73568 Time – 0.0105 Rotation speed. Temperature – 0.048450 Temperature. Time – 0.2847 Temperature^2 = 0.048389 Time^2 (4.49)

Final equation in terms of coded factors for *Adenia lobata* treated with Sodium Hydroxide;

Tensile strength (Mpa) = + 46.92 +1.70A + 2.60 B + 2.58C – 2.10AB -1.59BC – 2.93B^2 – 1.67C^2 (4.50)

Final equation in terms of Actual factors;

Tensile strength (Mpa) = - 1074.68697 + 1.8562 Rotation speed + 11.44373 Temperature + 7.52093 Time – 0.010475 Rotation speed. Temperature – 0.031750 Temperature. Time – 0.29322 Temperature^2 - 0.066889 Time^2 (4.51)

Final equation in terms of coded factors for *Adenia lobata* treated with Acetic Anhydride;

Tensile strength (Mpa) = + 46.56 + 1.69A + 2.07B + 0.26C – 1.98AB - 2.24BC – 2.79B^2 – 1.15C^2 (4.52)

Final equation in terms of Actual factors;

Tensile strength (Mpa) = - 1052.67954 + 1.77194 Rotation speed + 11.16772 Temperature + 9.03447 Time – 9.9250E - 003 Rotation speed Temperature – 0.044750 Temperature. Time – 0.02792 Temperature^2 - 0.045816 Time^2 (4.53)

Final equation in terms of coded factors for Adenia Lobtata treated with Nitric Acid;

Tensile strength (Mpa) = +43.65 + 1.83A + 1.96B + 0.30C – 2.03AB – 2.16BC – 2.74B^2 – 1.15C^2 (4.54)

Final equation in terms of Actual factors,

Tensile strength (Mpa) = - 1047.80362 + 1.81931 Rotation speed + 10.96665 Temperature + 8.76892 Time – 0.010762 Rotation speed. Temperature – 0.043100 Temperature. Time – 0.027387 Tempeature^2 – 0.046047 Time^2 (4.55)

Final equation in terms of coded factors for *Adenia lobata* treated with Zinc Chloride;

Tensile strength (Mpa) = + 40.58 + 2.10A + 2.03B + 0.64C – 1.92AB – 1.05AC – 2.24BC – $2.47B^2$ – $1.51C^2$ (4.56)

Final equation in terms of Actual factors;

Tensile strength (Mpa) = - 990.31737 + 1.89700 Rotation speed + 10.05428 Temperature + 10.39342 Time – 9.61250E-003 Rotation speed Temperature- 0.010525 Rotation speed. Time – 0.044750 Temperature. Time – 0.02473 Temperature^2 = 0.06547 Time^2 (4.57)

- Model equations for compounding of *Morinda morindoides* fiber

Final equation in terms of coded factors for Model equations for untreated Morinda morindoides after removing insignificant factors;

Tensile strength (Mpa) = +82.81 + 2.28A + 1.28B + 0.26C – 1.55AB – 1.05AC – 2.73BC – $2.91B^2$ – $1.33C^2$ (4.58)

Final equation in terms of Actual values

Tensile strength (Mpa) = - 1060.17349 +1.58706 Rotation speed + 11.76359 Temperature + 11.76580 Time – 7.7350E -003 Rotation speed. Temperature – 0.010500 Rotation speed. Time – 0.054550 Temperature. Time – 0.029149 Temperature^2 - 0.053295 Time^2 (4.59)

Final equation in terms of coded values for Morinda morindoides treated with sodium hydroxide;

Tensile strength (Mpa) = + 112.49 + 1.96A + 2.36B +0. 20C – 2.82AB – 1.98BC – $329B^2$ + - $0.98C^2$ (4.60)

Final equation in terms of actual values;

Tensile strength (Mpa) = - 1189.67046 + 2.49094 Rotation speed + 13.15891. Temperature + 7.96728 Time = 0.014075 Rotation speed – Temperature – 0.039700 Temperature. Time - 0.032946 Temperature^2 – 0.039284 Time^2 (4.61)

Final equation in terms of coded values for *Morinda morindoides* treated with acetic anhydride;

Tensile Strength (Mpa) = + 106.13 + 1.69A - 2.01B + 0.44C – 1.98AB – 2.22BC – $2.41B^2$ – $1.15C^2$ (4.62)

Final equation in terms of actual values;

Tensile strength (Mpa) = - 892.21816 + 1.76962 Rotation speed + 9.86799 Temperature + 9.02014 Time – 9.91250E – 003 Rotation speed. Temperature – 0.044400 Temperature Time – 0.024141 Temperature^2 – 0.046163 Time^2 (4.63)

Final equation in terms of coded values for *Morinda morindoides* treated with nitric acid;

Tensile strength (Mpa) = + 99.36 + 1.76A + 2.00B + 0.26C – 2.11 AB – 2.24BC – $273B^2$ – $1.21C^2$ (4.64)

Final equation in terms of actual values ;

Tensile strength (Mpa) = - 998.62809 + 188131 Rotation speed + 10.98288 Temperature + 9.10186 Time – 0.010550 Rotation speed Temperature – 0.044700 Temperature Time – 0.027259 Temperature^2 – 0.048337 Time^2 (4.65)

Final equation in terms of coded values for *Morinda morindoides* treated with zinc chloride;

Tensile strength (Mpa) = + 79.62 + 1.13A + 1.09B – 1.00E–002C – 1.48AB – 1.21BC – $1.57B^2$ – $0.82C^2$. (4.66)

Final equation in terms of Actual values;

Tensile strength (Mpa) = - 567.098494 + 1.31006 Rotation speed + 6.40718. Temperature + 5.09134 Time – 7.37500E- 003 Rotation speed.Temperature – 0.024150 Temperature Time – 0.015724 Temperature^2 – 0.032795 Time^2 (4.67)

- Model equations for the compounding of *Ampelosiccus leonensis* fiber

The model equations for the untreated fiber and fibers treated with four different chemicals are shown on equation 4.63 to equation 4.72 after removing insignificant factors.

Final equation in terms of the coded values for untreated *Ampelocissus leononsis* fiber

Tensile strength (Mpa) = + 18.67+ 1.55A + 1.85B + 0.34C -1.89 AB – 1.80 BC – 2.30 B^2 (4.68)

Final equation in term of actual values;

Tensile strength (Mpa) = -905.45500 + Rotation speed +9.30474 Temperature + 6.18800 Time – 9.4625 E – 003 Rotation speed. Temperature – 0.03600 temperature. Time - 0.023007. Temperarure^2 (4.69)

Final equation in terms of coded factors for Ampelocissus leonensis treated with sodium hydroxide;

Tensile strength (Mpa) = + 32. 07 + 1.40 A + 1.90B – 0.27C – 1.77 AB – 1.83 BC – 2.40B^2 (4.70)

Final equation in terms of actual factors;

Tensile strength (Mpa) = - 913.37250 + 1.57431 Rotation Speed + 9.60118 Temperature + 6.26800. Time – 8.8500E – 003 Rotation speed. Temperature – 0.036550 Temperature Time – 0.023985 Temperature^2 (4.71)

Final equation in terms of coded values for Ampelocissus leonensis treated with acetic anhydride;

Tensile strength (Mpa) = + 27.94 + 1.34 A + 1.98B + 0.18C – 1.92 AB – 1.68 BC – 2.43B^2 (4.72)

Final equation in terms of actual values;

Tensile strength (Mpa) = - 929. 71000 - + 1.69631 Rotation speed + 9.72865 Temperature + 5.73850 Time – 9.57500E $^{-\ 003}$ Rotation speed. Temperature – 0.033550 Temperature Time – 0.024299 Temperature^2 (4.73)

Final equation in terms of coded values for Ampelocissus leoneusis treated with nitric acid;

Tensile strength (Mpa) = + 20.64 + 1.69A + 197 B + 0.17C – 1.98AB – 2.04BC – 2.67B-2- 1.03C^2 (4.74)

Final equation in terms of actual values;

Tensile strength (Mpa) = - 1039.6342 - 1.76537 Rotation speed + 10.67778 Temperature + 8.22778 Time – 9.88750E – 003 Rotation speed Temperature – 0.040900 Temperature. Time – 0.026696 Temperature^2 – 0.041384 Time^2 (4.75)

Final equation in terms of coded values for Ampelocissus leonensis treated with zinc chloride;

Tensile strength (Mpa) = + 20.06 + 1.45A + 1.91 B + 0.47C – 1.77 AB – 1.84BC – 2.32B^2 (4.76)

Final equation in terms of actual values;

Tensile strength (Mpa) = - 903.40875 + 1.52500 Rotation speed + 9.322826 Temperature + 6.36675 Time – 8.83250E-003 Rotation speed. Temperature – 0.036900 temperature. Time – 0.023168 Temperature^2 (4.77)

4.9.4. Validation of model equations for compounding process

It is always necessary to check the adequacy of the model equations generated, if they can be used to obtain good predictions. The easiest way of doing this is to diagnose residuals generated. Residuals are estimate of experimental error obtained by subtracting the observed responses from the responses predicated by the model. Since residuals are considered as an experimental error, it is expected that they should be roughly normal. Therefore, ANOVA assumed that they have constant variance and followed normal distribution; Careful look at these residuals will tell one if the assumptions made by ANOVA are adequate and choice of model appropriate. Departure from these assumptions means that the residuals contain structures that were not accounted for in the model. Identifying the structure and adding terms representing it to the original model gives a better model. The residuals were diagnosed for patterns that indicate that something other than noise was present. If the residuals were pure noise, then, the analysis is complete.

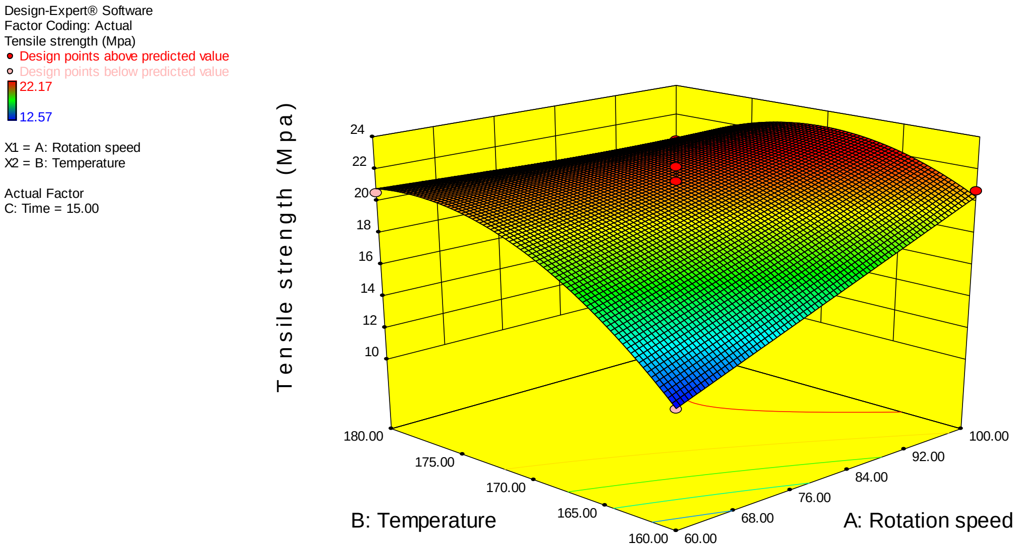
The best way of diagnosing the residual is to observe the behavior of normal probability plots, plots of residuals versus predicted response values, plots of residuals versus experimental run order and the graph of predicted response values versus actual response values. The graphs of the residual analysis showed that the residuals were pure noise.

4.9.4. 3D Surface plots

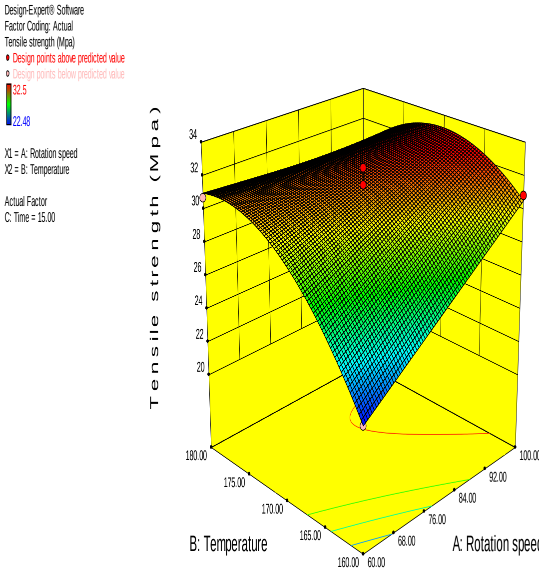
The 3D surface plot is a projection of the contour plot giving shape to the colours. The shape of the 3D surface plot equally gives an idea of the nature of the model equation and the type of optimization involved. The shape of the colour gives an idea on the region of the optimum conditions.

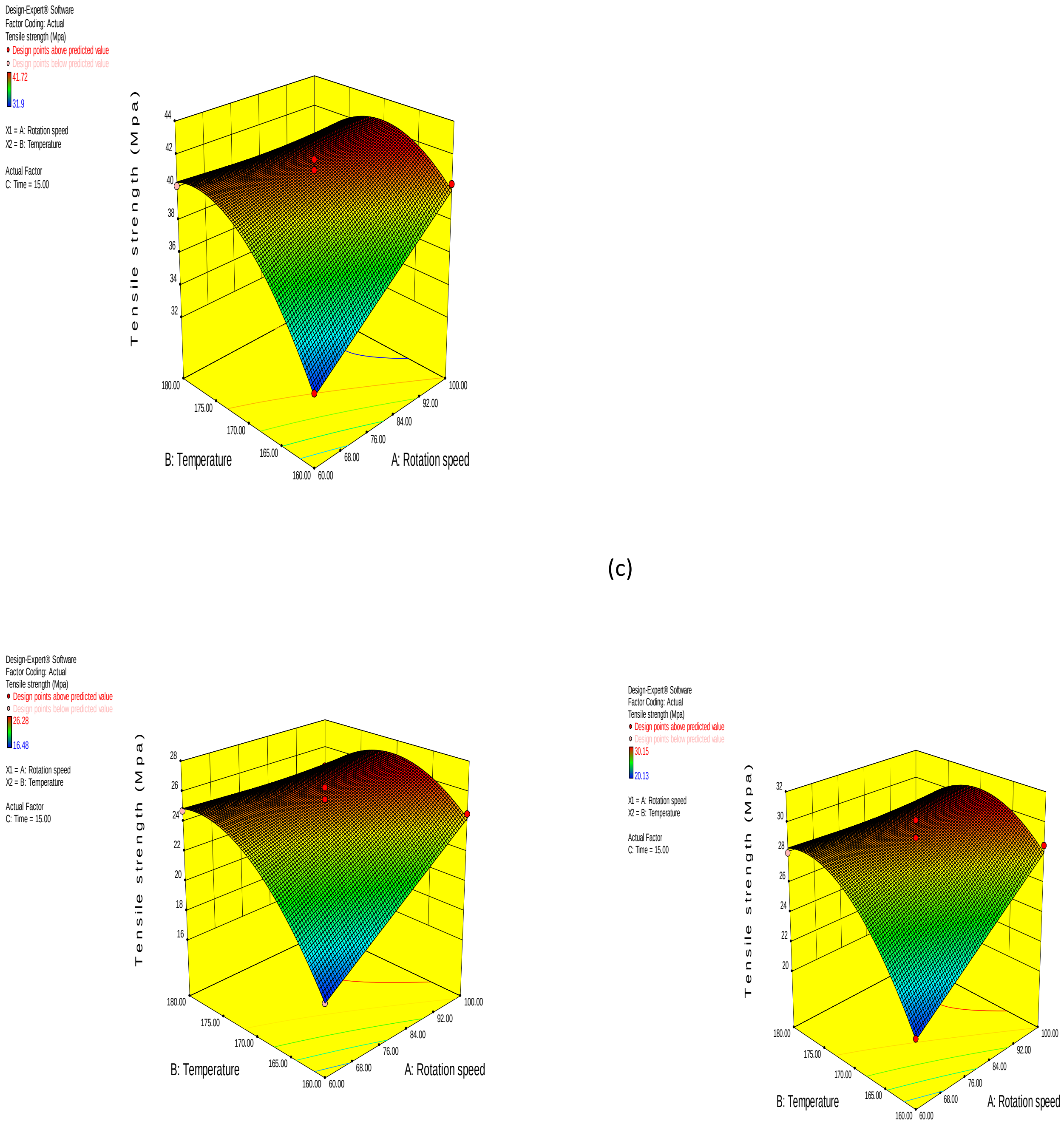
4.9.4.1. 3D Surface plot of interaction effect of temperature with rotation speed

The interaction effect of temperature with rotation speed was studied using 3D surface plot. The plots for all the untreated and treated fibers are shown in Figures 4.29 to 4.32. All the plots displayed the same shape for all the fibers. It bulged out showing maximization optimization, maximizing the response which is the tensile strength.



(a)



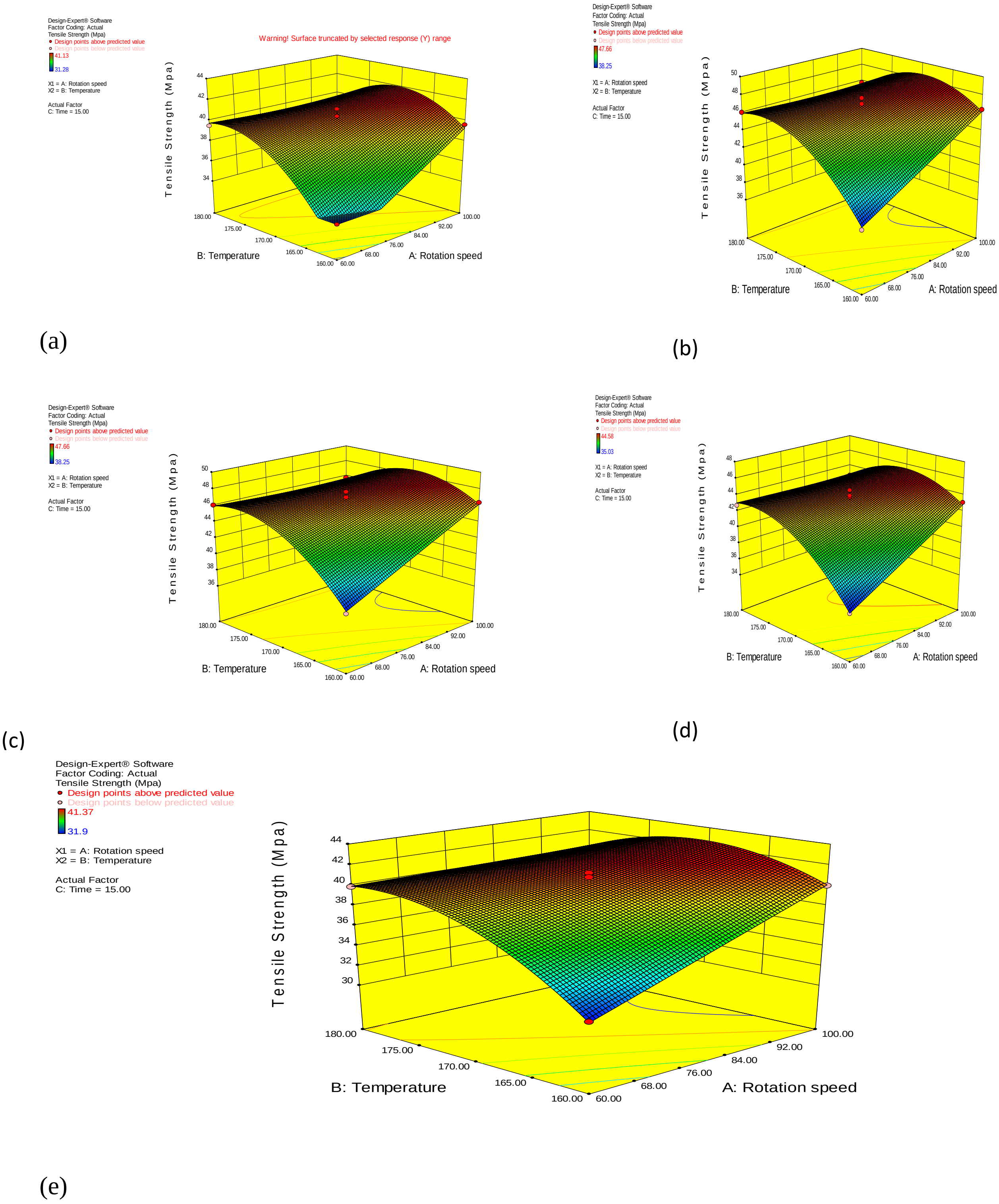


(c)

(d)

(e)

Fig. 4.29. 3D surface plots of interaction effect of temperature with rotation speed on the tensile strength of *ampelocissus cavicaulis* fiber reinforced composite (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.



(a)

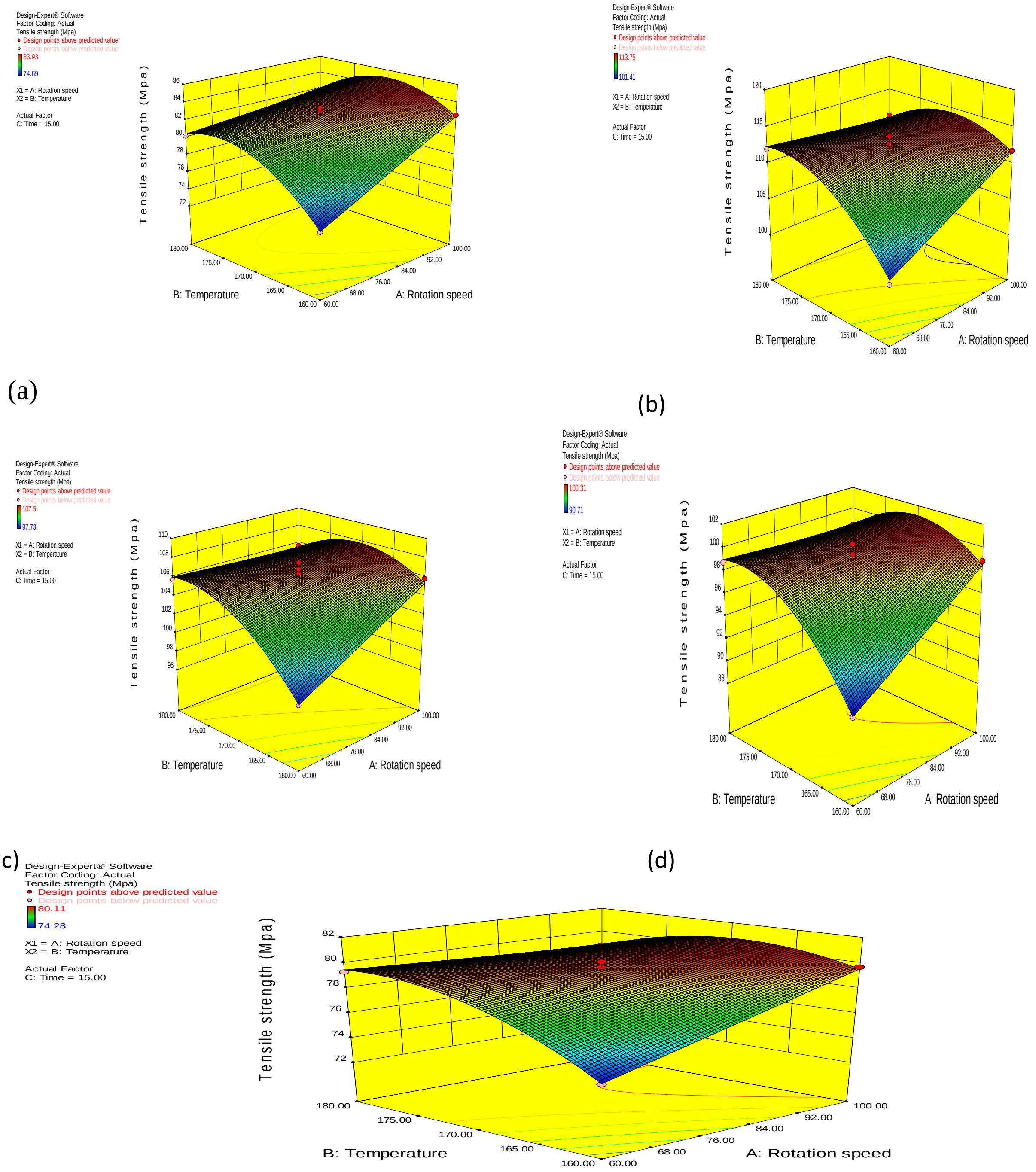
(b)

(c)

(d)

(e)

Fig. 4.30. 3D surface plots of interaction effect of temperature with rotation speed on the tensile strength of *adenia lobata* composite (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.



(a)

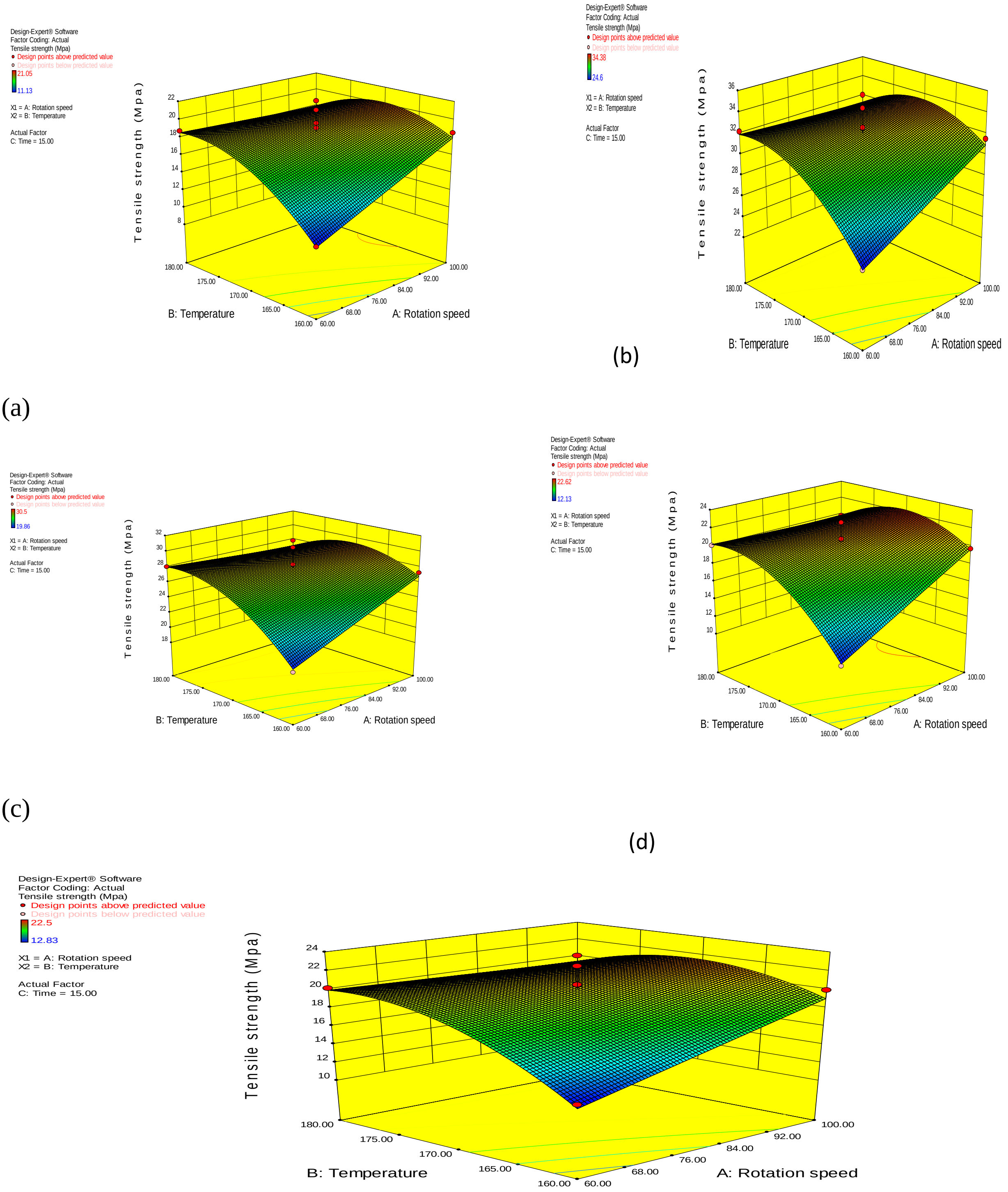
(b)

(c)

(d)

(e)

Fig. 4.31. 3D surface plots of interaction effect of temperature with rotation speed on the tensile strength of *morinda morindoides* composite (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.



(a)

(b)

(c)

(d)

(e)

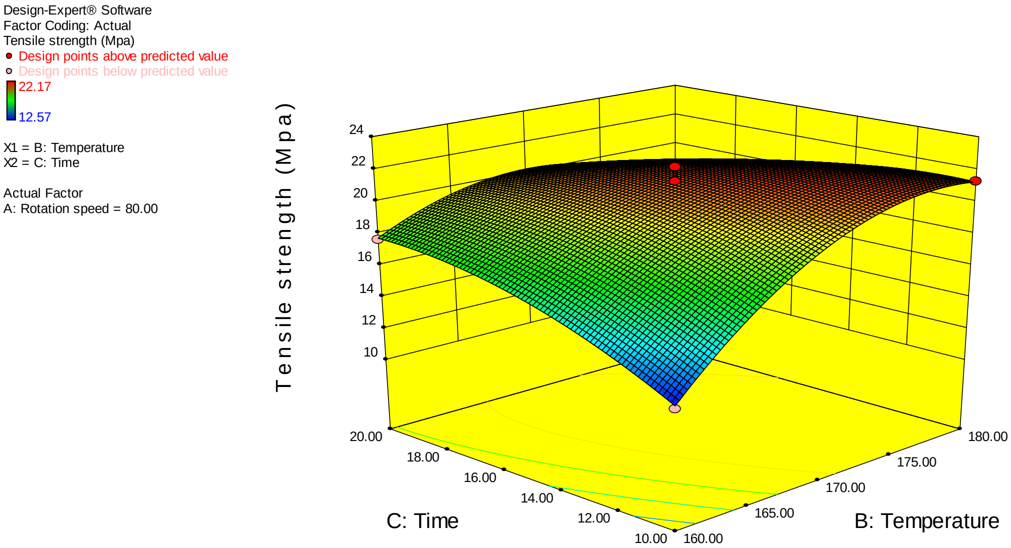
Fig. 4.32. 3D surface plots of interaction effect of temperature with rotation speed on the tensile strength of *ampelocissus leonensis* composite (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.

As the hill was climbed with increase in temperature and rotation speed, the tensile strength of the composites increased to the apex of the surface after which further increase in temperature and rotation speed resulted to decrease in tensile strength. Using the color coding, the colour was hottest at the apex of the hill, meaning that the tensile strength was highest at that point. Therefore the optimum conditions lied on the apex of the hill. The surface of the rotation speed was linear showing that tensile strength increased with increases in rotation speed at lower temperature range, but that of the temperature had curvature showing that temperature had limit at which when exceeded will result to decrease in tensile strength. At lower temperature, increase in rotation speed increased the inner temperature in the mixing chamber which increased the rheology of the polymer with consequent increase in the dispersion of the fiber on the polymer. At high temperature, increased rotation speed was not necessary because the chamber is hot enough to increase the flow of the polymer. This was why at high temperature; the increase in rotation speed did not have appreciable effect on the tensile strength.

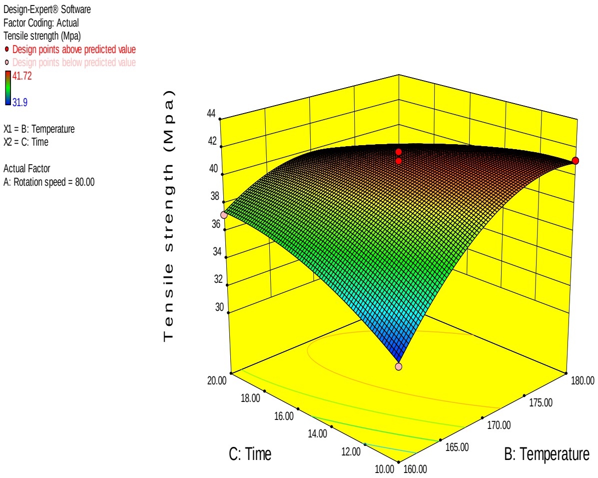
Temperature helped in melting the polymer for easier mixing with the fiber, but when the temperature was so high with prolonged mixing, it led to the decomposition and degradation of the fiber and the polymer.

4.9.4.2. 3D Surface plots of interaction effect of temperature with time

The 3D surface plot for the interaction effect of temperature with time was studied at the midpoint of rotation speed. The 3D surface plots for all the untreated and treated fibers are shown in Figures 4.33 to 4.36. All the plots displayed the same trend. It bulges out showing is a maximization optimization. The shape of the surface indicated that quadratic model explained the process well.


Design-Expert® Software
Factor Coding: Actual
Tensile strength (Mpa)
Design points above predicted value
Design points below predicted value
22.17
12.57
X1 = B: Temperature
X2 = C: Time
Actual Factor
A: Rotation speed = 80.00
Tensile strength (Mpa)
24
22
20
18
16
14
12
10
20.00
18.00
16.00
14.00
12.00
10.00
C: Time
160.00
165.00
170.00
175.00
180.00
B: Temperature

(a)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Factor Coding: Actual
Tensile strength (Mpa)
Design points above predicted value
Design points below predicted value
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31.9
X1 = B: Temperature
X2 = C: Time
Actual Factor
A: Rotation speed = 80.00
Tensile strength (Mpa)
44
42
40
38
36
34
32
30
20.00
18.00
16.00
14.00
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10.00
C: Time
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165.00
170.00
175.00
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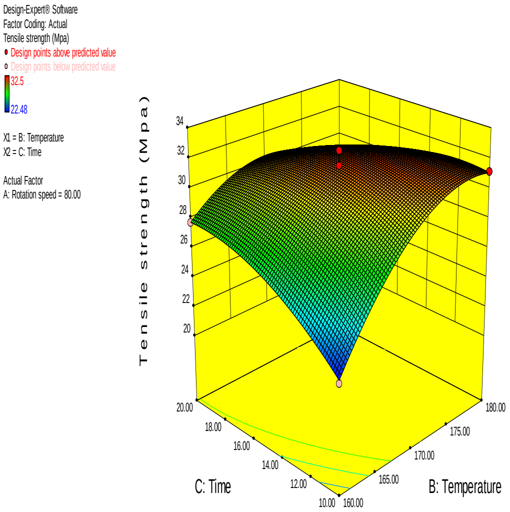
B: Temperature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Actual Factor
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Tensile strength (Mpa)
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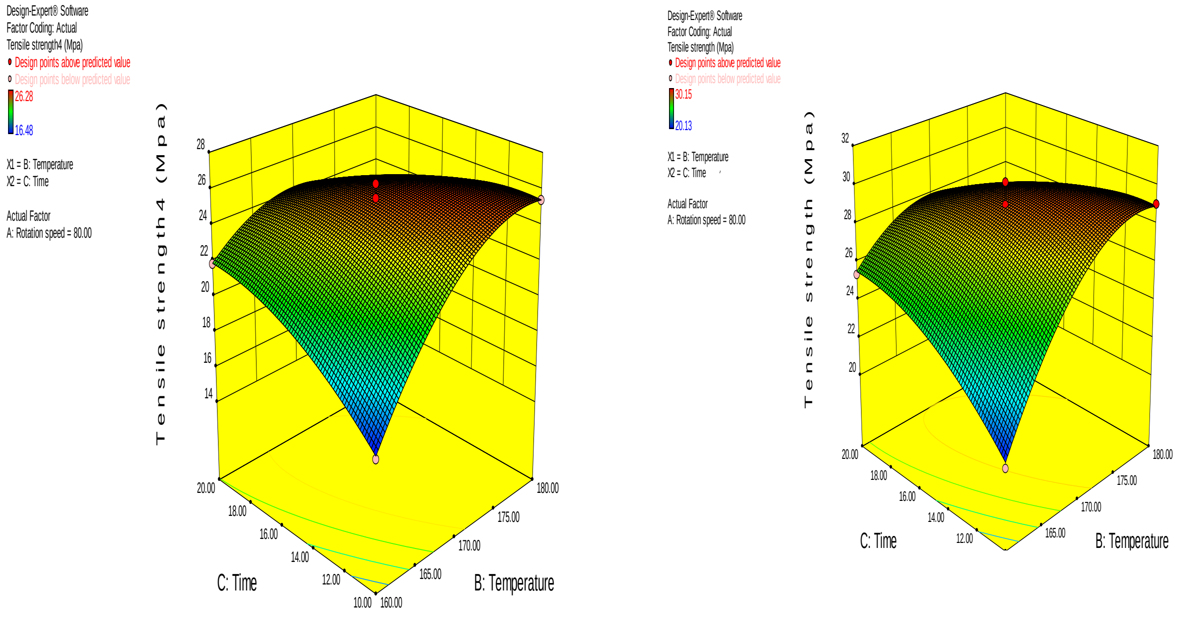
32
30
28
26
24
22
20
20.00
18.00
16.00
14.00
12.00
10.00
C: Time
160.00
165.00
170.00
175.00
180.00
B: Temperature

(c)

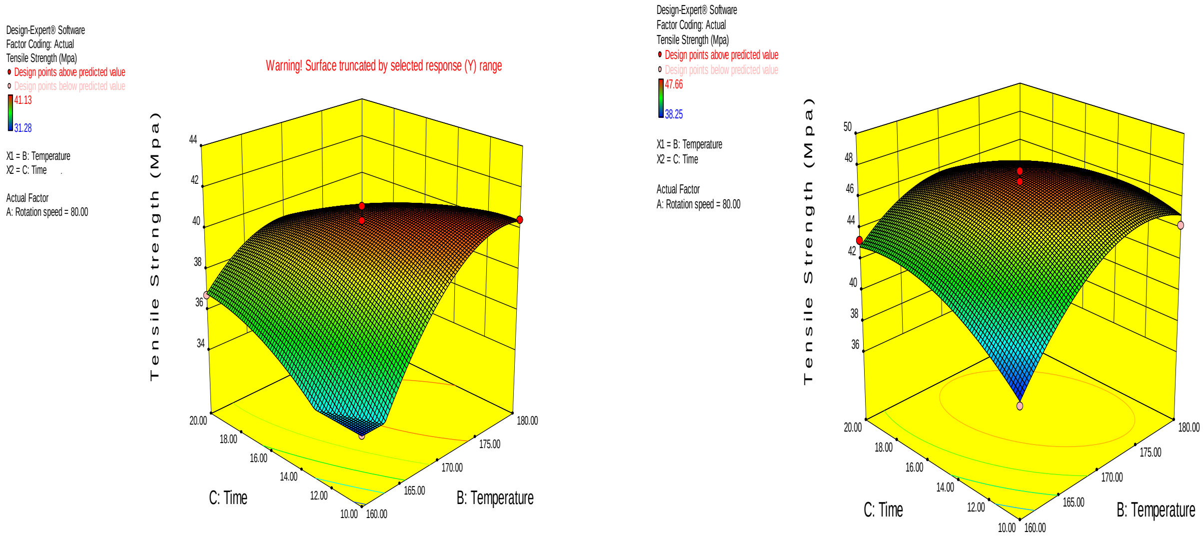
(b)



(e)

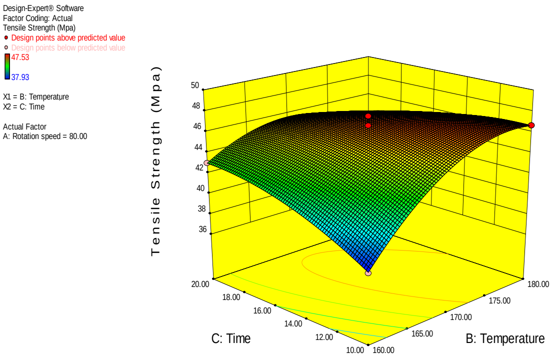
(d)

Fig. 4.33. 3D surface plots of interaction effect of temperature with time on tensile strength of *ampelocissus cavicaulis* composites (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.

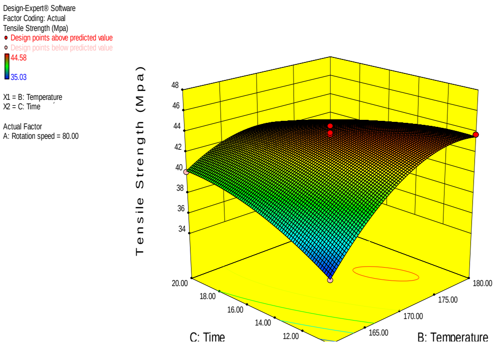


(a)

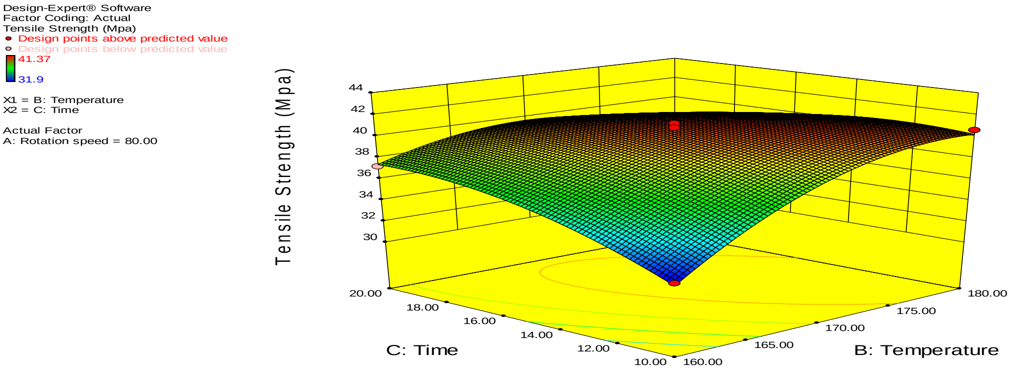
(b)



(c)

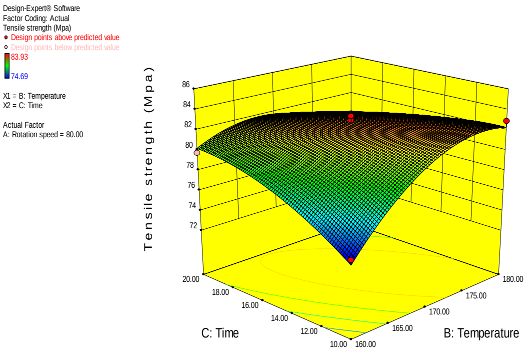


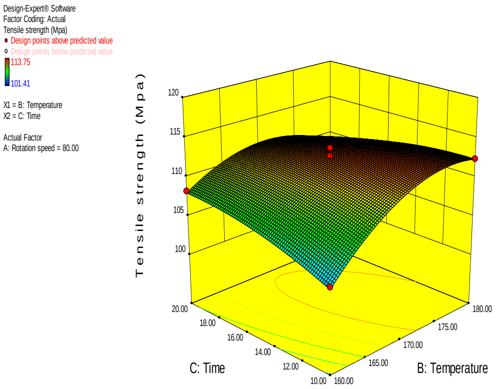
(d)



(e)

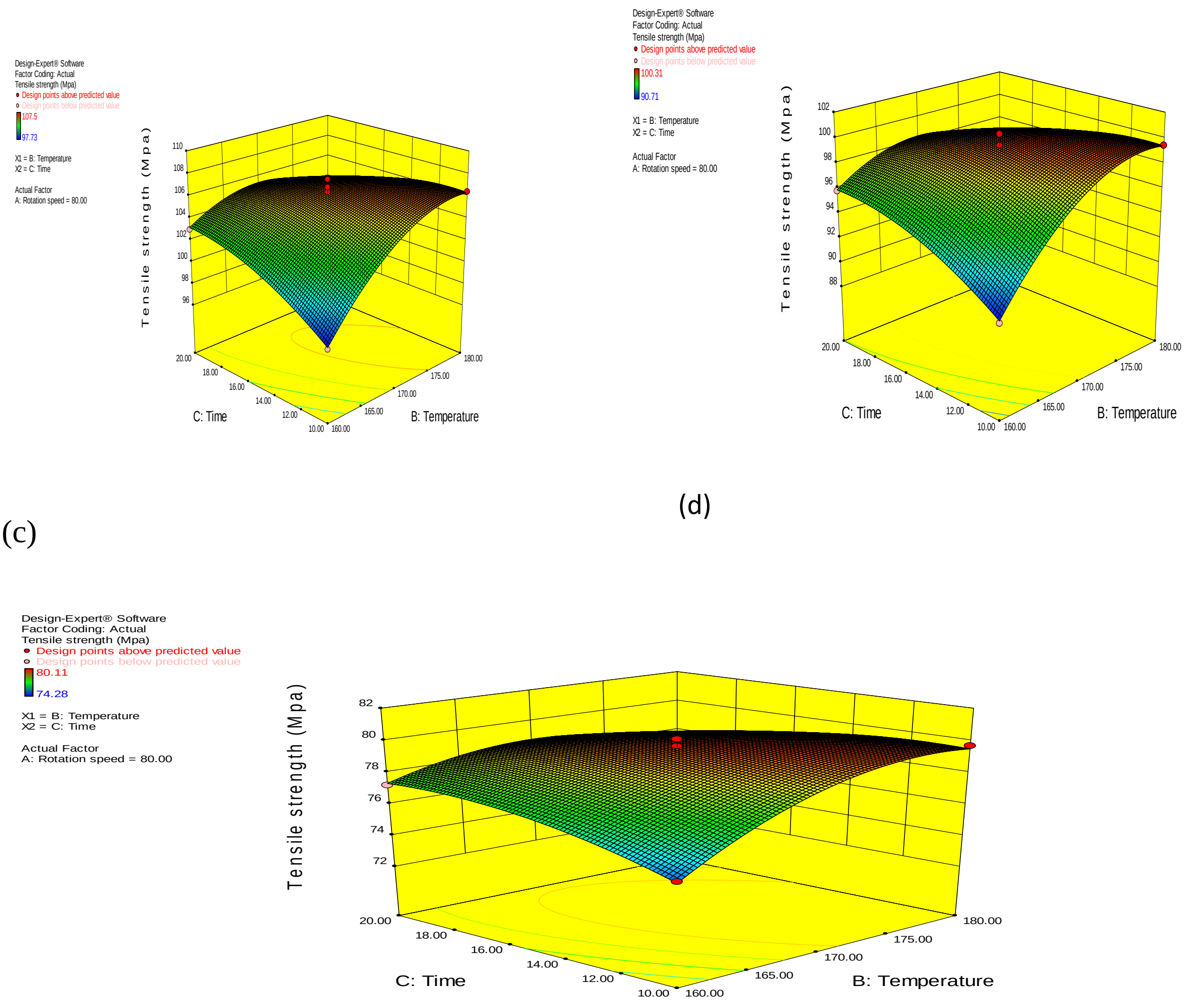
Fig. 4.34. 3D surface plots of interaction effect of temperature with time on tensile strength of *adenia lobata* composites (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.





(b)

(a)

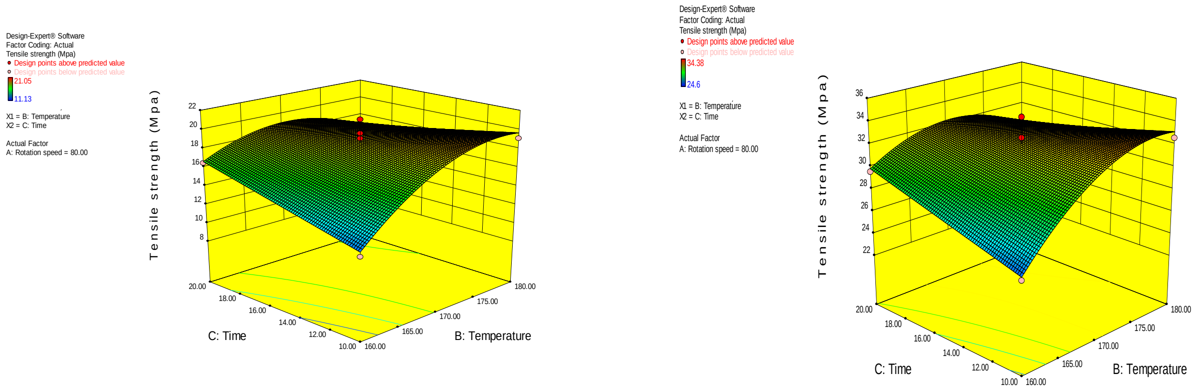


(c)

(d)

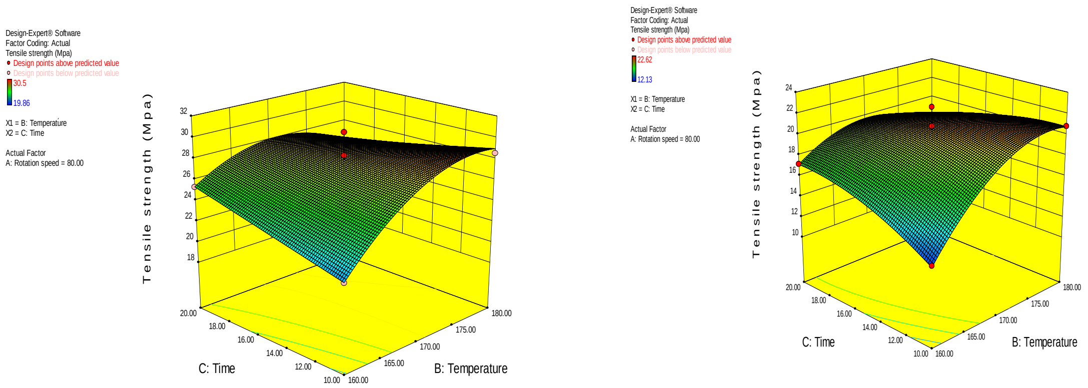
(e)

Fig. 4. 35. 3D surface plots of interaction effect of temperature with time on tensile strength of *morinda morindoides* composites (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.



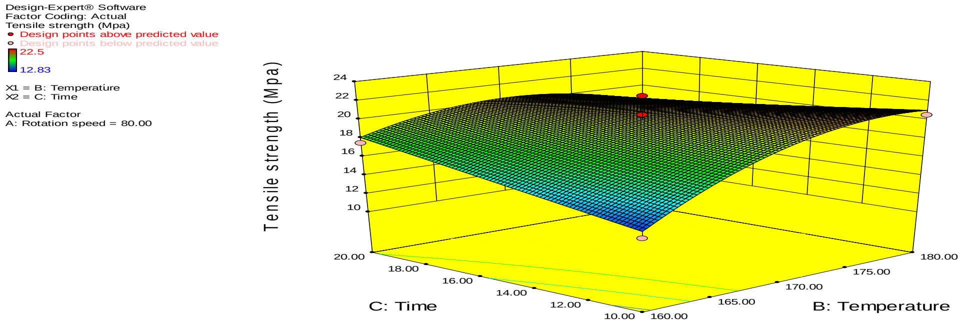
(a)

(b)



(d)

(c)



(e)

Fig. 4.36. 3D surface plots of interaction effect of temperature with time on tensile strength of *ampelocissus leonensis* composites (a) untreated blend (b) treated with NaOH (c) treated with acetic anhydride (d) treated with nitric acid (e) treated with zinc chloride.

The color was hotter at the apex of the hill showing that tensile strength was highest there. Therefore, the optimum conditions lied on the apex. As time was increased with increase in temperature, the tensile strength of the composite increased to a point on the apex, after which further increase in time and temperature resulted to decrease on the tensile strength of the composites. This implies that high dispersion time can only lead to increase on tensile strength when done at low temperature. Higher compounding temperature required lower dispersion time to avoid degradation of the fiber and the polymer.

4.9.5. Optimum conditions for the compounding process

The optimum conditions for the compounding of the polymer with the fibers were selected based on the factor settings with highest desirability. The goal that was met was the maximization of the tensile strength of the resultant composites after compounding. The optimum conditions were shown on Tables 4.19 to 4.22 based on the chemicals used for the pretreatments with the untreated fibers.

Table 4.19. Optimum compounding conditions for *Ampelocissus cavicaulis* fiber

Conditions	Untreated Fiber	NaOH Pretreated	Acetic Anhydride pretreated	Nitric acid pretreated	Zinc chloride pretreated
Rotation speed (rpm)	94.62	94.35	96.39	99.17	97.74
Temperature (^{0}C)	173.94	174.60	172.00	174.92	165.02
Time (minutes	13.65	12.21	13.52	11.20	17.99
Desirability	1.00	1.00	1.00	1.00	1.00
Predicted value (Mpa)	22.5223	42.059	32.504	26.3052	30.3755

Table 4.20. Optimum compounding conditions for *Adenia lobata* fiber.

Conditions	Untreated Fiber	NaOH Pretreated	Acetic Anhydride pretreated	Nitric acid pretreated	Zinc chloride pretreated
Rotation speed (rpm)	99.28	90.91	96.18	95.85	94.48
Temperature (^{0}C)	175.25	173.09	174.87	175.56	173.94
Time (minutes)	11.83	14.57	14.56	13.33	11.03
Desirability	1.0	1.0	1.0	1.0	1.0
Predicted value (Mpa)	41.3409	48.4293	47.6806	44.8531	41.8011

TABLE 4.21. Optimum compounding conditions for *Morinda morindoides* fiber.

Conditions	Untreated Fiber	NaOH Pretreated	Acetic Anhydride pretreated	Nitric acid pretreated	Zinc chloride pretreated
Rotation speed (rpm)	99.36	99.33	98.66	97.61	97.48
Temperature (^{0}C)	165.75	167.00	168.31	172.49	172.52
Time (minutes	13.43	13.17	17.41	13.46	11.67
Desirability	1.0	1.0	1.0	1.0	1.0
Predicted value (Mpa)	84.2002	113.761	107.734	100.806	80.1999

TABLE 4.22. Optimum compounding conditions for *ampelocissus leonensis*

Conditions	Untreated Fiber	NaOH Pretreated	Acetic Anhydride pretreated	Nitric acid pretreated	Zinc chloride pretreated
Rotation speed (rpm)	100.00	100.00	100.00	100.00	100.00
Temperature (^{0}C)	166.00	166.47	166.66	169.47	166.31
Time (minutes)	20.00	20.00	20.00	15.66	20.00
Desirability	0.988	0.965	0.930	0.974	0.978
Predicted value (Mpa)	20.9331	34.0409	29.7553	22.3445	22.2909

4.9.6. Validation of the optimum conditions for the compounding process

It is of paramount importance to validate the optimum conditions obtained from the model equation in order to ascertain its adequacy in predicting maximal response and to obtain its percentage deviation from the predicted optimum condition. The optimum conditions, predicted values, experimental values, and percentage errors as well as the desirability of all the fibers are shown on tables 4.23 to 4.26.

Table 4.23. Validation of the optimum compounding conditions for *Ampelocissus cavicaulis* fiber

Rotation speed (Rpm)	Temperature (°C)	Chemical treatment	Time (Mins)	Tensile Strength (Mpa)		Error (%)
				Predicted value	Experimental values	
94.62	173.94	Untreated	13.62	22.5223	22.475	0.2
94.35	174.60	NaOH	12.21	42.0599	41.984	0.1
96.39	172.0	Acetic anhydride	13.52	32.504	32.487	0.05
99.17	174.92	Nitric acid	11.20	26.3052	26.1158	0.7
97.74	165.02	Zinc chloride	17.99	30.3755	30.284	0.3

Table 4.24. Validation of the optimum compounding conditions for *Adenia lobata* fiber

Chemical treatment	Rotation speed (rpm)	Temperature (⁰C)	Time (minutes)	Tensile strength (Mpa)		
				Predicted Values	Experimented Values	Error (%)
Untreated	99.28	175.25	11.83	41.3409	41.3202	0.05
NaoH	90.91	173.09	14.57	47.4293	47.3818	0.10
Acetic anhydride	96.18	174.87	14.56	47.6806	47.489	0.4
Nitric acid	95.85	175.56	13.33	44.8531	44.8396	0.03
Zinc chloride	94.48	173.94	11.03	41.8011	41.7509	0.12

Table 4.25. Validation of the optimum compounding conditions for *morinda morindoides*

Chemical treatment	Rotation speed (rpm)	Temperature (^{0}C)	Time (minutes)	Tensile strength (Mpa)		
				Predicted Values	Experimented Values	Error (%)
Untreated	99.36	165.75	13.43	84.2002	84.183	0.02
NaoH	99.33	167.00	13.17	113.761	113.747	0.03
Acetic anhydride	98.66	168.31	17.41	107.73	107.396	0.31
Nitric acid	97.61	172.49	13.49	100.806	100.766	0.04
Zinc chloride	97.48	172.52	11.67	80.1999	80.184	0.02

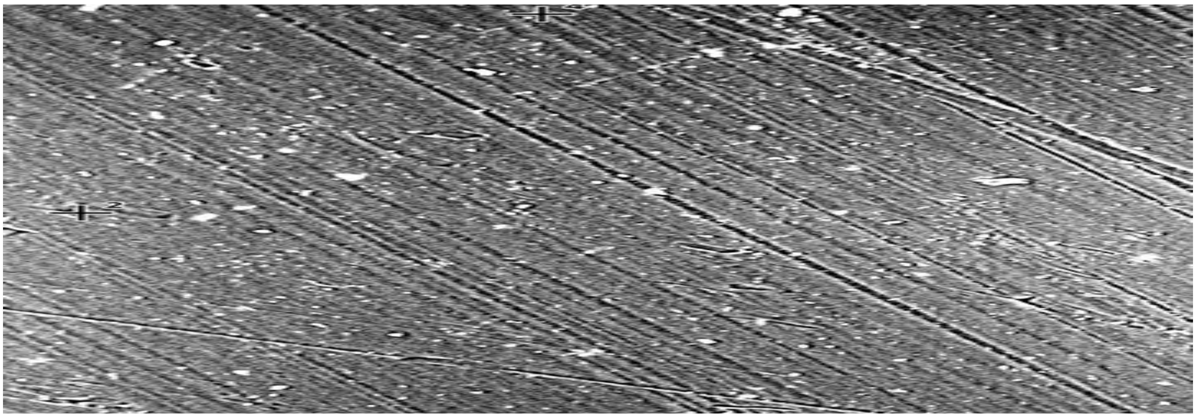
Table 4.26. Validation of optimum compounding conditions for *Ampelocissus leonensis* fiber.

Chemical treatment	Rotation speed (rpm)	Temperature (^{0}C)	Time (minutes)	Tensile strength (Mpa)		
				Predicted	Experimented	Error (%)
Untreated	100.00	166.00	20.00	20.9331	20.9120	0.1
NaOH	100.00	166.47	20.00	34.0409	33.8707	0.50
Acetic anhydride	100.00	166.66	20.00	29.7553	29.7613	0.02
Nitric acid	100.00	169.47	15.66	22.3445	22.362	0.08
Zinc chloride	100.00	166.31	20.00	22.2909	22.3804	0.4

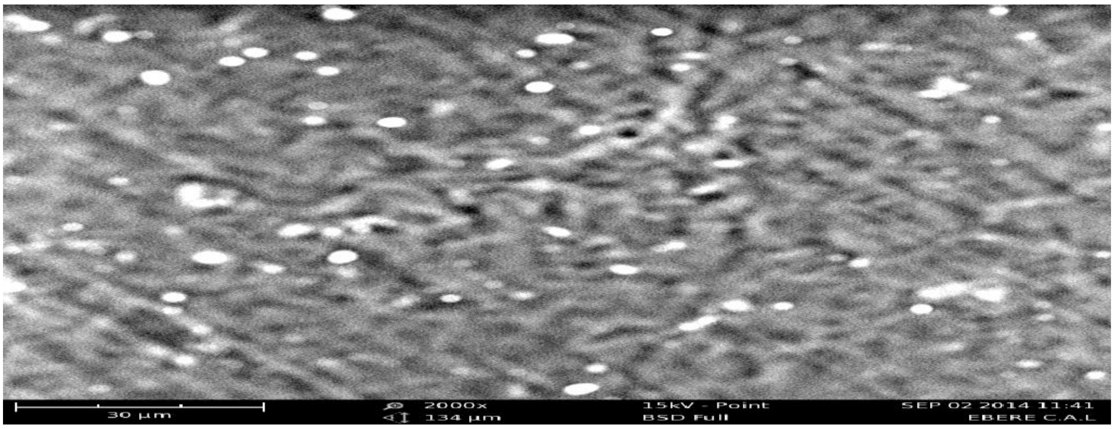
The error values obtained from the experimental values obtained from the lab using the optimum conditions were less than one percent. Therefore, it can be deduced that the optimum conditions obtained were good enough to be used in maximizing the tensile strength of the composite because of little errors calculated.

4.9.7. Scanning Electron Microscopy (SEM) Analyses of compounded and uncompounded composites

Figure 4.37 shows micrographs of the surfaces of composite samples for both compounded and uncompounded composites. The morphological result in Fig 4.37a shows that there was proper and intimate mixing of the fiber with the polymer. Composites thus synthesized, revealed that the fiber was well wetted by the polymer and the surface was thick without voids. The micrographs of uncompounded composite (Fig.4.37b) revealed that there was no intimate mixing between the fiber and the polymer. This was evidenced by the presence of white dots which indicated the polymer separated from the fiber. This obviously led to lower tensile strength of the composite.



(a)



(b)

Fig 4.37 SEM analysis of the composite (a) compounded (b) uncompounded

4.10. Effect of compression molding conditions on the tensile strength of the composites

The controlling parameters in compression molding method to develop superior and desired properties of the composites are fiber loading ratio, fiber length, temperature, time and pressure. The effects of these factors were investigated on the tensile strength of the untreated and treated fiber reinforced composites.

4.10.1. Effect of fiber loading on the tensile strength of the composites

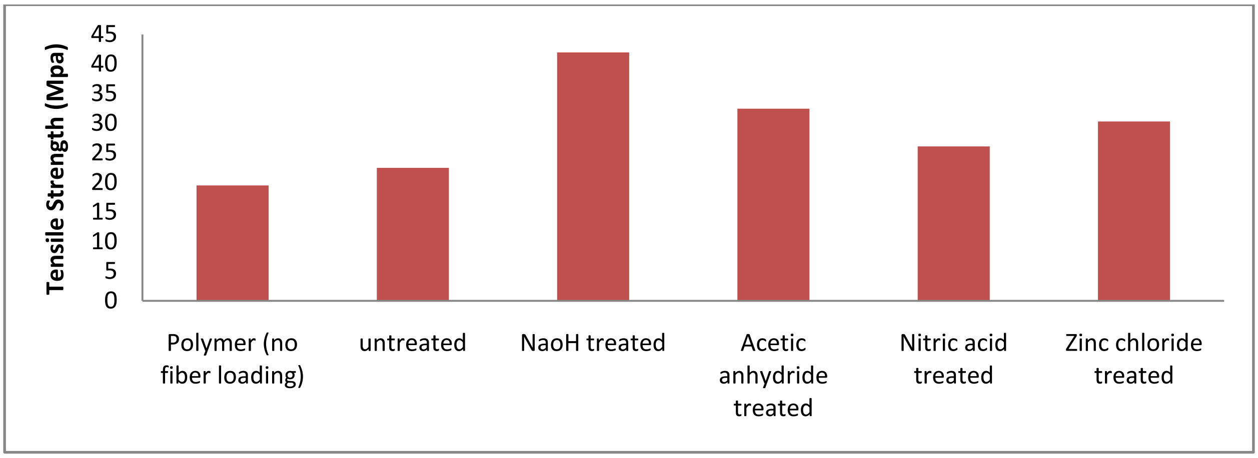
The properties of fiber reinforced polymer composites depend on the percentage of fiber volume/loading on the matrix. Tensile strength measurements are among the most important indicators of strength in a material and are most widely specified property. Tensile test is a measurement of the property of a material to withstand forces that tend to pull it apart and to determine to what extent the material stretches before breaking. Effect of fiber loading was studied at temperature of 170^0C, fiber length of 3mm, compression time of 10mins and holding pressure of 30 bars. The fiber loading was studied at 5wt%, 10wt%, 20wt%, 30wt%, 40wt% and 50wt% with 0wt% as control.

Studies about composites show that there is a relationship between fiber loading and tensile strength and that the tensile strength of the composite always exceeds that of the polymer (Nagaraja and Rekha, 2013). This was confirmed from Fig. 4.38 on all the fibers, that the tensile strength of the composites was greater than the tensile strength of the polymer compressed without the fiber. It means that the fiber has proven its function by increasing the tensile strength of the composite compared to the polymer.

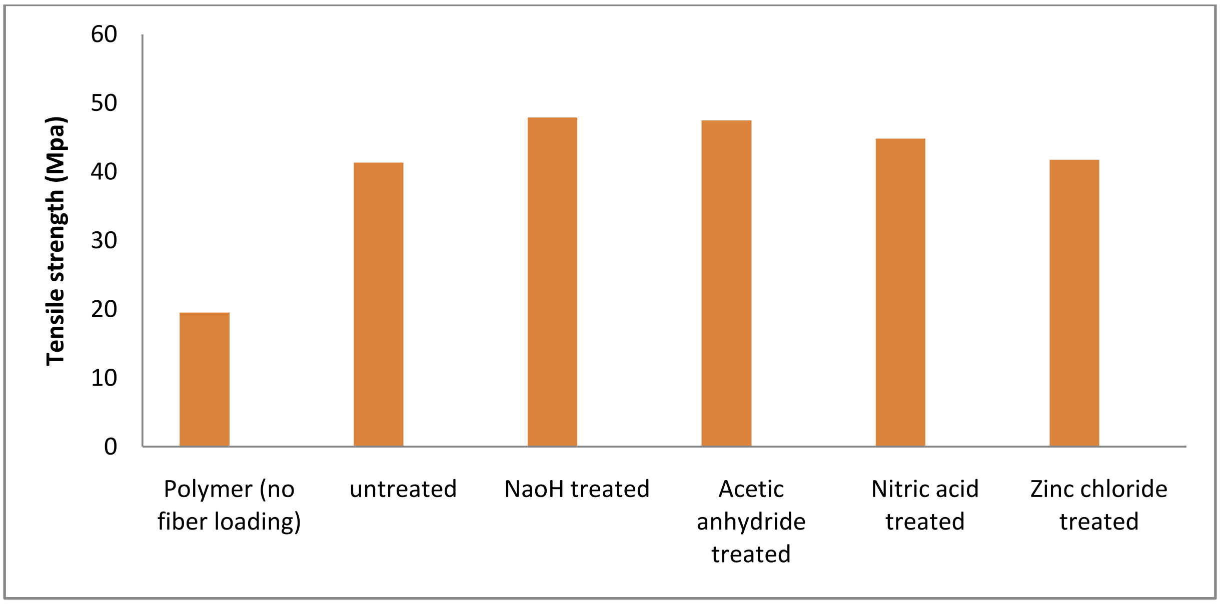
Fig. 4.38 shows that addition of fiber increased the tensile strength of the composite to a certain level, depending on the type of fiber used as reinforcement and the chemical used for the pretreatment beyond which there was decrease in tensile strength. It can be observed from Figure 4.39, that 30wt% fiber content showed the highest tensile strength for untreated *ampleocissus cavicaulis* and 20wt% for *adenia lobata* fiber treated with all the chemicals. For *morinda morindoides* fiber, 10wt% fiber content represents the optimal fiber content for both treated and untreated fiber. 40wt% fiber content was the optimal fiber content for Ampelocissus leonensis composite. Based on the results, there exists, similar trend for all the fibers where the tensile strength of the composites showed steep decline beyond their optimal values.

As the fiber was increased beyond the optimum content, the fibers may not sufficiently interact with the polymer matrix and the interfacial adhesion that is responsible for the composite stiffness and strength would be much lower as the fibers agglomerate (Medium et al., 2013). As a result, it led to decrease in the tensile strength. Improper adhesion hinders the considerable increment of tensile strength (Beckermann et al., 2004). Thus as fiber percentage increases, gathering of fibers takes place instead of dispersion and melted HDPE cannot wet them properly due to non entrance of melt through the adjacent two fibers. Since no adhesion is present between the fibers and fibers are also not bounded with matrix, failure occurs before attaining the theoretical strength of composite. Thus higher fiber content was limited by the incompatibility issue unless coupling agents is used (Wollerdorfer and Bader, 1998). Composites of higher fiber content usually display more void formation (Vaxman et al., 2004). Higher void content is responsible for the lower fatigue resistance, greater affinity to water diffusion and increase variation (scatter) in mechanical properties (Kenneth, 1992; Vaxman, 2004).

Figure 4.39 equally displayed the effect of different chemical treatments on the tensile strength of the fiber. As can be seen from the results, that the tensile strength of the composite with treated reinforcement were higher than the untreated ones. The increase was more significant in the case of alkali treatment. During treatments, waxes, hemicelluloses and part of the lignin present on the fiber surface which acted as cement covering the cellulose were removed. The removal of these compounds enhanced the surface roughness, which allows mechanical interlocking. The formation of strong covalent bonds between the polymer and the hydroxyl groups of cellulose lead to significant improvement on the tensile strength.



(a)



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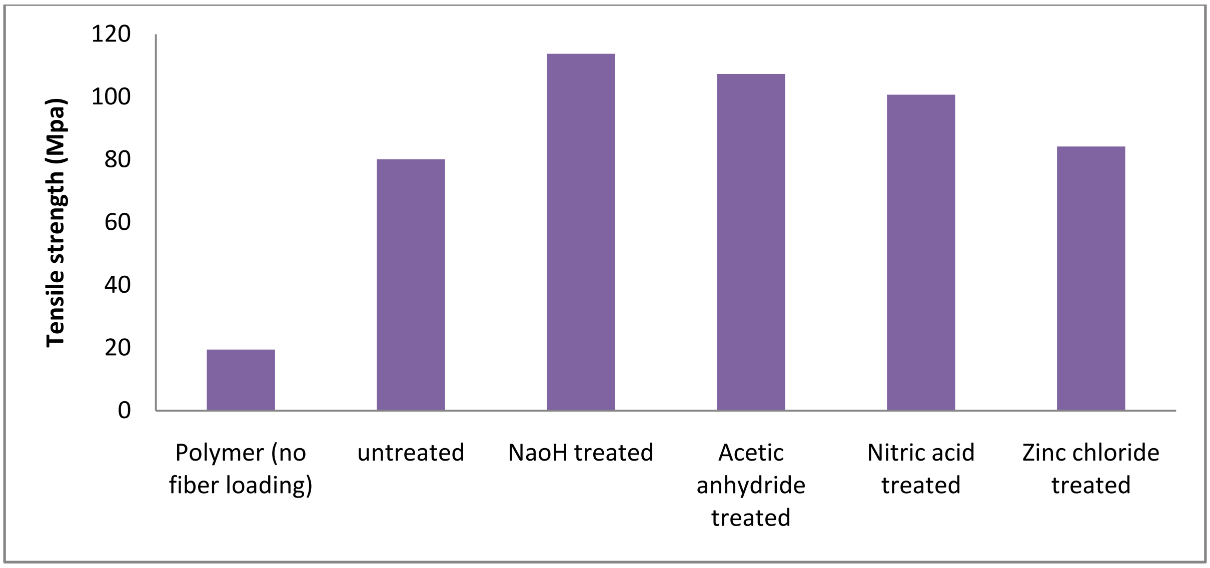

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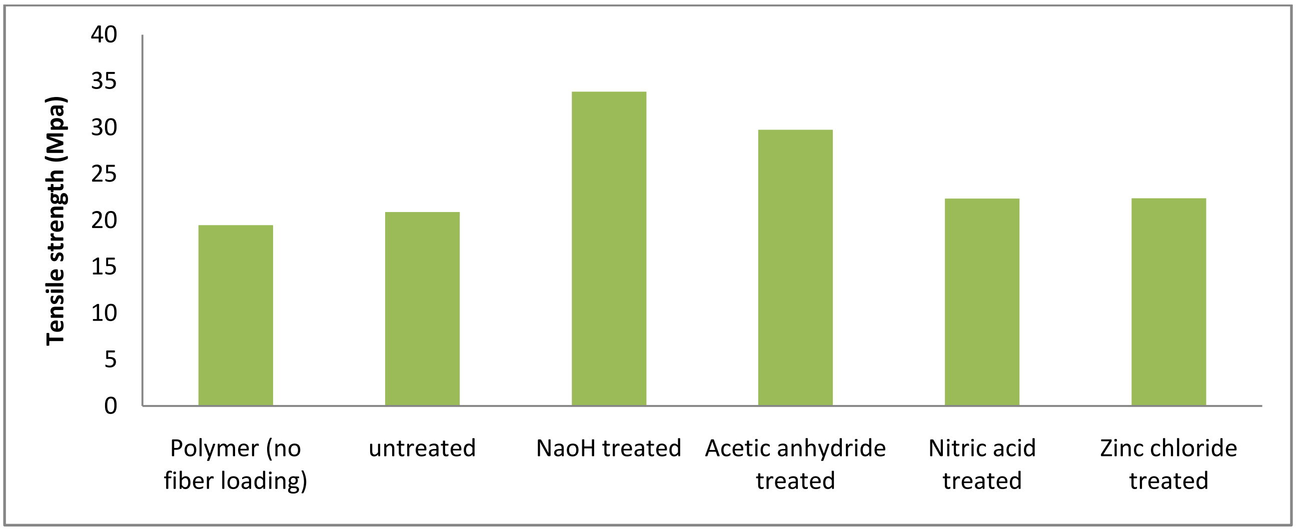
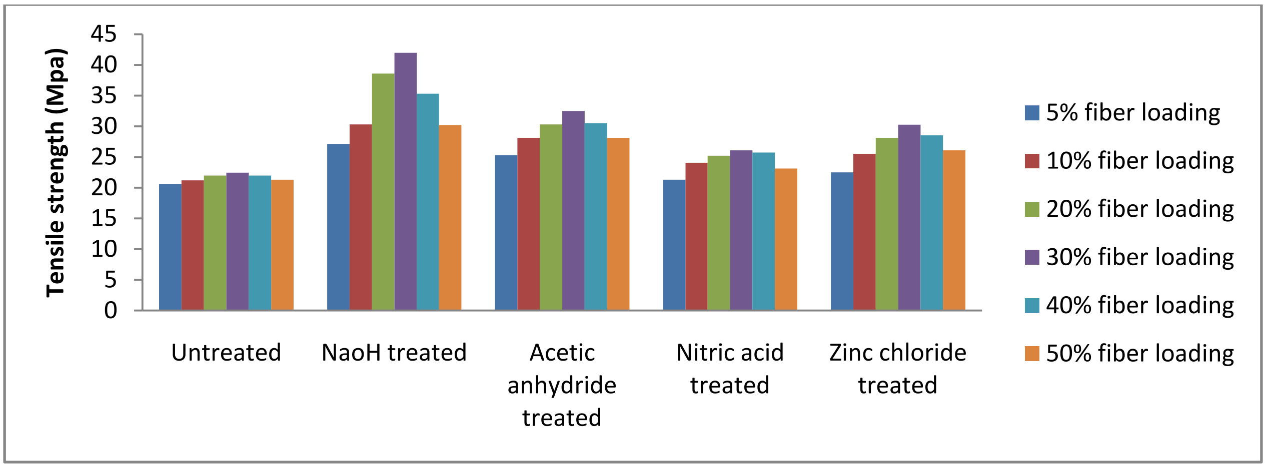


(d)

Fig. 4.38. Effect of fiber as reinforcement on composite (a) Ampelocissus cavicaulis fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber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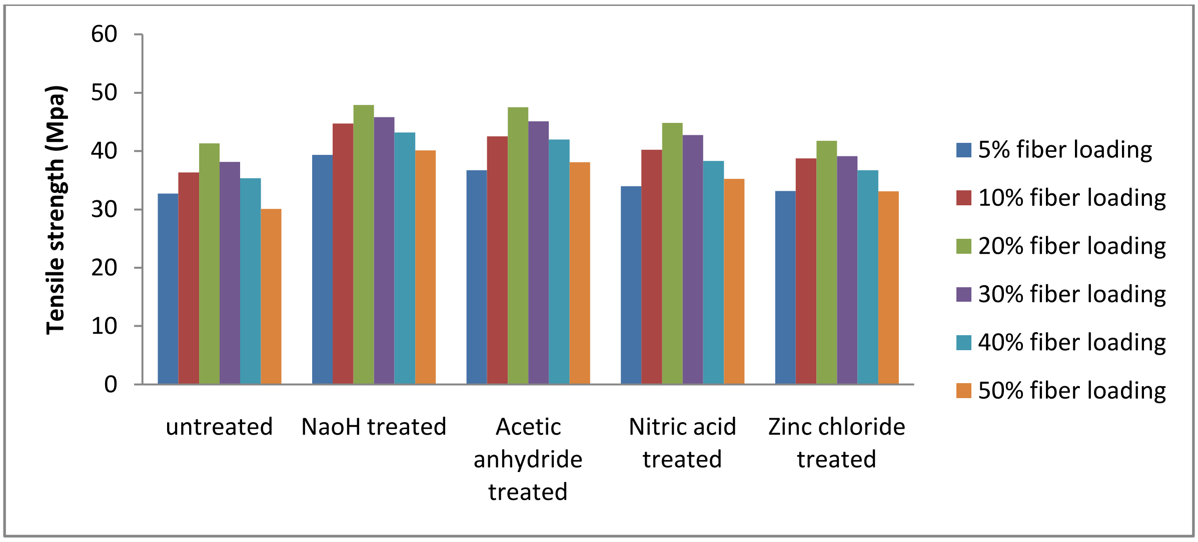
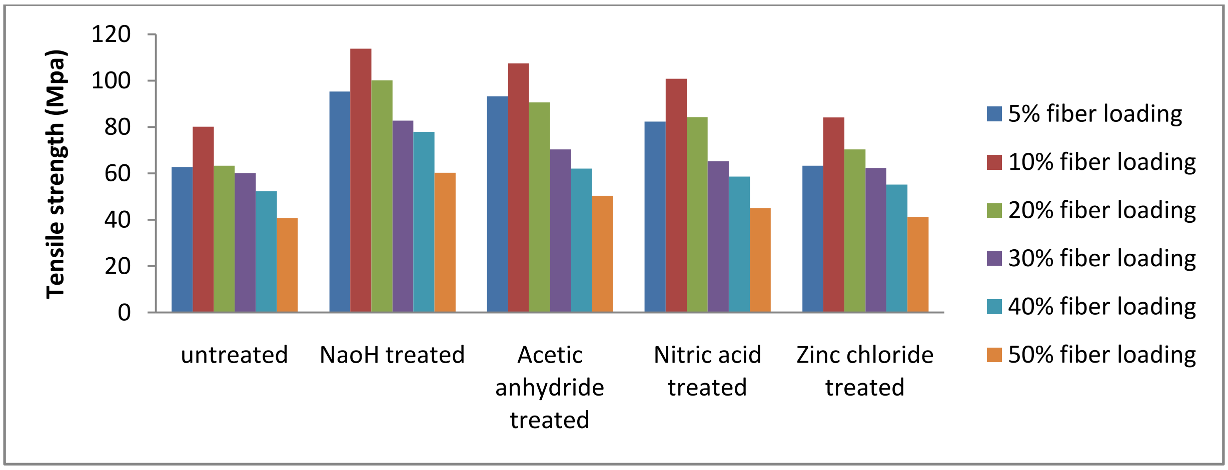
40
35
30
25
20
15
10
5
0
Untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5% fiber loading
10% fiber loading
20% fiber loading
30% fiber loading
40% fiber loading
50% fiber loading

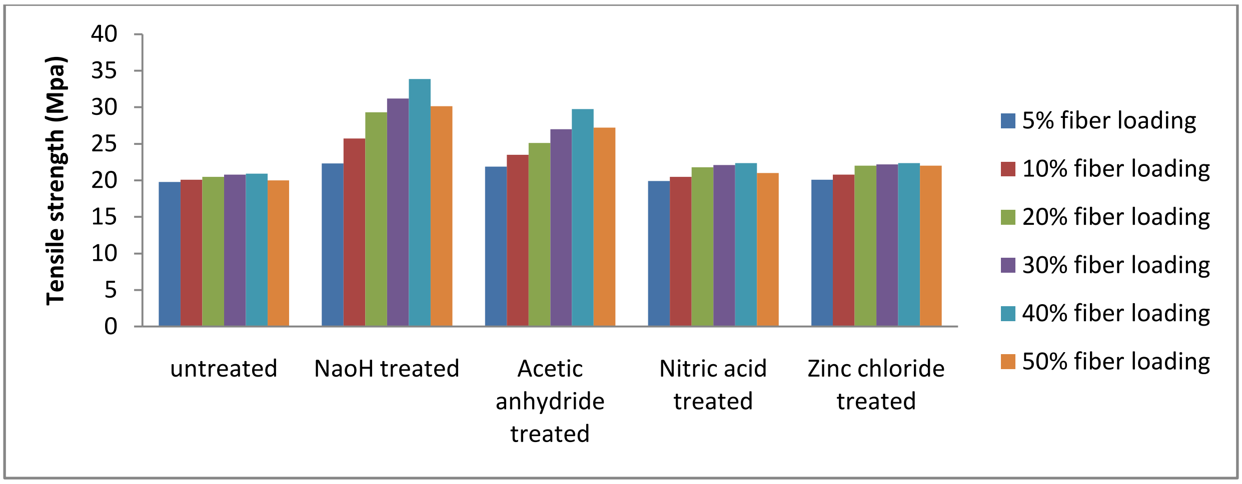
(a)


Tensile strength (Mpa)
60
50
40
30
20
10
0
untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5% fiber loading
10% fiber loading
20% fiber loading
30% fiber loading
40% fiber loading
50% fiber loading

(b)


Tensile strength (Mpa)
120
100
80
60
40
20
0
untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5% fiber loading
10% fiber loading
20% fiber loading
30% fiber loading
40% fiber loading
50% fiber loading

(c)



(d)

Fig. 4.39. Effect of chemical treatments and various fiber loading on the tensile strength of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

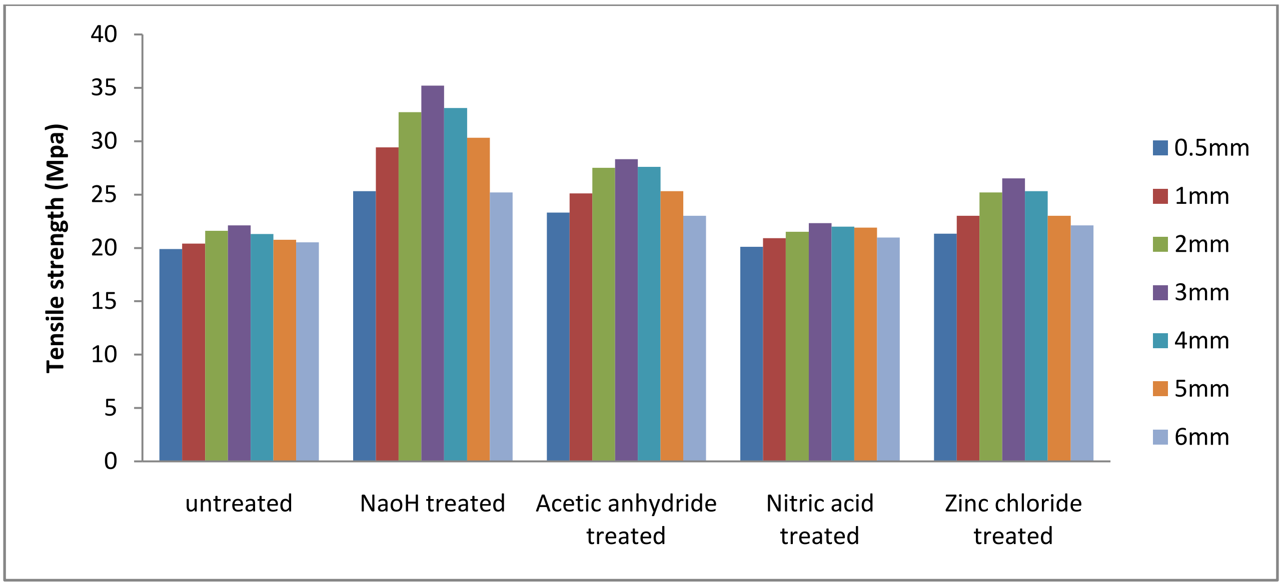
4.10.2. Effect of Fiber Length on the Tensile Strength of the Composites

The treated and untreated fibers were cut to the desired lengths of 0.5mm, 1mm, 2mm, 3mm, 4mm, 5mm, and 6mm. The effect was studied at 30wt% fiber loading, pressure of 30bars, temperature of 170^{0}C and compression time of 10mins in a random oriented manner. The properties of the fiber reinforced polymer composites depend on the fiber length, when load is applied to the matrix, stress transfer occurs by shear at both the interface along the fiber length and at the ends of the fiber (Kabir, et al., 2011). The extent of load transmitted to the fiber is a function of critical fiber length (aspect ratio), direction and orientation of fiber relative to each other. By controlling factors such as fiber length, the dispersion and orientation of fibers, considerable improvements in composites properties can be accomplished (Fakirov, 2007; Leonard and Ansell, 1999; Joseph, 2011). Fiber length has profound impact on the tensile strength of the composites, besides holding the fiber together; the matrix has the important function of transferring applied load to the fiber. The efficiency of a fiber reinforced composite depends on the fiber matrix interface and the ability to transfer stress from the matrix to the fiber (Karani et al., 1997).

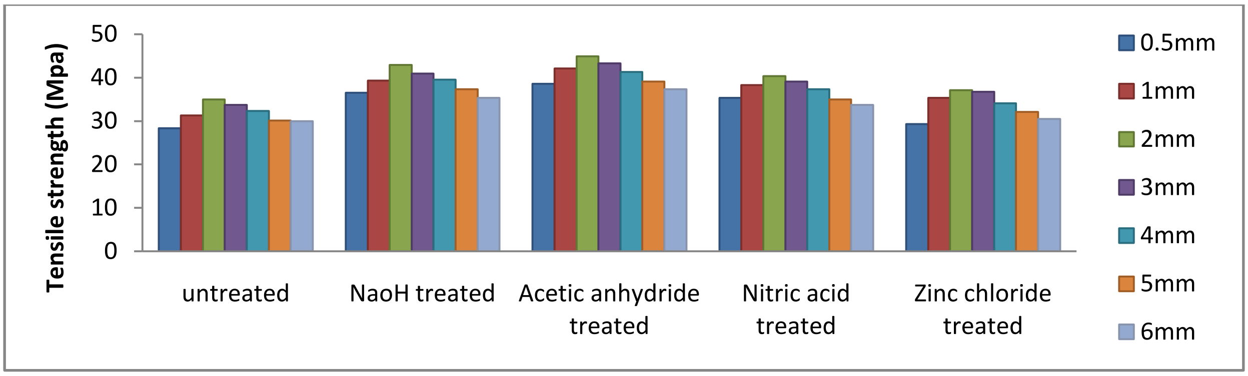
From the Fig. 4.40, it can be seen that the tensile strength of the composites increased at 0.5mm to 3mm fiber length loading beyond which there was a decrease on the tensile strength for the untreated and treated *ampelocissus cavicalus* and *ampelocissus leonesis* fibers reinforced composites.

The tensile strength increased to 2mm and started decreasing for treated and untreated *adenia lobata* and *morinda morindoides* fibers reinforced composites.

Using small fiber sizes below the optimum sizes, tensile strength was low due to the fact that there is now a short fiber length for stress transfer by shear to occur. As proper length is not available for stress distribution, failure thus occurs easily. On the other hand for the composites of longer fiber sizes above the optimum sizes, tensile strength decreased. The probable reason is that a long fiber may not become compatible with the matrix properly. Thus improper bonding occurs between the fiber and the matrix (Rashed et al., 2006). Moreover, fibers may be folded and there is no bounding between the folded and unfolded portion of fiber which resulted in a lower strength. Fiber entanglement may also contribute to reduced tensile strength (Joseph et al., 2002). For *adenia Lobata* and *morinda morindoides* that had higher fiber length as the optimum, in this case (3mm), it was believed that probably the 3mm length of fiber was still not enough to create fiber entanglement or folding inside the matrix. It can be equally observed from the Figure 4.40 that the tensile strengths of the treated fiber reinforced composites were greater than the untreated ones. The same trend was observed on all the fibers. This was due to improved fiber wetting, dispersion and fiber-matrix adhesion resulted from the chemical treatments.



(a)



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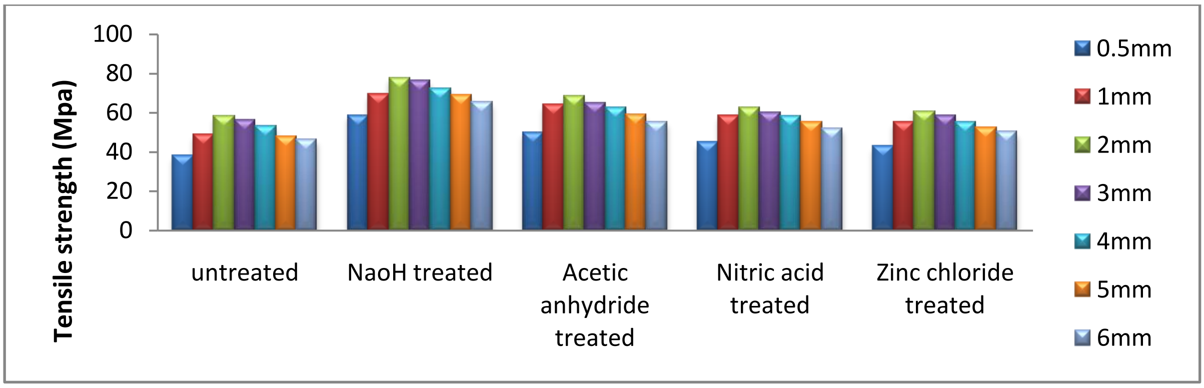

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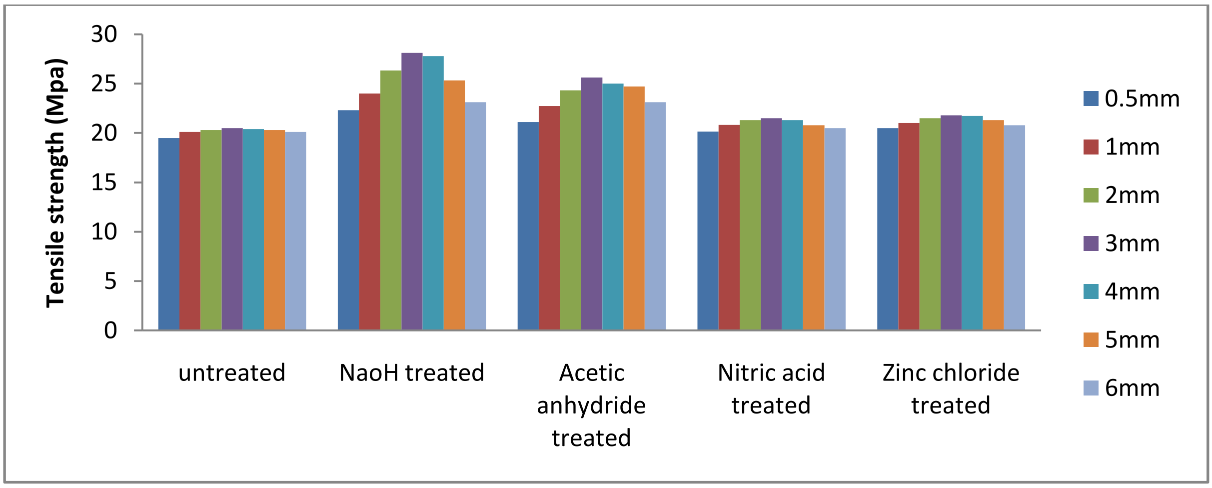


(d)

Fig.4.40. Effect of chemical treatments at various fiber lengths on the tensile strength of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

4.10.3. Effect of molding temperature on the tensile strength of composites

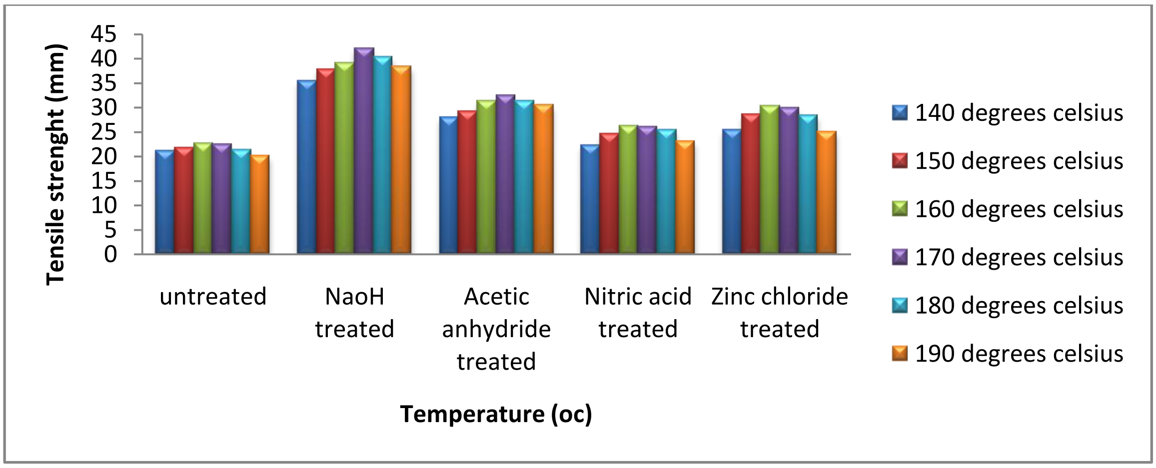
Temperature is an important property that affects compression molding of composites. The temperature effects were studied at fiber length of 3mm, fiber loading of 30wt% and different time intervals of 5mins and 10mins. There are some reasons behind choosing these temperatures and times. The reasons are the degradation temperature of fiber and polymer. The melting and processing temperature of HDPE and degradation temperatures of the fibers were considered. The melting temperature for HDPE is 130 to 137^{o}C (Li et al., 2005) and the recommended processing temperature for HDPE is 177 to 260^{o}C (Rosata et al., 2000). Decomposition temperature of HDPE is relatively high which is 340 to 440^{o}C (Li et. al., 2005). However, though the processing temperature of the fibers were not determined but from literature survey, temperature more than 200^{o}C will be critical for natural fiber as thermal degradation might start affecting mechanical properties of the natural fiber (Bogoeva –Gaceva et. al., 200; El – Shekeil et. al., 2013). The higher temperature will result to fiber degradation and weight loss. Therefore, the compression temperature was limited to 190^{o}C to avoid fiber degradation, although higher temperature can be used at short period of time.

It can be seen from Fig.4.41 and Fig.4.42 that for *ampelocissus cavicaulis* fiber, 160^{o}C was the best temperature for the treated and the untreated except fiber treated with sodium hydroxide and acetic anhydride that had 170^{o}C as their optimum at 10mins. But at lower time of 5mins, the treated and untreated composites had 170^{o}C as their optimum except sodium hydroxide and acetic anhydride treated reinforced composite that had 180^{o}C as their optimum. (Fig 4.42)

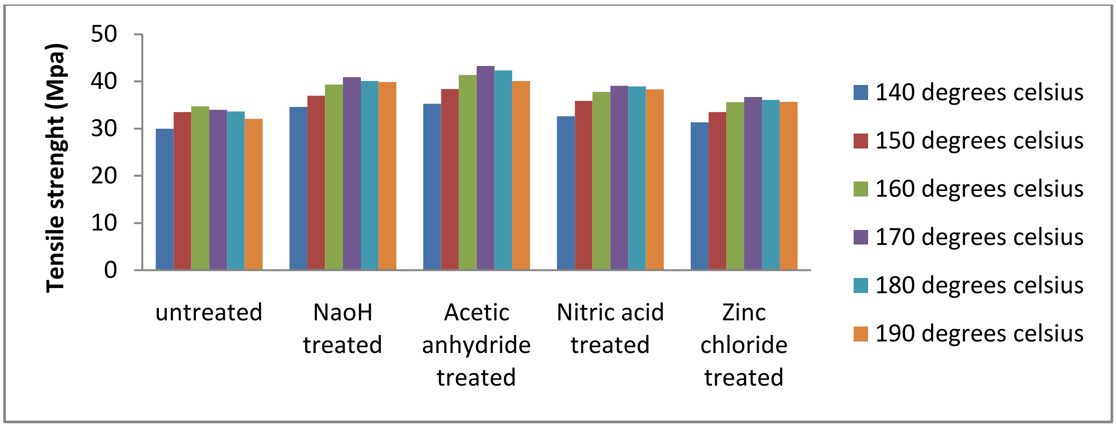
For *adenia lobata* fiber, 160^{o}C was the optimum for the untreated fiber reinforced composite while 170^{o}C was the optimum for all the treated fiber reinforced composites at 10mins. At 5mins, the optimum for the untreated fiber reinforced composite increased to 170^{o}C, while that of all the treated fiber reinforced composites increased to 180^{o}C. For *morinda morindoides,* the optimum compression temperature was 180^{o}C for all the treated and treated fiber reinforced composites at 5mins. But at 10mins, the temperature reduced to 170^{o}C. For *Ampelocissus leonensis* fiber, the temperature was optimum at 150^{o}C at 10 mins for the untreated and fiber treated with nitric acid and zinc chloride. But fiber reinforced composites treated with sodium hydroxide and acetic anhydride had 160°C as their optimum temperature at 10mins. At 5mins holding time, the optimum for all the treated and untreated fiber reinforced composite were 160°C.

The various optimum temperatures obtained for all the fiber using the same matrix were as a result of differences on the physical and chemical compositions of the fiber which resulted to different degradation temperature for all of them. It can equally be seen from the plots that the compression temperatures for the treated fiber reinforced composites were higher than the untreated. This can be attributed to the effect of chemicals used for the treatments which made the fiber more stiff than the untreated thereby increasing their degradation temperatures.

All composites showed, regardless of the type of fiber employed, a comparable performance with maximum tensile strength in the medium temperature range. For lower and higher processing temperature, a more or less distinctive decline can be observed. This can be ascribed to the interrelation of two effects opposing each other. An increasing processing temperature led to lower viscosity of the binder component and thus to improved flow capabilities. This resulted to an improved fiber embedment during compression and therefore higher stability of the composites. However, at the same time the increase of temperature led to progressive decomposition of the reinforcing fiber, resulting in a decrease of fiber strength. Furthermore, a modification of the fiber surface caused by thermal decomposition, leading to a deterioration of adhesion between fiber and matrix, can also be assumed (Mueller, 2004).



(a)



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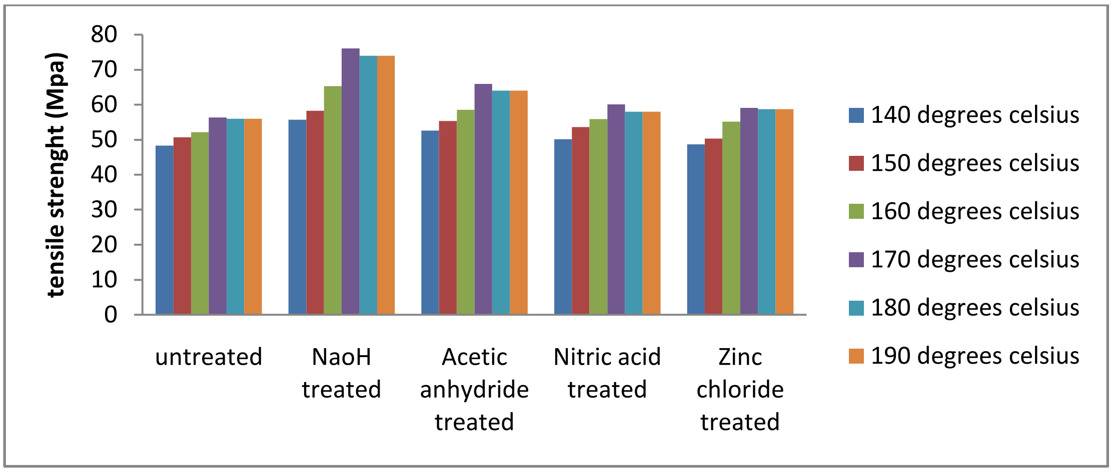

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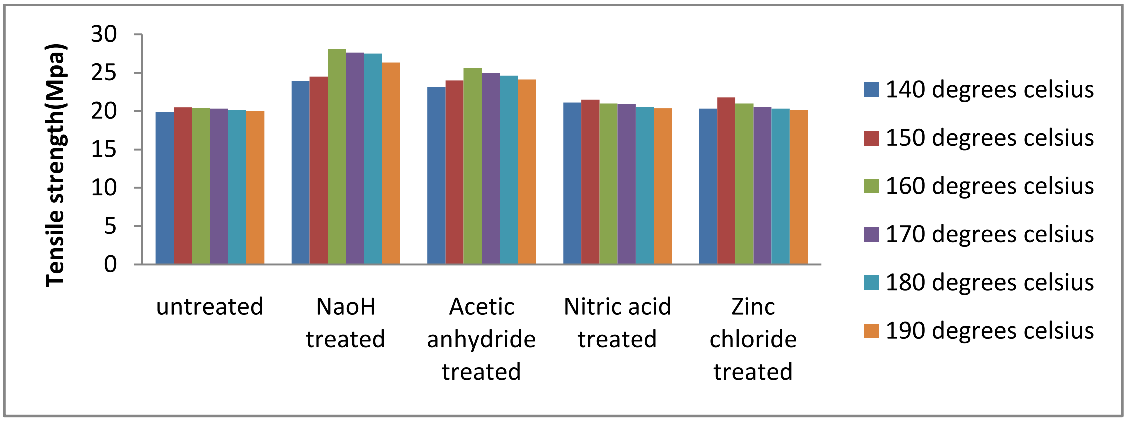
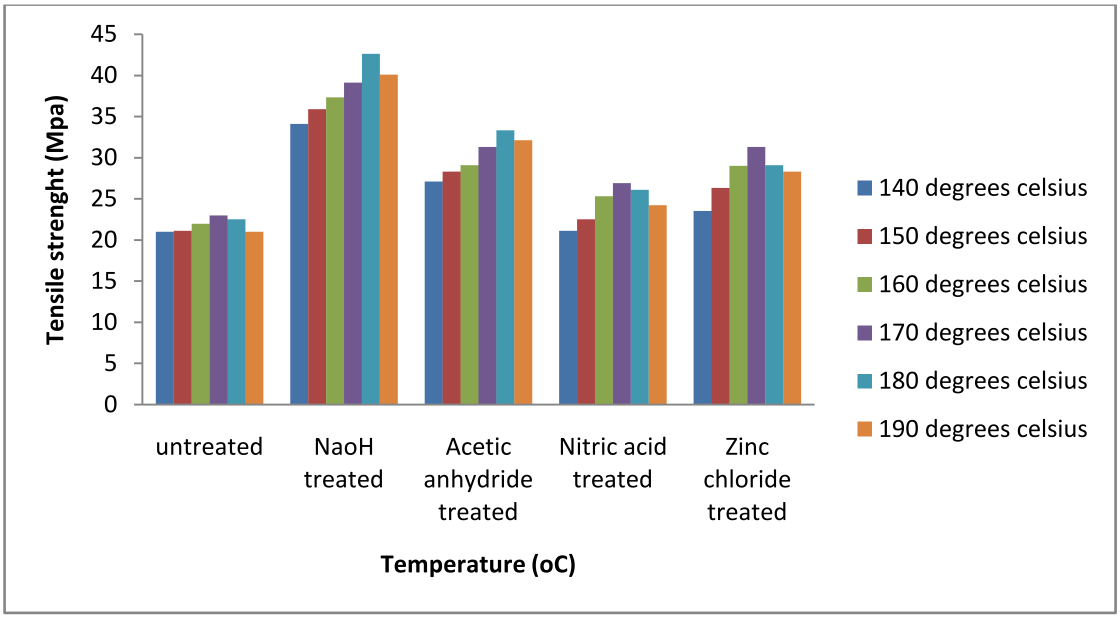
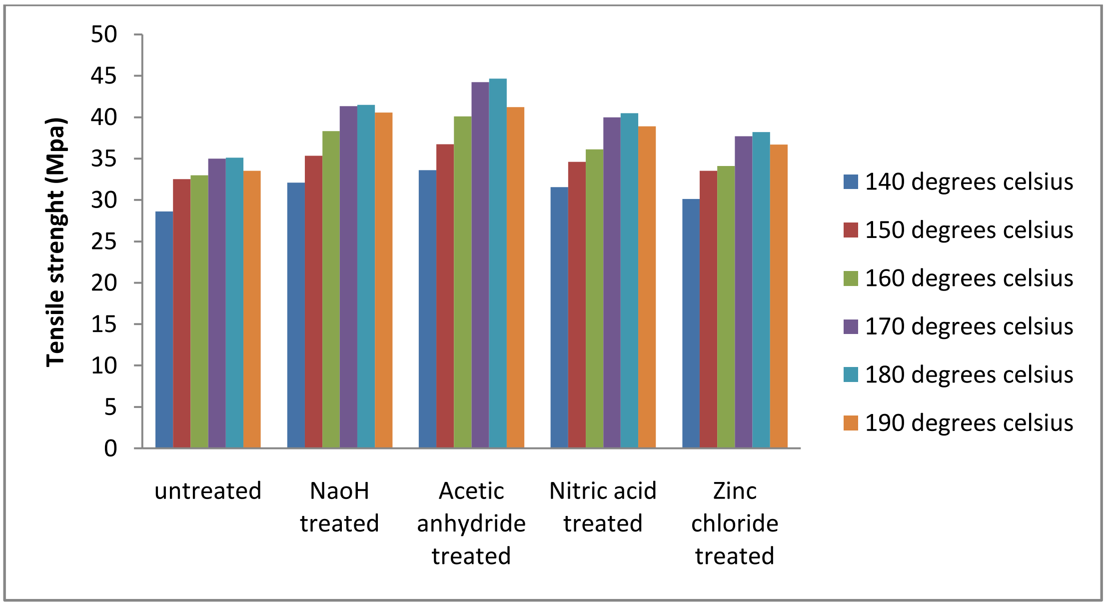


(d)

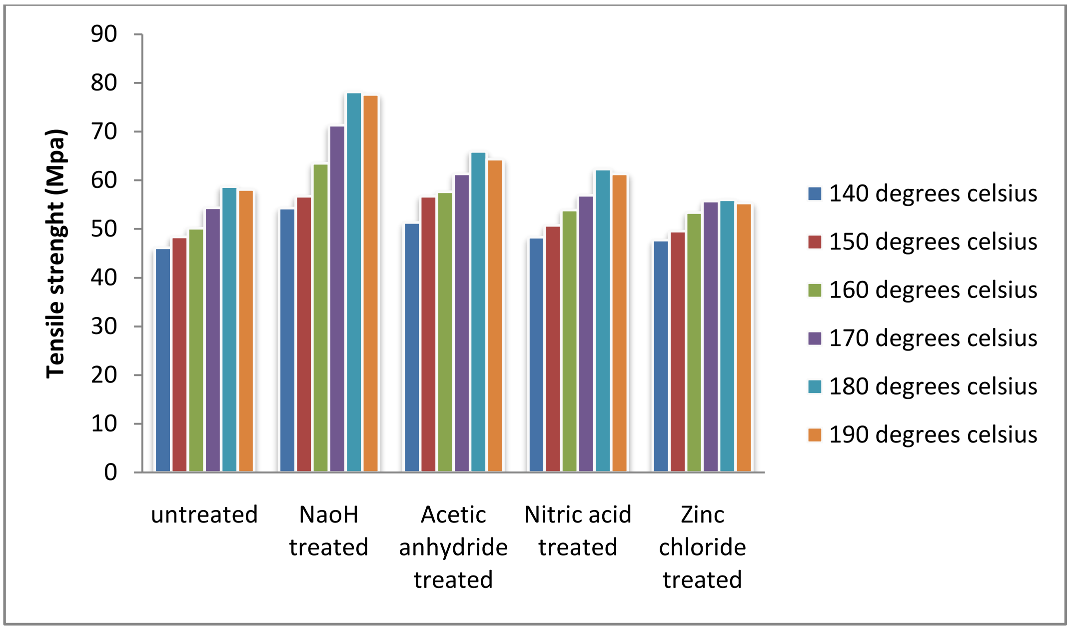
Fig. 4.41. Effect of Temperature on the tensile strength of composites at 10mins (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata fiber* (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.


Tensile strenght (Mpa)
45
40
35
30
25
20
15
10
5
0
untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
Temperature (oC)
140 degrees celsius
150 degrees celsius
160 degrees celsius
170 degrees celsius
180 degrees celsius
190 degrees celsius

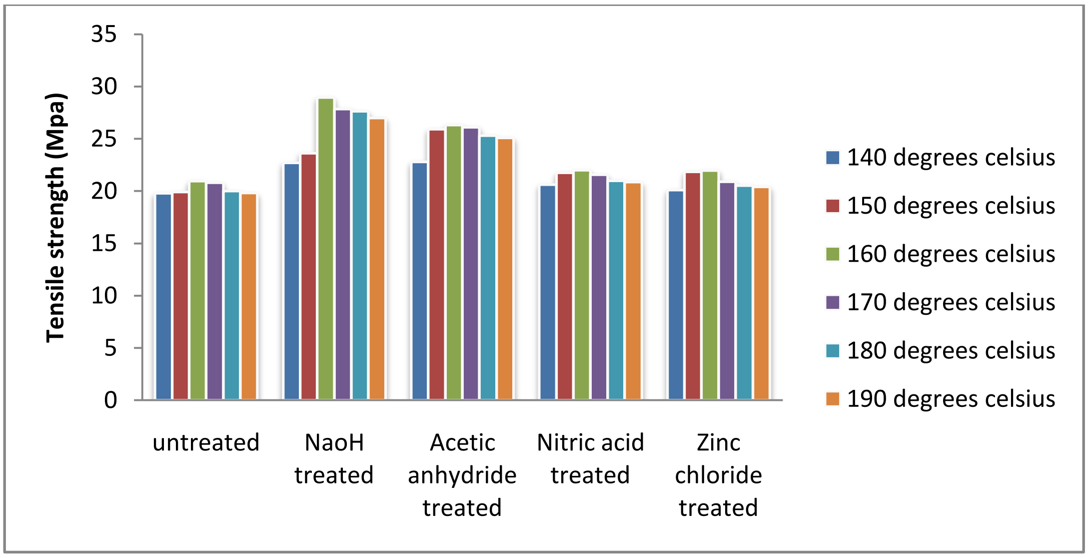
(a)


Tensile strenght (Mpa)
50
45
40
35
30
25
20
15
10
5
0
untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
140 degrees celsius
150 degrees celsius
160 degrees celsius
170 degrees celsius
180 degrees celsius
190 degrees celsius

(b)



(c)



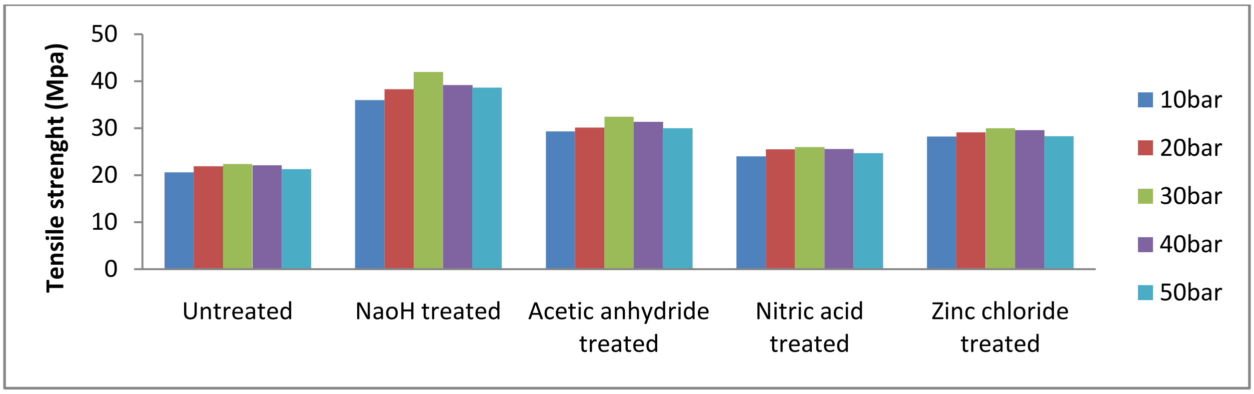
(d)

Fig. 4.42. Effect of Temperature on the tensile strength of composites at 5mins (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

4.10.4. Effect of molding pressure on the tensile strength of the composites

The effect of molding pressure on the tensile strength of the composite were studied at 10, 20, 30, 40, 50 bar compression pressures and temperature of 170^{o}C, fiber length of 3mm and molding time of 10mins. Pressure is necessary because if pressure is not sufficient, it will lead to poor interfacial adhesion of fiber and matrix. If pressure is too high, it may cause fiber breakage and expulsion of enough resin from the composite system.

From the result obtained for *ampelocissus cavicaulis* fiber reinforced composite in Figure 4.43, it was noticed that the tensile strength of the composite both treated and untreated increased as the pressure was increased to 30 bar beyond which the tensile strength started decreasing. For both *adenia lobata* and *ampelocissus leonensis* fiber reinforced composites, the same trends of increase in tensile strength till 30bar was observed. *Morinda morindoides* treated and untreated fiber reinforced composite followed equally the same trend. The tensile strength was increased as the pressure was increased to 40bar beyond which there was decrease in tensile strength. The higher holding pressure needed for *morinda morindoides* fiber reinforced composite can be attributed to the nature of the fiber. Naturally, the fiber had high tensile strength which tremendously increased when treated with chemical. This can be the reason why it was able to withstand higher pressure without suffering the negative effect of fiber breakage. The treated fiber reinforced composites showed higher tensile strength compared to the untreated fiber reinforced composites, though with the same optimum pressure. This can be attributed to the effect of the chemical treatment as shown on the plots. Natural fibers are amenable to modifications as they bear hydroxyl groups from cellulose and lignin. These hydroxyl groups may be involved in the hydrogen bonding within the cellulose molecules there by reducing the activity towards the matrix. Chemical treatments may activate these groups or can introduce new moieties that can effectively interlock with the matrix. Regardless of the type of fiber employed, increasing holding pressure increases molecular orientation of the polymer chains of the matrix (Pantani et al., 2007). Increased molecular orientation results in increased level of crystallinity of the composites. Depending on the orientation of the fibers, majority of the fiber are aligned along the tensile axis (Megat-Yusoff, et. al., 2011). Due to this effect, the composites are said to be anisotropic (Megat-Yusoff, et. al., 2011). These explained the observed improvement in tensile properties of the fiber reinforced composite as the holding pressure was increased. However, if the holding pressure is increased beyond the optimal level, the molecular chains of the composites become overly packed resulting in chain entanglements and reduced crystallinity (Ota et al., 2005; Cermak et al., 2005). Hence, increasing the holding pressure beyond its limit during compression molding of the fiber – HDPE composite could contribute to poor tensile properties.



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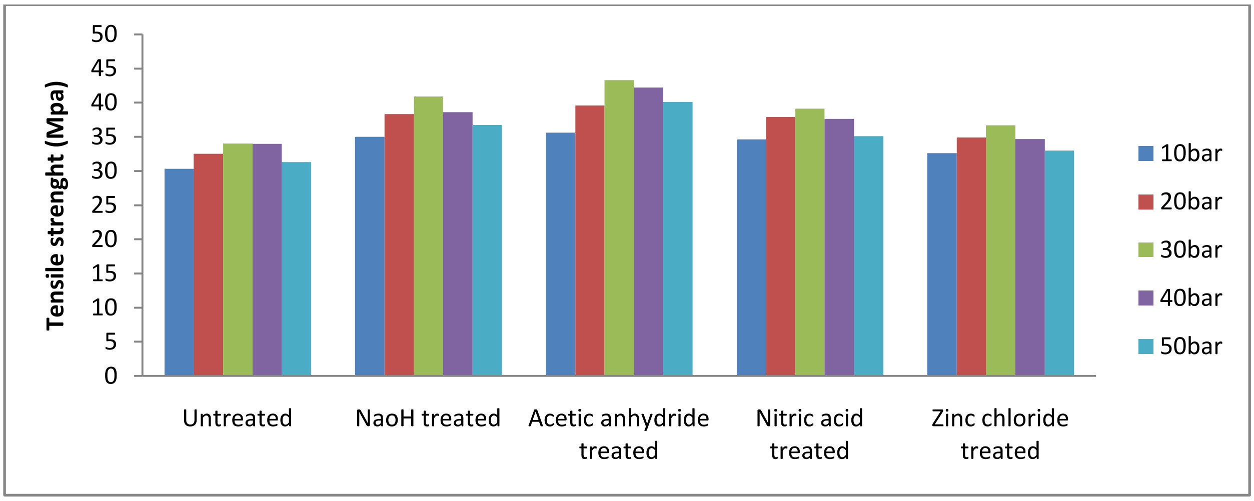

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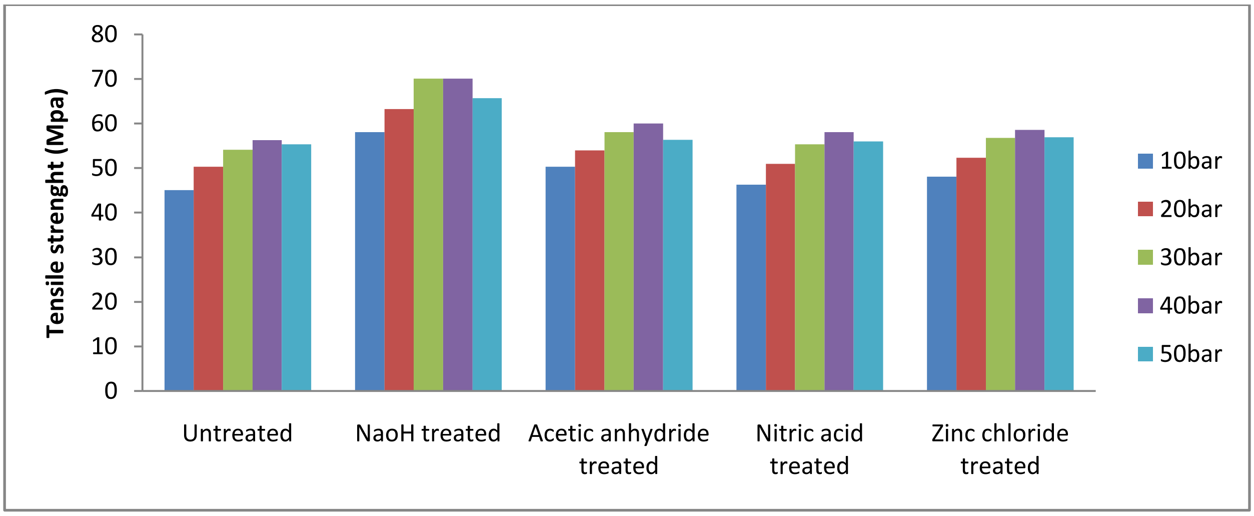

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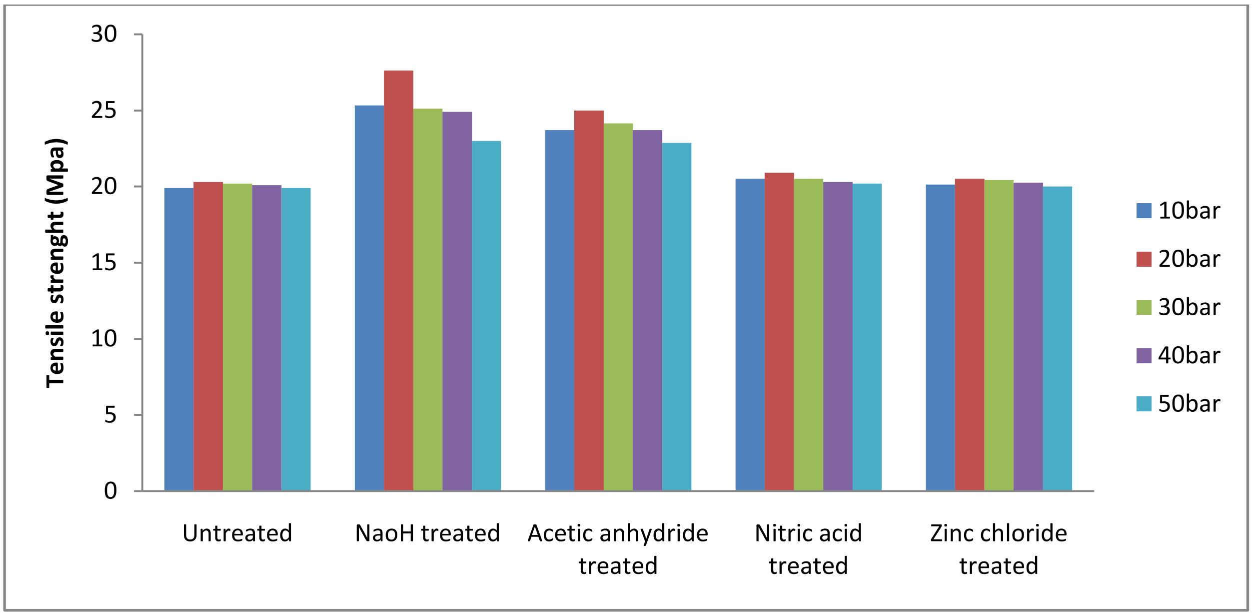


(d)

Fig.4.43. Effect of chemical treatments at various pressures on the tensile strength of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

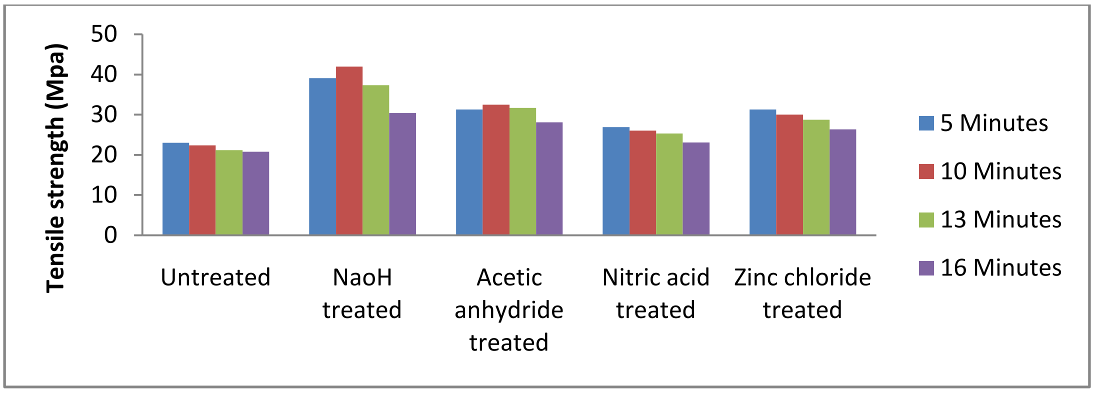
4.10.5. Effect of molding time on the tensile strength of the composite

The effect of molding time on the compression molding of fiber reinforced composites were studied at holding time of 5mins, 10mins, 13mins, 16mins, and 20mins. Molding temperature of 170°C and 140°C, molding pressure of 30bar, fiber length of 3mm and fiber loading of 30wt% were used. The holding time is necessary because if time of application of these factors (temperature and pressure) is not sufficient (high or low), it may cause any of defects associated with insufficient pressure or temperature. The summaries of the results are shown in Figures 4.44 and 4.45. It can be seen from the results of *Ampelocissus cavicaulis* treated with NaoH and acetic anhydride fibers reinforced composites that the tensile strength of the composites were increased as time was increased up to 10minutes beyond which there were decrease in tensile strength. This was not the case with untreated *ampelocissus cavicaulis* fiber and fiber treated with zinc chloride and Nitric acid. In this case, the tensile strength started decreasing beyond 5mins at molding temperature of 170°C. The same trend was observed for treated and untreated *Adenia lobata* fiber and treated and untreated *Ampelocissus leonensis fiber*. Beyond 5mins, there was decrease in tensile strength of the fibers. *Morinda morindoides* treated and untreated fiber reinforced composites had different effect on the composite. The tensile strength of the composites increased as time was increased to 10mins, beyond which further increase resulted to decrease in tensile strength. This can be attributed to the natural hardness of morinda morindoides fiber when compared to other fibers.

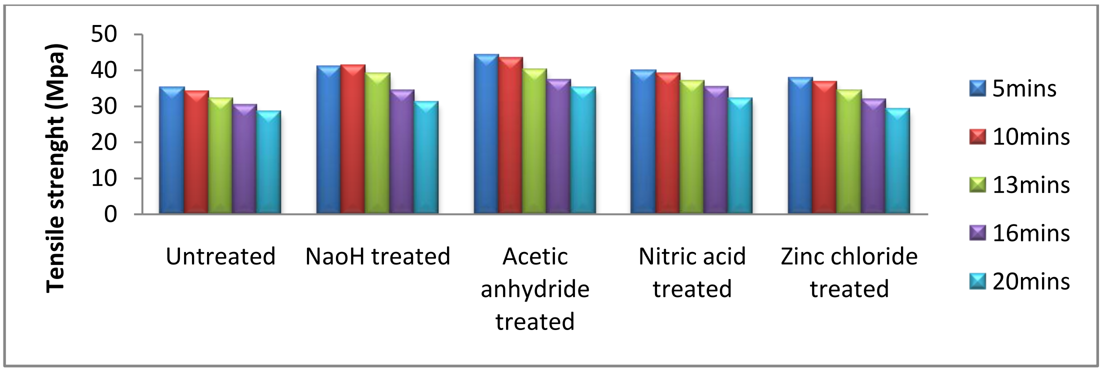
It can equally be observed from the plots (Fig. 4.45) on the effect of the same holding time on the composite when molded at lower temperature of 140^0C, that the optimum holding time for the entire composite increased (Fig. 4.45). It was observed that as the holding time was increased the tensile strength of all the fibers both treated and untreated increased. This can be attributed to the fact that the melting temperature of HDPE is between 130 to 137^0C, and it took longer time for the polymer to melt and intimately mix with the fibers at lower temperature. The lower tensile strength observed when compared to higher temperature of 170^0C despite the longer time interval involved was as a result of high viscosity of the polymer component and thus lower flow capability was observed. This resulted to reduced fiber embedment during compression and therefore lower stability of the composites.

Using lower molding temperature can result in the presence of void on the composites due to inadequate interlock between the matrix and fiber as a result of poor wetting of the polymer. Higher void content is responsible for the lower fatigue resistance, greater affinity to water diffusion and increase variation (Scatter) in the mechanical properties (Kenneth, 1992; Vaxman, et al., 2004).

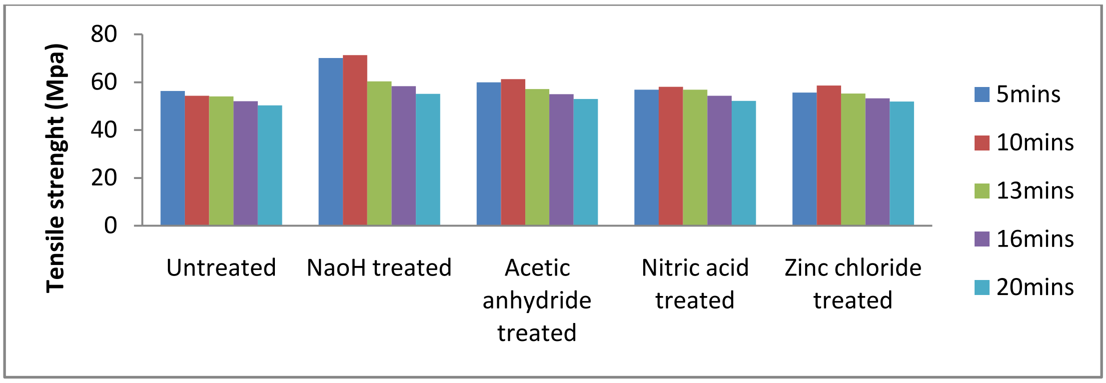
It was equally observed from Figures 4.44-4.45 that the tensile strengths of the treated fibers reinforced composites were higher than the untreated fibers reinforced composites. This situation can be linked to the fact that the chemical treatment enhanced the capability of the fibers to bind effectively with the polymer thus, resulting to higher tensile strength.


Tensile strength (Mpa)
50
40
30
20
10
0
Untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5 Minutes
10 Minutes
13 Minutes
16 Minutes

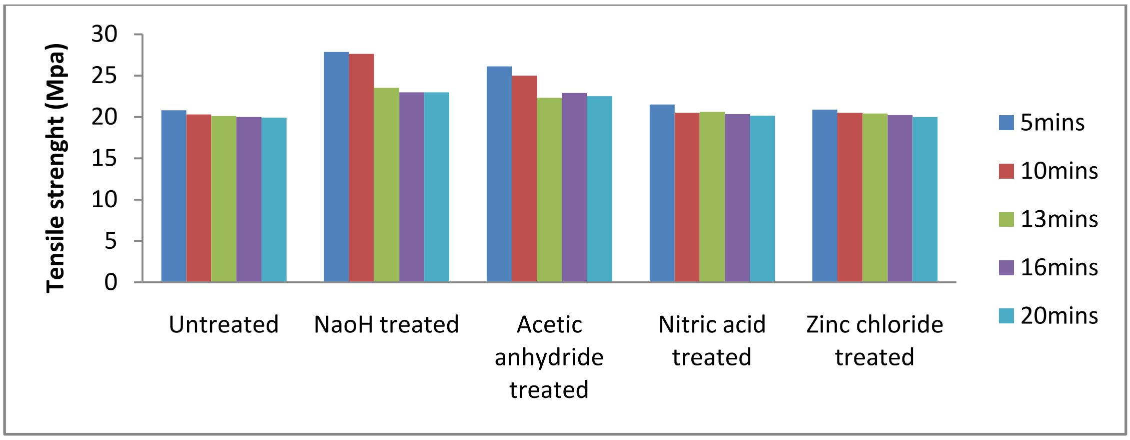
(a)


Tensile strenght (Mpa)
50
40
30
20
10
0
Untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5mins
10mins
13mins
16mins
20mins

(b)

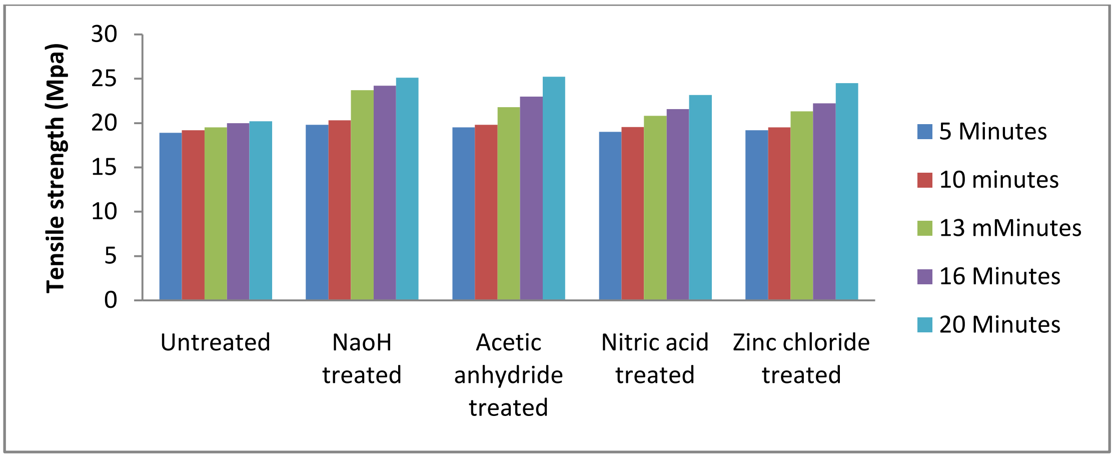

Tensile strenght (Mpa)
80
60
40
20
0
Untreated
NaoH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated
5mins
10mins
13mins
16mins
20mins

(c)

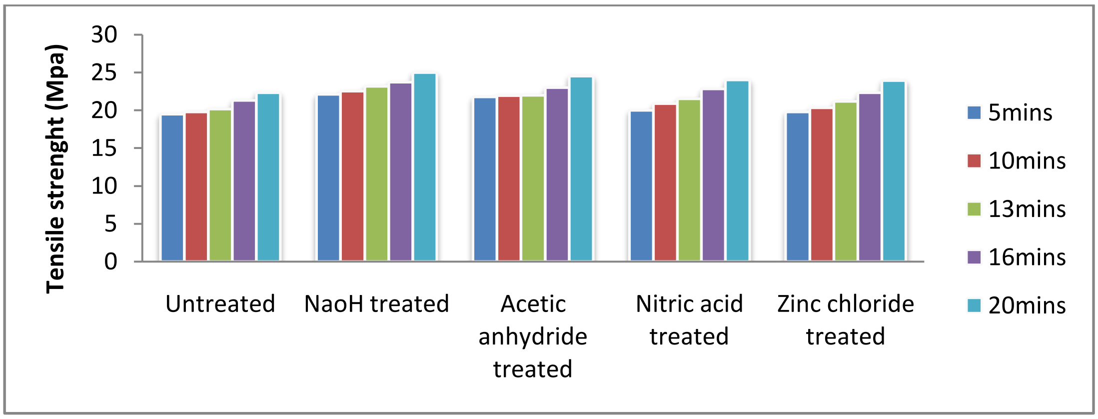


(d)

Fig. 4.44. Effect of holding time at 170^{o}C on the tensile strength of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.



(a)



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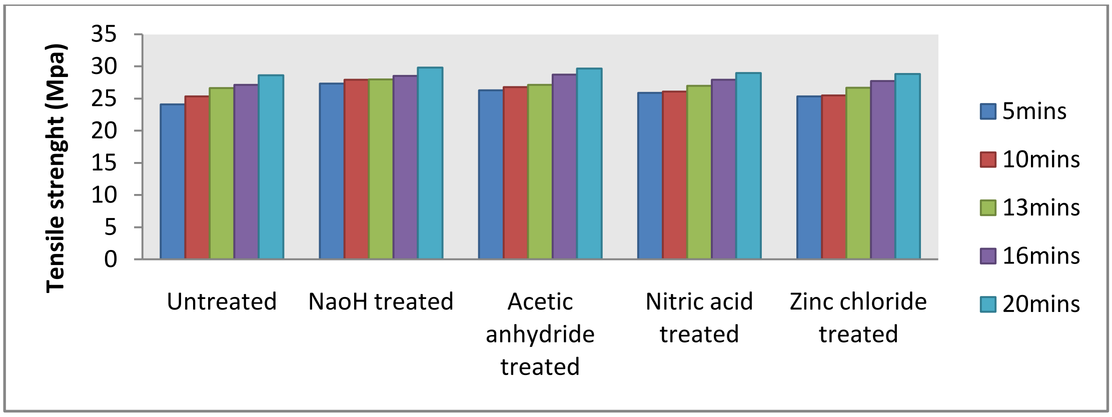

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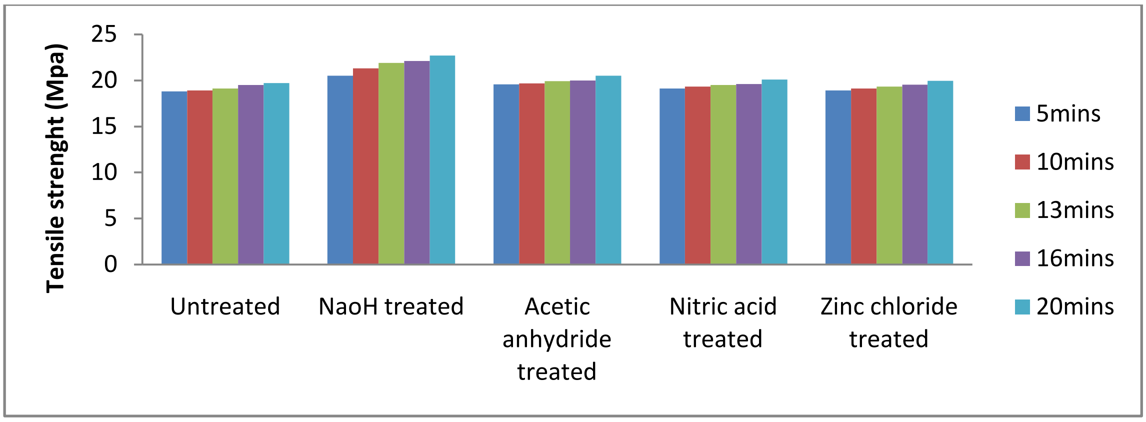


(d)

Fig. 4.45. Effect of holding time at 140°C on the tensile strength of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

4.11. Effect of chemical treatments on the physical properties of the composites

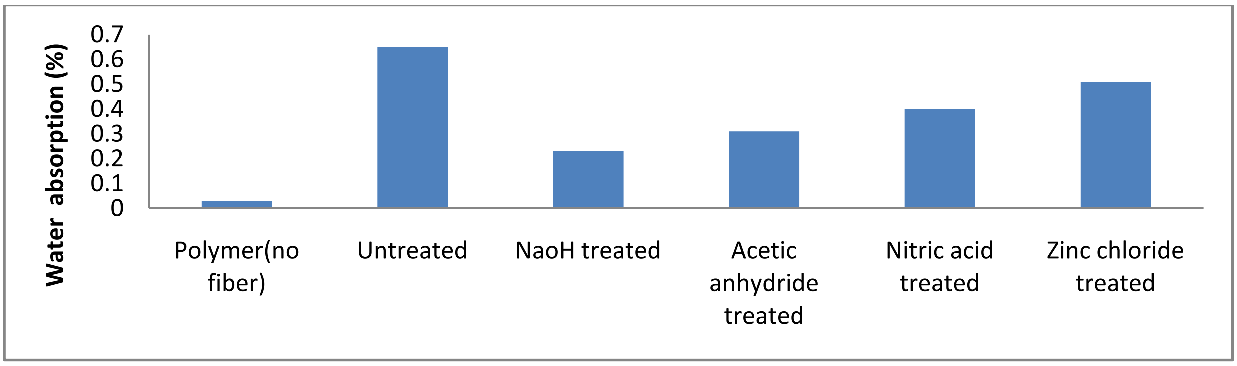
The commercial viability of the newly fabricated natural fibre reinforced polymer composites lies in their physical and chemical properties. Keeping in mind the commercial viability of the composites, a comprehensive study on moisture absorption, swelling behaviors and chemical resistance behavior against different solvents on the fiber reinforced composites has been carried out to assess the potential application of these lignocellulosic fibres as reinforcing material in a number of composites for engineering parts.

4.11.1. Water Absorption Test

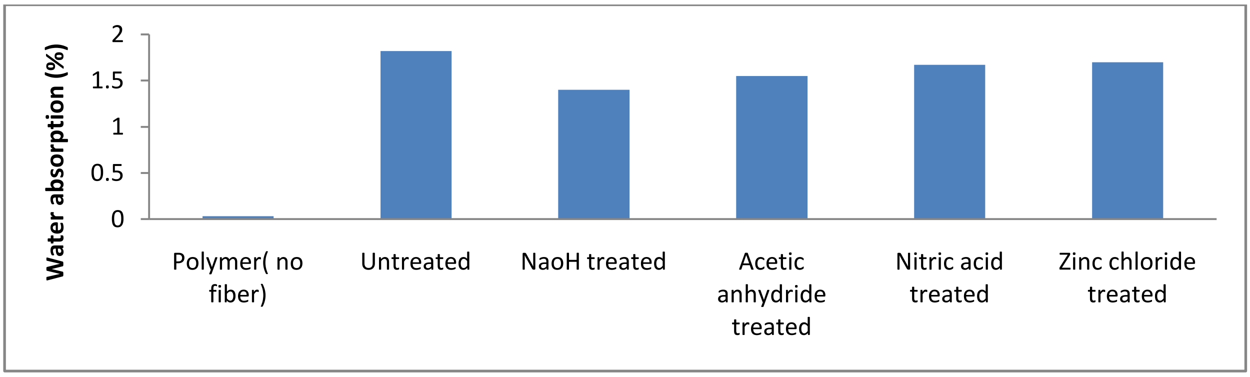
Water absorption is one of the important properties considered for different commercial application of composite. Higher water adsorption leads to poor dimensional stability and hence is not recommended. The water absorption tests of the polymer, untreated and chemically treated fiber based composites were presented as a percentage of dry weight after immersion in water. Natural fiber-HDPE composites have higher water absorption than the plastic polymer as seen in Fig.4.46. Therefore, fiber surface modification, which can reduce the hydroxyl groups in the cell wall of cellulose molecules, is necessary in the reduction of water absorption in composite. The results show that the water absorption of the chemically treated fiber reinforced composites were lower than that of the untreated fiber reinforced composites. The untreated composites absorbed most of the water and the NaOH treated composite absorbed the least for all the composites produced from all the fibers. This suggests that changes in surface chemistry have reduced the affinity of fibers to moisture. There is a large amount of hydrogen bonds (hydroxyl groups –OH) presents between the macromolecules in the plant fiber cell wall. When moisture from the atmosphere comes in contact with the fiber, the hydrogen bonds breaks and hydroxyl groups form new hydrogen bonds with water molecules (Kabir, et al., 2011). The cross section of the fiber becomes the main access to the penetrating water. As a result, when hydrophilic fiber is reinforced with hydrophobic polymer, fiber swelling within the matrix occurs (Kabir, et al., 2011). The water absorption of the fiber can be reduced by different chemical treatments such as alkali, silane, acetylation, benzoylation and peroxide treatment on the fiber surface to remove hydrophilic hydroxyl bonds (Wang et al., 2007). Equally, strong intermolecular fiber-matrix bonding decreased the rate of moisture absorption in composites.

From the results on Fig. 4.47, it was evident that water absorbance was significantly influenced by fiber content. The higher the fiber content, the higher the water uptake. The highest water uptake was seen at the 50wt% fiber loading for almost all the fibers used.

As the fiber loading increases, the formation of agglomerations increases hence it becomes difficult to achieve homogenous dispersion of a fiber at high fiber loading. This agglomeration of the fiber in composites increases the water absorption of the composites. Dimensional stability of composites is important since construction materials should have the ability to withstand the stress of shrinkage or swelling due to changes in temperature and moisture (Motoke et al., 2012). Also from the results, it is evident that water absorption of the composites followed the increasing order of *Ampelocissus leonensis*, *Ampelocissus cavicaulis*, *Adenia lobata*, *Morinda morindoides*. This increase can be attributed to the chemical compositions of the fibers. The order is the order of the increase in cellulose content of the fibers.



(a)



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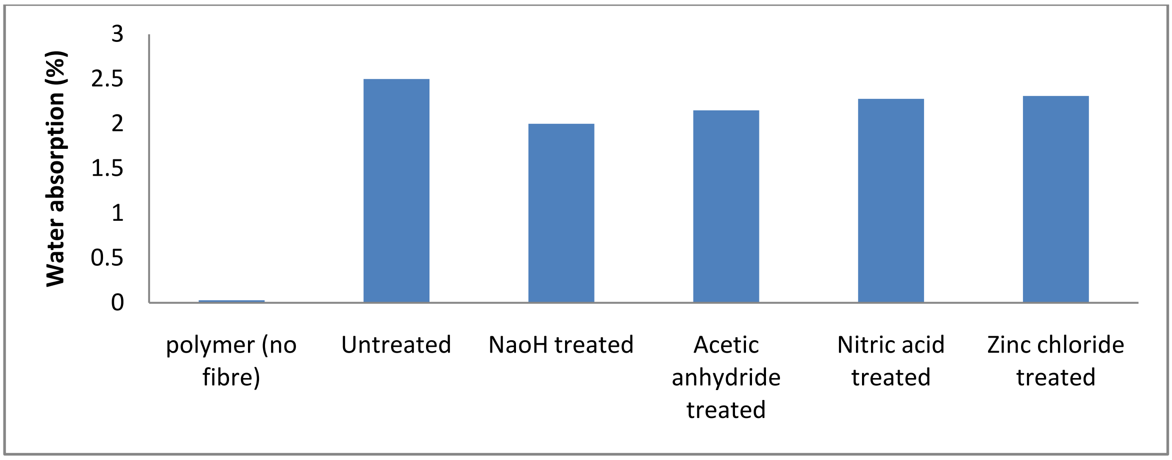

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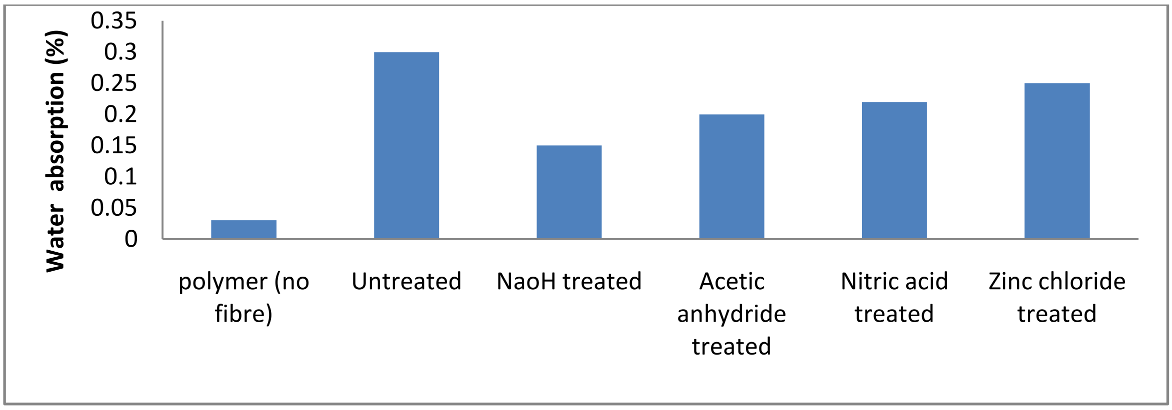
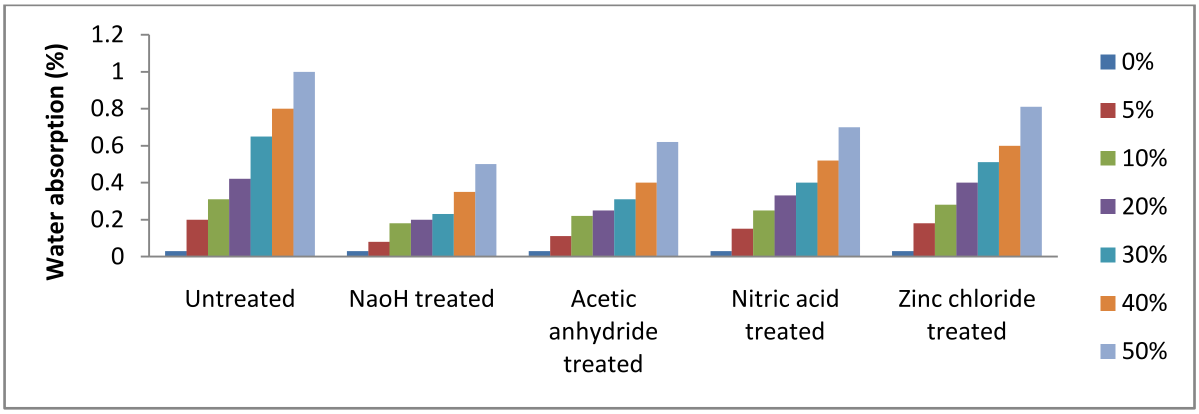
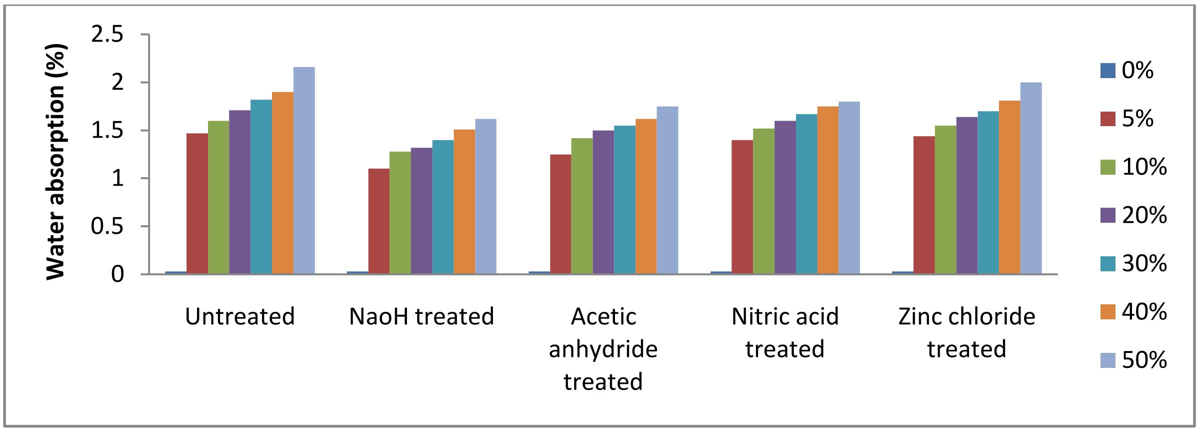


(d)

Fig.4.46. Water absorption test of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.



(a)



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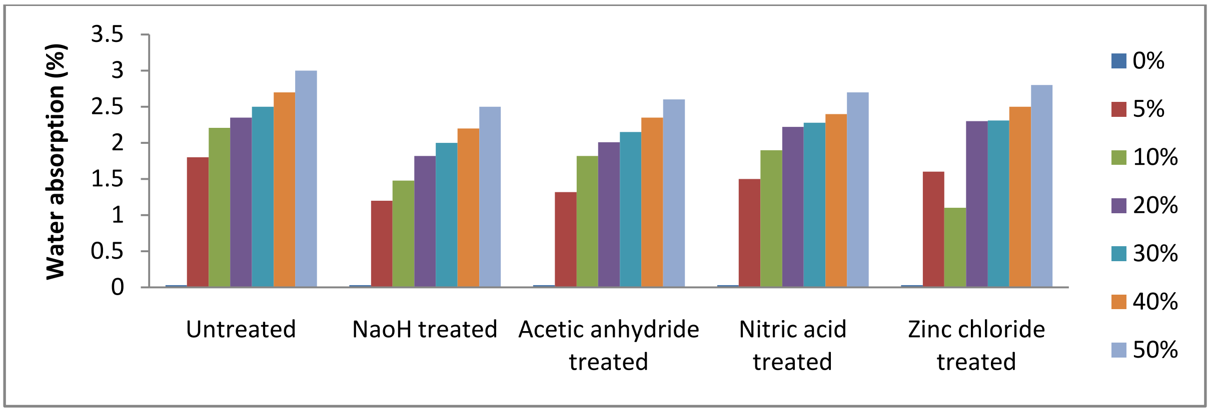

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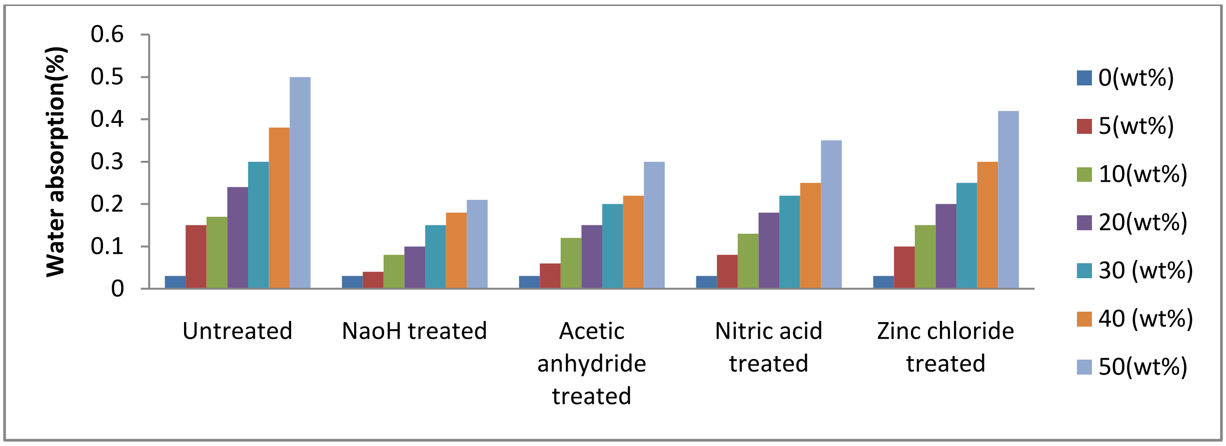


(d)

Fig.4.47. Effect of chemical treatment at various fiber loading on the water absorption of the composites (a) *Ampelocissus cavicaulis* fiber, (b) *Adenia lobata* fiber, (c) *Morinda morindoides* fiber, (d) *Ampelocissus leonensis* fiber.

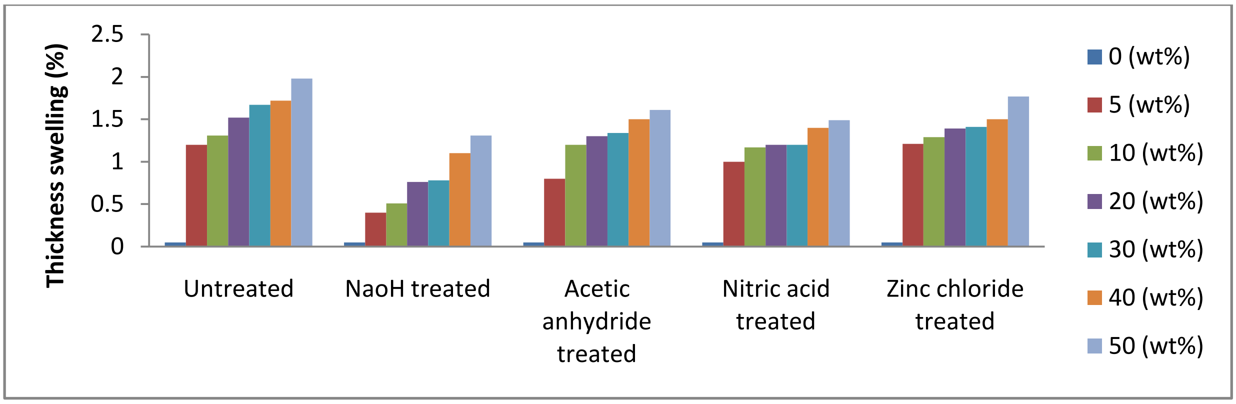
4.11.2. Thickness swelling

The dimensional stability of the composites was equally ascertained by the thickness swelling test performed. Fig. 4.48 shows the thickness swelling behavior of the fiber reinforced composite after being immersed in water for twenty four hours. Significant amount of water absorbed resulted in the swelling of the fiber until the cell walls are saturated with water. As the fiber content decreases, the thickness swelling deceases due to the decrease in water absorption into the composites.

Higher void content (over 20% by volume) is responsible for the lower fatigue resistance, greater affinity to water diffusion and increased variation (Scatter) in mechanical properties (Kenneth, 1992, Vaxman etal 2004). Composites of higher fiber content usually display more void formation (Vaxman etal., 2004). The increase in thickness swelling at higher fiber content is consistent with the fact that more water molecule were able to accumulate in the voids, leading to an increase in the water absorption which in turn led to higher thickness swelling. At higher fiber loading, the micro-level processing of the composites become difficults and leads to fiber layering, which creates micro-voids and cracks within the composites (Khall et al., 2008). Micro-void and crack formation in the composites creates pathways for the water molecules to diffuse easily in composite materials, thereby enhancing water absorption. This was in agreement with the work done by Kumear and siddaramaiah (2005) who noted that swelling thickness is directly proportional to the fiber content of the composites due to hydrophilic nature of lignocellolusic fibers causing the thickness to swell in composites.

It can equally be observed from the graphs (Fig. 4.48) that thickness swelling was more with the untreated fiber than the treated fiber. This was as a result of the chemical treatments done on the fibers before being used as reinforcement. Pretreatment of the fibers replaced some of the hydroxyl groups in the cell wall of the fiber molecule which reduced the hygroscopic nature of the fiber reinforced composites (Sreakala et al., 2000). This reduced the water absorption of the composites and in turn the swelling thickness.

When the composites were exposed to moisture, the fiber swells, as a result of fiber swelling, micro cracking of the brittle HDPE occurs. The high cellulose content in the fibers further contributes to more water penetrating into the interface through the micro crack induced by swelling of fibers creating swelling stresses leading to composites failure (Matoke et al., 2012). As the composite cracks and gets damaged, capillarity and transport via micro cracks becomes active. The capillarity mechanism involves the flow of water molecules along fiber matrix interfaces and a process of diffusion through the bulk matrix. The water molecules actively attack the interface, resulting in debonding of the fiber and matrix (Matoke et al., 2012).



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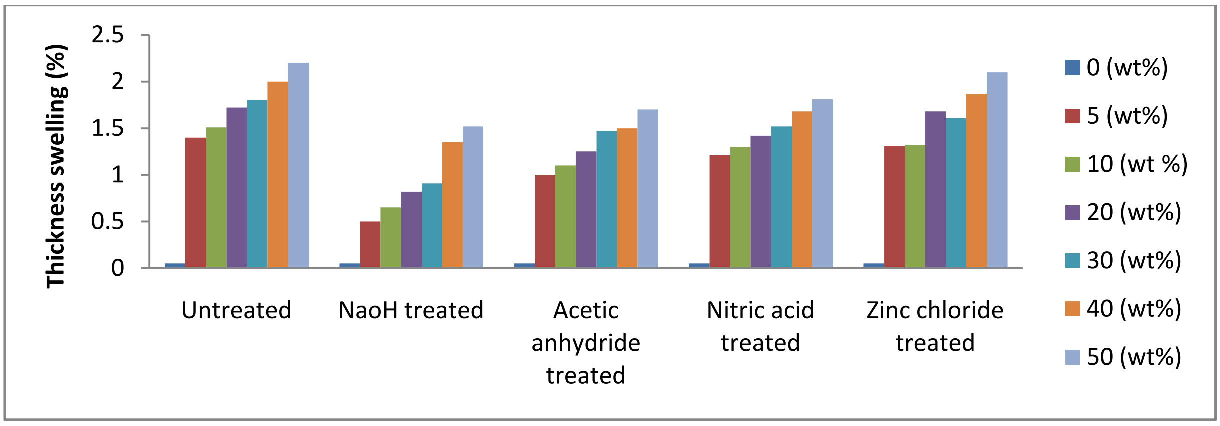

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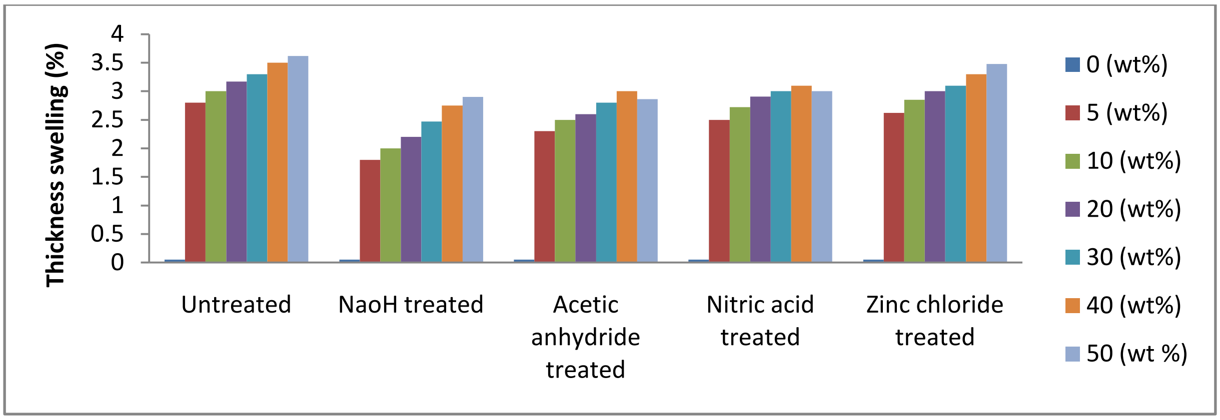

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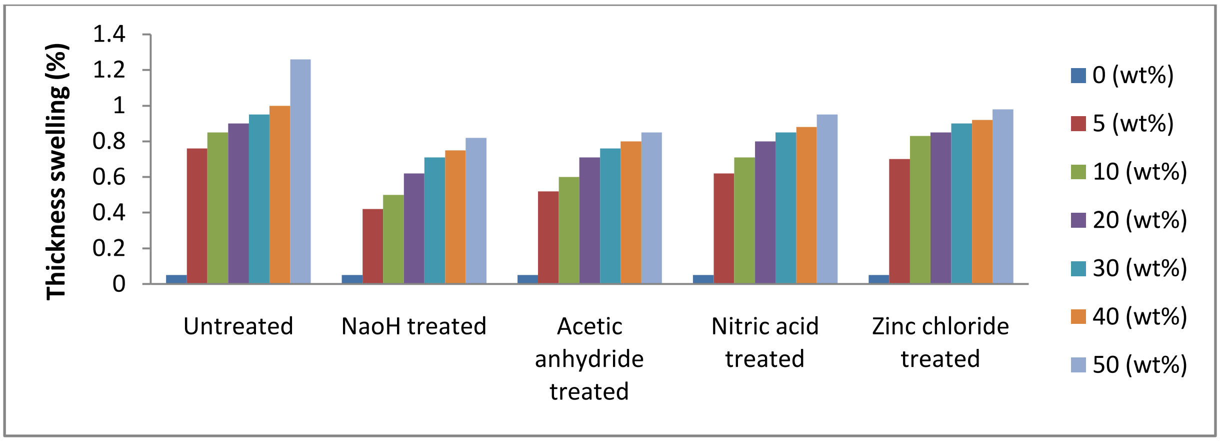
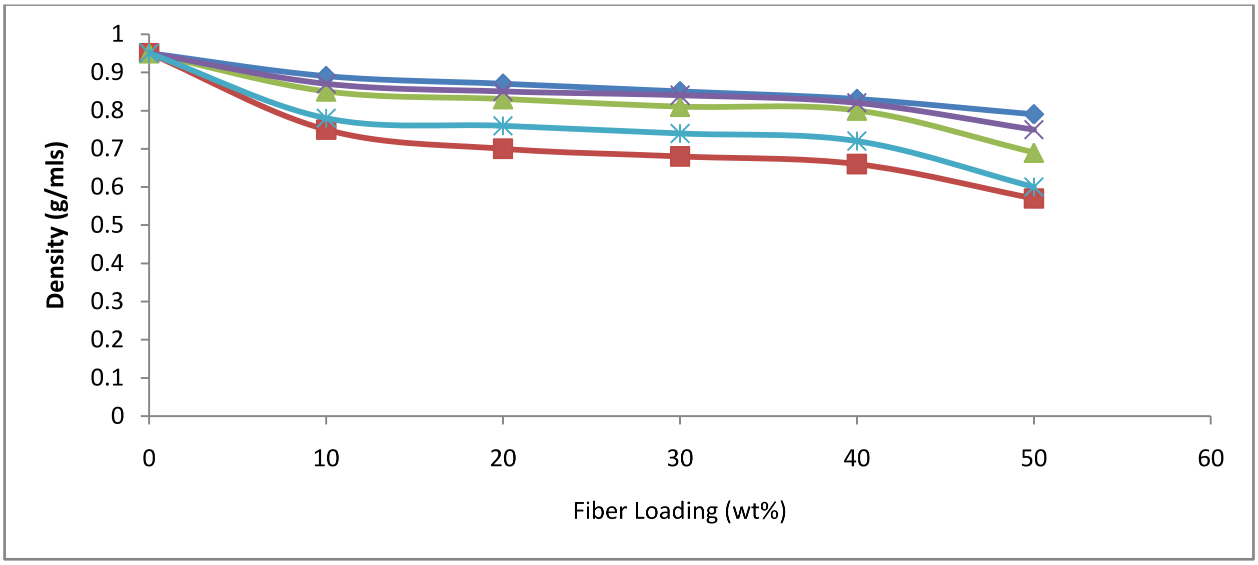


(d)

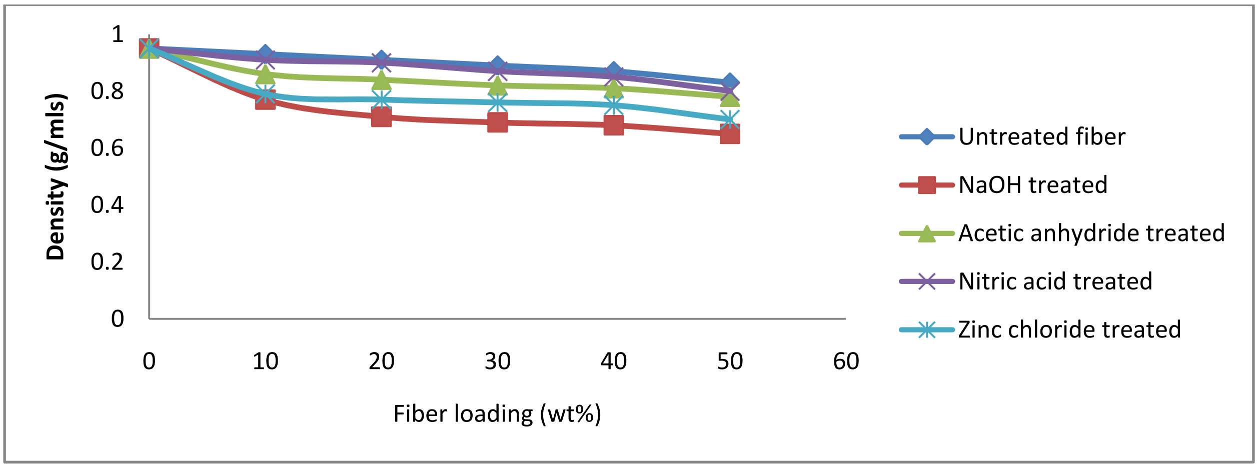
Fig.4.48. Effect of fiber loading on the thickness swelling of the composites (a) Ampelocissus cavicaulis fiber, (b) Adenia lobata fiber, (c) Morinda morindoides fiber, (d) Ampelocissus leonensis fiber.

4.11.3. Density of composites

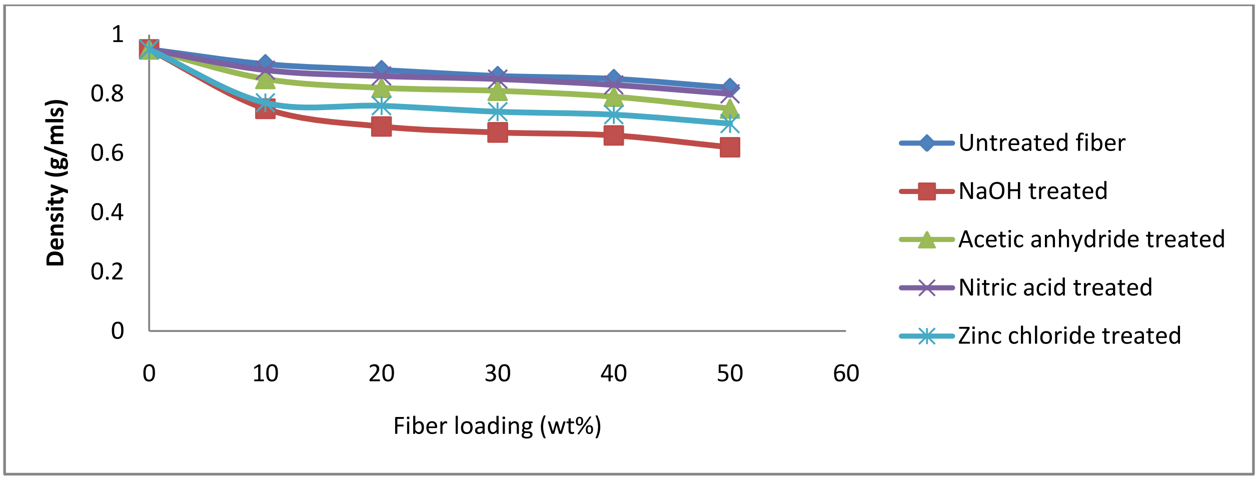
Density is one of the most important factors determining the properties of the composites and it depends on the relative proportion of matrix and reinforcing materials (Kaundal et al, 2012). The effect of the content of the natural fibers and their chemical treatments on the density of the composite is shown in Fig. 4.49. We noticed a weak reduction in the density with the increase in fiber content. This decrease is due to the difference between the density of the fibers and the matrix. The presence of fibers as reinforcement in the HDPE matrix reduces the density of the polymer composite and hence makes them light weight. This may be due to presence of the higher air content from the fiber. Weak improvements on the density of the composites were observed when the fibers were treated with the chemicals. These results can be attributed to the effect of the chemicals which eliminates the cell wall components. This was in conformity with the work done by Bouhank and Nekkaa (2014), on effect of chemical treatment on the structural, mechanical and morphological properties of poly (Vinyl Chloride) / *spartium junceum* fiber composites. Sodium hydrochloride treated fiber reinforced composite had lowest density compared to other chemically treated fiber composite. This observation can be attributed to the effectiveness of sodium hydroxide in removing the lignin and hemicellulose composites of the fiber thus, decreasing the density of the fibers. Besides, the presence of voids is also playing a role in reducing the density of all the composites. The chemical treatment helped to remove the waxes, hemicellulose and part of the lignin present on the fiber surface which acts as cement covering the cellulose. The removal of these untreated substances decreased the weight of the fiber, thus, decreased the density of the treated fiber. This explained the reasons why the density of the treated fiber reinforced composites were lower than the untreated fiber reinforced composites. The effectiveness of each chemical in removing these unwanted substance explained the trend observed on the graph. The more effectiveness of the chemical in treating the fiber, the lower the density of the composite prepared from the fiber. It was observed that the density of nitric acid treated fiber reinforced composite was higher than other chemically treated composites. According to Vantard et al (2013), oxidation of fiber surface in nitric acid creates a rough surface which significantly increases the specific surface area and also generates a high density of hydroxyl group, carboxylic acid and lactones. These hydroxyl groups added to the fiber increased the density of the treated fiber and subsequently increased that of the composites.


Density (g/mls)
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
0
10
20
30
40
50
60
Fiber Loading (wt%)

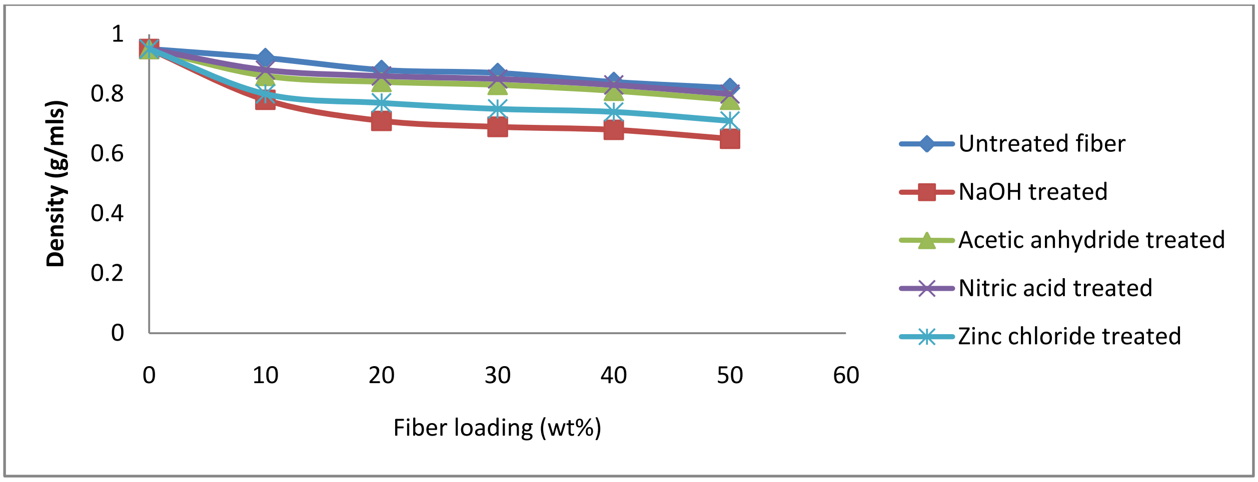
(a)


Density (g/mls)
1
0.8
0.6
0.4
0.2
0
0
10
20
30
40
50
60
Fiber loading (wt%)
Untreated fiber
NaOH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated

(b)


Density (g/mls)
1
0.8
0.6
0.4
0.2
0
0
10
20
30
40
50
60
Fiber loading (wt%)
Untreated fiber
NaOH treated
Acetic anhydride treated
Nitric acid treated
Zinc chloride treated

(c)



(d)

Fig.4.49. Effect of fiber loading on the density of fiber reinforced composite (a) *Ampelocissus leonensis* (b) *Adenia lobata* (c) *Ampelocissus cavicaulis* (d) *Morinda morindoides*

4.11.4. Chemical Resistance Test

The chemical tests were conducted for both treated and untreated fiber reinforced matrix composites. Chemical resistance test was equally conducted on the polymer matrix without fiber. The effect of some acids, alkali and solvent that is, methylethyl ketone (MEK), 20% ammonium hydroxide, 1N HCl, 0.5N NaOH, benzene, methanol, acetic acid and water on untreated and treated fibers reinforced composites were studied. Tables 4.27 to 4.30 below show percentage weight loss or percentage weight gain values of the composites immersed in the chemicals. It was clearly evident that weight gain was observed for almost all the chemicals reagent except benzene and 20% ammonium hydroxide. This is understandable as the matrix is cross linked and as a result, formation of gel takes place instead of dissolution (Bai and Raghavendra 2014). It was equally observed from the tables that all the composites had weight loss in 20% ammonium hydroxide and Benzene. The matrix without the fiber had weight loss in 0.5N NaOH. The reason was attack of the chemicals on the matrix which resulted to its dissolution in the chemicals. It was equally observed that the composites were also resistant to water.

It was equally observed from the tables that the pretreatment of the fibers with chemicals contributed to good chemical resistance due to good adhesion between the fibre and the matrix. The lower void content that resulted from good adhesion caused lower chemical absorption by the composites. This is because the composite materials were swollen with gel formation rather than dissolving the composites due to higher adhesion between the fiber and the polymer. Chemical resistance test observation of the composites suggests that these composites can be used for making storage tanks for water and chemical and can equally be used in automobiles and transport applications.

Table 4.27. Chemical resistance (%) weight gain/loss for *ampelocissus cavicaulis* fiber reinforced composites

Chemicals	Matrix (no fiber)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
Water	0.03	0.68	0.31	0.35	0.38	0.42
0.5N NaOH	-0.2	0.58	0.15	0.30	0.32	0.40
20% ammonium hydroxide	-1.4	-1.12	-1.12	-1.20	-1.30	-1.32
acetic acid	0.52	0.36	0.36	0.48	0.55	0.58
IN Hcl	0.15	0.38	0.38	0.45	0.50	0.51
Methanol	0.53	0.25	0.25	0.25	0.38	0.42
Benzene	-3.5	-1.5	-1.5	-1.92	-2.21	-2.55

Table 4.28. Chemical resistance (%) weight gain/loss for *Adenia lobata* fiber reinforced composites

Chemicals	Matrix	Untreated	NaOH treated	Acetic anhydride	Nitric acid treated	Zinc chloride treated
Water	0.03	1.90	1.45	1.52	1.64	1.68
0.5N NaOH	-0.2	0.60	1.00	1.15	1.20	1.48
20% ammonium hydroxide	-1.40	-1.6	-1.25	-1.32	-1.5	-2.0
acetic acid	0.52	0.76	1.42	1.55	1.68	1.60
IN Hcl	0.15	0.80	1.36	1.42	1.52	1.55
Methanol	0.53	0.68	0.92	1.09	1.16	1.45
Benzene	-3.5	-3.8	-1.85	-2.20	-2.36	-2.9

Table 4.29. Chemical resistance (%) weight gain/loss for *Morinda morindoides* fiber reinforced composites

Chemicals	Matrix	Untreated	NaOH treated	Acetic anhydride	Nitric acid treated	Zinc chloride treated
Water	0.03	2.2	1.97	2.10	2.25	0.23
0.5N NaOH	-0.2	1.8	1.42	1.51	1.62	1.75
20% Ammonium hydroxide	-1.40	-1.58	-1.62	-1.81	-2.0	-2.20
Acetic acid	0.52	1.72	1.38	1.42	1.55	1.60
INHcl	0.15	1.15	1.20	1.21	1.32	1.35
Methanol	0.53	0.55	0.61	0.75	0.82	0.96
Benzene	-3.5	-2.0	-2.4	-2.6	-3.0	-3.20

Table 4.30. Chemical resistance (%) weight gain/loss for *Ampelocissus leonensis* fiber reinforced composites

Chemicals	Matrix	Untreated	NaOH treated	Acetic anhydride	Nitric acid treated	Zinc chloride treated
Water	0.03	0.31	0.17	0.20	0.22	0.28
0.5N NaOH	-0.2	0.28	0.18	0.21	0.24	0.25
20% Ammonium hydroxide	-1.40	-0.52	-0.25	-0.30	-0.46	-0.50
Acetic acid	0.52	1.32	0.81	1.10	1.15	1.20
INHcl	0.15	1.00	0.58	1.10	0.75	0.80
Methanol	0.53	0.32	0.10	0.16	0.21	0.26
Benzene	-3.5	-1.80	-2.0	-2.2	-3.0	-3.20

4.12. Statistical and Optimization of Composites Production Based on Optimal Fiber Modification and Compounding conditions.

Compression molding is well known technique to develop variety of composite products. The materials are placed in between the molding plates. They flow due to application of pressure and heat and consequently acquire the shape of the mold cavity with high dimensional accuracy which depends upon mold design. The controlling parameters in compression molding method to develop superior and desired properties of the composites are temperature, time and pressure. All the factors are critical and have to be optimized effectively to achieve tailored composite products. If the applied pressure is not sufficient, it will lead to poor interfacial adhesion of fiber and matrix. If the pressure is too high, it may cause fiber breakage, expulsion of enough resin from the composite system (Malloy, 1994). If temperature is too high, properties of fiber and matrix may get changed. If the temperature is lower than desired, fibers may not get properly wetted due to high viscosity of polymers especially for thermoplastic. If time of application of these factors is not sufficient (high or low), it may cause any of defects associated with insufficient pressure or temperature.

In this work, the basic factors that affect compression molding process were optimized using Box Behnken design (BBD) for maximal tensile strength of the composites. Box Behnken design is a type of response surface design that does not contain an embedded factorial or fractional factorial design. The design points fall at the combination of the high and low factor levels and their midpoints. It involved three numeric factors of temperature, time and pressure with one categoric factor of condition of the fiber. Box Behnken designs do not have axial points, thus one is sure that all the design points fall within the safe operating zones.

4.12.1. Analysis of variance for the compression molding process

Analysis of variance is an important technique that was used to analyze the effect of the factors on the response.

The ANOVA Tables for the four fibers reinforced composites are shown on Tables 4.31 to 4.34 below.

For *ampelocissus cavicaulis* (Table 4.31), the model F-value of 137.62 implied that the model was significant. There was only 0.1% chance that a “Model F-Value” this large could occur due to noise. Values of “Prob> F” less than 0.0500 indicated model terms were significant. In this case, single effect of temperature (A) pressure (C), time (B), and fiber conditions (D), quadratic effect of temperature (A^2) and quadratic effect of pressure (C^2) were significant. Values greater than 0.100 indicated the model terms were not significant. The “lack of fit F-value” of 1.20 implied the lack of fit was not significant relative to pure error. There was a 10.13% chance that a “lack of fit F-value” this large could occur due to noise.

R-squared of 0.9510 was high which meant that 95.10% of the variability of the response data can be explained by the model. Adjusted R squared of 0.9444 was in close range with the predicted R-squared of 0.9351. Adequate precision, which measured the signal to noise ratio was high, 41.386.

Table 4.31. ANOVA Table for *Ampelocissus cavicaulis* fiber reinforced composite

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	2755.18	11	250.47	137.62	<0.001
A-Temperature (^{0}C)	21.30	1	21.30	11.70	0.0013
B-Time (mins)	9.8	1	9.8	5.38	0.0476
C-Pressure (Bar)	23.90	1	23.90	13.13	0.0007
D-Fiber condition	2463.87	4	615.97	338.45	<0.0001
AB	6.62	1	6.62	3.64	0.0666
A^2	138.43	1	138.43	76.06	<0.0001
C^2	77.68	1	77.68	42.68	<0.0001
Residual	132.58	73	1.82		
Lack of fit	115.84	53	2.19	1.20	0.1013
Pure Error	16.74	20	0.84		

R – squared = 0.9510, Adj R-squared = 0.94441, Predicted R-squared = 0.9351, Adeq precision = 41.386

For *adenia lobata* (Table 4.32), the model F-value of 29.63 implied the model was significant. There was only a 0.01% chance that a “Model F-Value” this large could occur due to noise.Values of “Prob>F” less than 0.0500 indicate model terms were significant. In this case single effect of temperature (A), single effect of time (B), single effect of pressure (C), single effect of fiber condition (D), interaction effect of temperature and time (AB), quadratic effect of temperature (A^2) and quadratic effect of pressure (C^2) were significant model terms. Values greater than 0.100 indicate the model terms are not significant

The “Lack of Fit F-value” of 1.14 implied that the lack of fit was not significant relative to the pure error. There was a 24.12% chance that a “lack of Fit F-value” this large could occur due to noise. Adeq. precision measured the signal to noise ratio. A ratio greater than 4 was desirable. The value of 23.208 indicated an adequate signal.

TABLE 4.32. ANOVA Table for *Adenia lobata* fiber reinforced composite

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	1046.25	11	95.11	29.63	<0.0001 significant
A-Temperature (^{0}C)	0.016	1	48.16	15.00	0.0003
B-Time (mins)	14.7	1	14.7	4.58	0.0302
C-Pressure (Bar)	46.68	1	46.68	14.54	0.0004
D-Fiber condition	504.48	4	126.12	39.29	<0.0001
AB	15.14	1	15.14	4.72	0.0369
A^2	251.38	1	251.38	78.31	<0.0001
C^2	143.34	1	143.34	44.65	<0.0001
Residual	234.68	73	3.21		
Lack of fit	194.11	53	3.66	1.14	0.2412 not significant
Pure Error	40.57	20	2.03		

R-Squared= 0.8065, Adj R-squared= 0.7803, Pred R-squared=0.7451, Adeq.Preccision= 23.208.

For *morinda morindoidies* (Table 4.33), the Model F-value of 231.13 implies the model was significant. There was only a 0.01% chance that a “Model F-value” this large could occur due to noise. Value of “prob>F” less than 0.0500 indicate model terms were significant.

In this case single effect of temperature (A), single effect of time (B), single effect of pressure (C), single effect of fiber condition (D), quadratic effect of temperature (A^2), and quadratic effect of pressure (C^2) were significant model terms. Value greater than 0.1000 indicate the model terms were not significant. The “Lack of Fit F-Value” of 0.99 implied the lack of Fit was not significant relative to the pure error. There was a 58.24% chance that a “Lack of Fit-value” this large could occur due to noise. The “Pred R-squared” of 0.9552 was in reasonable agreement with the “Adj.R-squared” of

0.9610. “Adeq Precision” measures signal to noise ratio. A ratio greater than 4 was desirable. Ratio of 60.365 indicated an adequate signal.

Table 4.33. ANOVA Table for *Morinda morindoides* fiber reinforced composites

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	1580.94	9	175.66	231.13	<0.001 significant
A-Temperature (^{0}C)	0.58	1	20.58	27.08	0.0001
B-Time (mins)	11.90	1	11.90	15.65	0.0002
C-Pressure (Bar)	4.21	1	4.21	5.54	0.0213
D-Fiber condition	25.83	4	306.46	403.24	<0.0001
A^2	298.95	1	298.46	392.71	<0.0001
C^2	28.80	1	28.80	37.89	<0.0001
Residual	57.06	75	0.76		
Lack of fit	41.21	55	0.75	0.99	0.5824 not significant
Pure Error	15.84	20	0.79		

R-Squared = 0.9652, Adj. R-squared = 0.9610, Pred R-squared = 0.9552, Adeq Precision = 60.365.

For *ampelocissus leonensis* (Table 4.34), the Model F-value of 499.74 implied the model was significant. There was only a 0.01% chance that a “Model F-value” this large could occur due to noise. Values of “Prob>F” less than 0.0500 indicate model terms were significant. In this case single effect of temperature (A), single effect of time (B), single effect of pressure (C), and single effect of fiber condition (D) and quadratic effect of temperature (A^2) were significant model terms. Value greater than 0.1000 indicated the model terms were not significant.

The “Lack of Fit F-Value” of 0.6 implied the lack of Fit was not significant relative to the pure error. There was a 99.87% chance that a “Lack of Fit-value” this large could occur due to noise.

The “Pred R-squared” of 0.9168 was in reasonable agreement with the “Adj.R-squared” of 0.9233.

"Adeq Precision" measures signal to nosie ratio. A ratio greater than 4 was desirable. A ratio of 47.859 indicated an adequate signal.

Table 4.34. ANOVA Table for *Ampelocissus leonensis* fibre reinforced composite

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	1399.26	8	174.91	499.74	<0.0001 significant
A-Temperature (^{0}C)	130.65	1	130.65	373.29	<0.0001
B-Time (mins)	65.13	1	65.13	186.09	<0.0001
C-Pressure (Bar)	9.32	1	9.32	26.63	0.0091
D-Fiber condition	1120.64	4	280.16	800.46	<0.0001
A^2	73.51	1	73.51	210.03	<0.0001
Residual	26.49	76	0.35		
Lack of fit	10.79	56	0.192	0.6	0.9987 not significant
Pure Error	15.7	20	0.785		

R-Squared = 0.9297, Adj. R-Squared = 0.9233, Pred R-Squared = 0.9168, Adeq Precision = 47.859.

4.12.2. Predictive model equations for the compression molding of the composites

Model equations were developed both in actual and coded form. Table 4.35 shows the coded levels of the categoric factors used in this work. Zinc chloride being the last level used served as the reference point with "-1-1-1-1" as the level. D[1] means runs involving only untreated fiber, D[2] means runs involving only sodium hydroxide treated fiber, D[3] means runs involving only acetic anhydride treated fiber and D[4] means runs involving Nitric acid treated fiber.

The coded equations can be seen as five equations, one comprising D[1] with all its interactions, two comprising D[2] with all its interactions, three comprising D[3] with all its interactions, four comprising D[4] with all its interactions, while the fifth one, D[5] is the one in which all the terms and their interactions were eliminated.

Table 4.35. Coded levels of the categoric factors for compression molding process.

	Name	D[1]	D[2]	D[3]	D[4]
1	Untreated	1	0	0	0
2	NaoH treated	0	1	0	0
3	Acetic anhydride treated	0	0	1	0
4	Nitric acid treated	0	0	0	1
5	Zinc chloride treated	-1	-1	-1	-1

- Model equation for compression molding of *Ampelocissus cavicaulis* fiber reinforced composite

The model equations were developed in both actual and coded forms after removing the insignificant factors.

Final model equation in coded form:

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C – 5.27 D[1] + 9.32D[2] + 1.66 D[3] – 5.21 D[4] – 0.58AB – 2.56A^2 – 1.92C^2. (4.78)

Final equations in actual form:

Untreated:

Tensile strength (Mpa) = - 178.65673 +2.27062 Temperature + 0.94710 Time + 0.24908 Pressure – 5.75500E-003 Temperature. Time – 6.40171E – 003 – Temperature^2 – 4.79546E – 003 Pressure^2 (4.79)

NaOH Treated:

Tensile strength (Mpa) =- 164.05849 + 2.27062 Temperature + 0.94710 Time + 0.24908 Pressure – 5.75500E – 003 Temperature Time – 6.40171E-003 Temperature^2+ - 4.79546E-003 Pressure^2 (4.80)

Acetic anhydride treated:

Tensile strength (Mpa) = - 171.72320 + 2.27062 Temperature + 0.94710 Time + 0.24908 Pressure – 5.7550E-003 Temperature. Time – 6.40171E-003 Temperature^2 – 4.79546E-003 Pressure^2 (4.81)

Nitric acid treated:

Tensile strength (Mpa) = - 178.59555 + 2.27062 Temperature + 0.94710 Time + 0.24908 Pressure – 5.7550E-003 Temperature. Time – 6.40171E-003 Temperature^2 – 4.79546E-003 Pressure^2 (4.82)

Zinc chloride treated:

Tensile strength (Mpa) = - 173.88202 + 2.27062 Temperature + 0.94710 Time + 0.24908 Pressure – 5.7550E-003 Temperature. Time – 6.40171E-003 Temperature^2 – 4.79546E-003 Pressure^2 (4.83)

Equation 4.78 can be split into five different equations, each for each chemical type.

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C – 5.27 D[1] – 0.58AB – 2.56A^2 – 1.92C^2 (4.84)

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C +9.32 D[2] – 0.58AB – 2.56A^2 – 1.92C^2 (4.85)

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C – 1.66 D[3] – 0.58AB – 2.56A^2 – 1.92C^2 (4.86)

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C – 5.21 D[4] – 0.58AB - 2.26A^2 – 1.92C^2 (4.87)

Tensile strength (Mpa) = + 30.46 + 0.73A – 0.16B – 0.77C - 0.58AB – 2.56A^2 – 1.92C^2 (4.88)

In equation 4.78, effect of the factors used for molding gave intercept of 30.46. Effect of using untreated fiber adjusted the slope negatively by 5.27. The use of NaOH treated fiber as reinforcement increased the sensitivity by 9.32. The effect of acetic anhydride treated fiber equally increased the sensitivity by 1.66; effect of nitric acid treated fiber decreased the slope by 5.21. Equation 4.88 was used as the reference equation where all the categorical factors with their interaction were eliminated. It shows that single effect of temperature increased the slope by 0.73, while its quadratic effect decreased the slope by 2.56. Effect of time decreased the slope by 0.16 while its interaction with temperature equally decreased the slope by 0.58. Single effect of pressure decreased the sensitivity by 0.77 while its quadratic effect decreased it by 1.92.

- Model equations for compression molding of *Adenia lobata* fiber reinforced composite

The model equation for the compression molding of *Adenia lobata* fiber reinforced composite is shown on the equations below after removing the insignificant factors.

Final Equation in terms of coded factors;

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C - 3.58 D[1] + 1.01 D[2] + 3.47 D[3] + 0.85 D[4] – 0.87AB – 3.45A^2 – 2.61^2 (4.89)

Final equation in terms of actual factors:

Untreated:

Tensile strength (Mpa) = -241.47515 =3.07497 temperature (^{0}C) + 1.44070 time (mins) + 0.33684 Pressure (Bar) – 8.70000E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 (^{0}C)^2 – 6.51428E-003 Pressure (Bar)^2 (4.90)

Fiber condition: NaOH treated

Tensile strength (Mpa) = -236.88397 +3.07497 Temperature (^{0}C) + 1.44070 Time (mins) + 0.33684 Pressure (Bar) – 8.70000E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 (^{0}C)^2 – 6.51428E-003 Pressure (Bar)^2 (4.91)

Acetic anhydride treated:

Tensile strength (Mpa) = 234.42809 + 3.07497 Temperature (^{0}C) + 1.44070 Time (mins) + 0.33684 Pressure (Bar) – 8.70000E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 (^{0}C)^2 – 6.51428E-003 Pressure (Bar)^2 (4.92)

Nitric acid treated:

Tensile strength (Mpa) = – 237.04632 + 3.07497 Temperature (^{0}C) + 1.44070 Time (mins) + 0.33684 Pressure (Bar) – 8.70000E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 (^{0}C)^2 – 6.51428E-003 Pressure (Bar)^2 (4.93)

Zinc chloride treated:

Tensile strength (Mpa) = – 239.65456 + 3.07497 Temperature (^{0}C) + 1.44070 Time (mins) + 0.33684 Pressure (Bar) – 8.70000E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 Temperature (^{0}C) Time (mins) – 8.62678E-003 (^{0}C)^2 – 6.51428E-003 Pressure (Bar)^2 (4.94)

Equation 4.89 can be seen as five different equations;

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C – 3.58D[1] – 0.87AB – 3.45A^2 – 2.61C^2 (4.95)

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C + 1.01 D[2] – 0.87AB – 3.45A^2 – 2.61C^2 (4.96)

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C + 3.47 D[3] – 0.87AB – 3.45A^2 – 2.61C^2 (4.97)

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C + 0.85 D[4] – 0.87AB – 3.45A^2 – 2.61C^2 (4.98)

Tensile strength (Mpa) = + 39.39 + 1.10A – 0.19B – 1.08C + 0.87AB – 3.45A^2 – 2.61C^2 (4.99)

- Model equations for compression molding of *Morinda morindoides* fiber reinforced composite

Both the coded and actual form of the model equation is shown below after removing the insignificant factors.

Final Equation in terms of coded factors:

Tensile strength (Mpa) = + 23.97 + 0.72 A – 0.55B – 0.32C – 6.42 D[1] + 4.58 D[2] + 2.58 D[3] + 0.87 D[4] – 3.76 A^2 + 1.17 C^2 (4.100)

Final Equation in terms of Actual factors:

Tensile strength (Mpa) = + 256.22204 + 3.23448 Temperature (^{0}C) – 0.10910 time (Mins) – 0.19140 Pressure (bar) – 9.40770E – 003 Pressure (Bar)^2 (4.101)

NaoH treated;

Tensile strength (Mpa) = + 245.22616 + 3.23448 Temperature (0C) – 0.10910 time (mins) – 0.19140 Pressure (bar) – 9.40770E – 003 Pressure (Bar)^2 (4.102)

Acetic anhydride treated;

Tensile strength (Mpa) = + 247.22734 + 3.23448 Temperature (^{0}C) – 0.10910 time (mins) – 0.19140 Pressure (bar) – 9.40770E – 003 Pressure (Bar)^2 (4.103)

Nitric acid treated;

Tensile strength (Mpa) = + 248.93910 + 3.23448 Temperature (^{0}C) – 0.10910 time (mins) – 0.19140 Pressure (bar) – 9.40770E – 003 Pressure (Bar)^2 (4.104)

Zinc Chloride treated;

Tensile strength (Mpa) = + 251.40792 + 3.23448 Temperature (0C) – 0.10910 time (MIns) – 0.19140 Pressure (bar) – 9.40770E – 003 Pressure (Bar)^2 (4.105)

Equation 4.100 can be split into five different equations as shown below;

Tensile strength (Mpa) = + 23.97 + 0.72A – 0.55B – 0.32C – 6.42D[1] – 3.76A^2 + 1.17C^2 (4.106)

Tensile strength (Mpa) = + 23.97 + 0.72A – 0.55B – 0.32C + 4.58D[2] – 3.76A^2 + 1.17C^2 (4.107)

Tensile strength (Mpa) = + 23.97 + 0.72A – 0.55B – 0.32C + 2.58D [3] – 3.76A^2 + 1.17C^2 (4.108)

Tensile strength (Mpa) = + 23.97 + 0.72A – 0.55B – 0.32C + 0.87D [4] – 3.76A^2 + 1.17C^2 (4.109)

- Model equations for compression molding of *Ampelocissus leonensis* fiber reinforced composite

The model equations were presented in both coded and actual forms in equations below, after removing the insignificant factors.

Final Equation in Terms of Coded factors;

Tensile strength (Mpa) = +23.40 – 1.81A – 1.28B – 0.48C – 6.25D[1] - 4.33 D[2] + 2.33 D[3] + 0.92 D[4] (4.110)

Untreated fiber;

Tensile strength (Mpa) = + 35.78246 + 090362 Temperature (^{0}C) – 0.25520 time (mins) – 0.024137 Pressure (Bar) (4.111)

NaoH treated fiber;

Tensile strength (Mpa) = + 46.37010 + 090362 Temperature (^{0}C) – 0.25520 time (Mins) – 0.024137 Pressure (Bar) (4.112)

Acetic anhydride treated;

Tensile strength (Mpa) = + 44.36481 + 090362 Temperature (^{0}C) – 0.25520 time (mins) – 0.024137 Pressure (Bar) (4.113)

Nitric acid treated fiber;

Tensile strength (Mpa) = + 42.95187 + 090362 Temperature (^{0}C) – 0.25520 time (Mins) – 0.024137 Pressure (Bar) (4.114)

Zinc chloride treated;

Tensile strength (Mpa) = + 40.70657 + 090362 Temperature (^{0}C) – 0.25520 time (Mins) – 0.024137 Pressure (Bar) (4.115)

Equation 4.110 can be split into five different equations;

Tensile strength (Mpa) = + 23.40 – 1.81A – 1.28B – 0.48C – 6.25 D[1] (4.116)

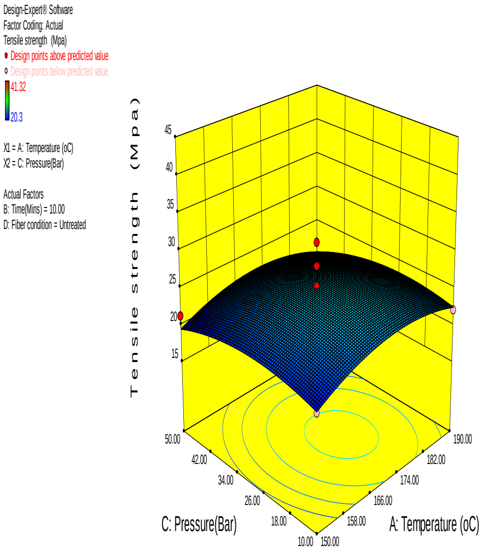
Tensile strength (Mpa) = + 23.40 – 1.81A – 1.28B – 0.48C – 4.33 D[2] (4.117)

Tensile strength (Mpa) = + 23.40 – 1.81A – 1.28B – 0.48C – 2.33 D[3] (4.118)

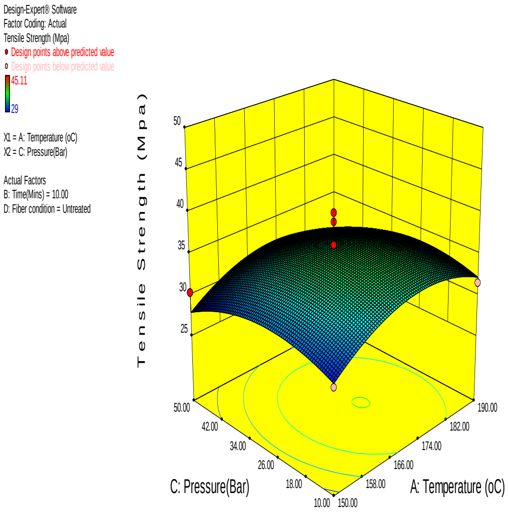
Tensile strength (Mpa) = + 23.40 – 1.81A – 1.28B – 0.48C (4.119)

4.12.3. Three dimensional plots for compression molding process

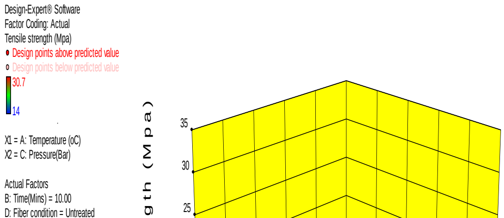
Three dimensional plots of the processing factors (pressure and temperature) on the tensile strength of the fiber were shown on Fig. 4.50. The shape of the plots revealed the nature of the model and the type of the optimization involved. From the 3D plot of *Ampelicissus cavicalulis*, *Adenia lobata* and *Morinda morindoides*, it was observed that quadratic model was involved, but Amplelocissus *leonensis* showed a linear model. Equally, the four plots indicated that maximization optimization was involved as was seen that the plots bulged out. The optimum conditions can be obtained by climbing the hill to the apex. Descending the hill leads to decrease in tensile strength. The 3D plots of *ampelocissus cavicaulis*, *Adenia lobata* and *Morinda morindoides* show that tensile strength was initially increased as temperature and pressure were increased to a point that further increase in both factors resulted to decrease in tensile strength. The nature of the 3D surface of the *Ampelocissus leonensis* showed that there was decrease in tensile strength as both factors were increased.

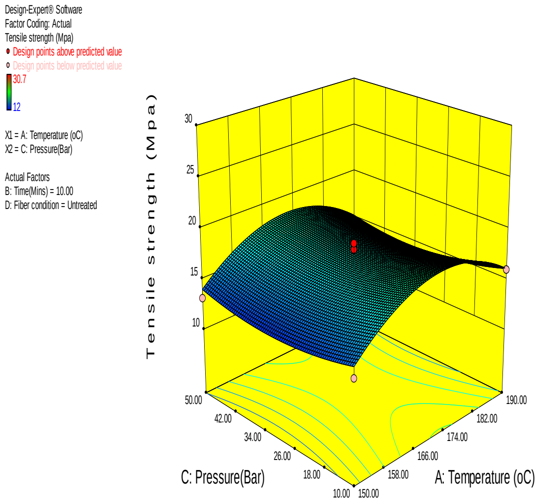


(a)



(b)





(c) (d)

Fig. 4.50. 3D Surface plots for temperature interaction with pressure for compression molding process (a) *Ampelocissus cavicaulis* fiber (b) *Adenia lobata* fiber (c) *Morinda morindoides* fiber (d) *Ampelocissus leonensis* fiber

4.12.4. Optimum conditions for compression molding of composites

The maximization optimization was set as goal to be achieved. The optimum conditions were selected based on the highest desirability. The optimum conditions were shown on Tables 4.36 to 4.39 for all the fiber types used as reinforcement.

Table 4.36. Optimum conditions for compression molding of *Ampelocissus cavicaulis* fiber reinforced composite

Factors	Untreated fiber	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chlorine treated
Temperature (^{0}C)	171.31	177.83	178.31	171.32	170.11
Time (Mins)	5.0	5.00	5.00	5.00	5.00
Pressure (Bar)	28.19	26.33	29.10	29.33	29.12
Desirability	0.500	0.944	0.883	0.522	0.79
Predicted values Tensile strength (MPa)	23.59	40.196	32.531	25.65	30.3706

Table 4.37. Optimum conditions for compression molding of *Adenia lobata* fiber reinforced composites

Factors	Untreated fiber	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chlorine treated
Temperature (^{0}C)	175.68	172.89	172.88	171.86	171.31
Time (Mins)	5.0	5.00	5.0	5.0	5.0
Pressure (Bar)	23.02	26.77	25.69	28.51	26.34
Desirability	0.560	0.897	0.988	0.745	0.673
Predicted values Tensile strength (MPa)	36.370	42.0073	43.463	40.83	38.18

Table 4.38. Optimum conditions for compression molding of *Morinda morindoides* fiber reinforced composites

Factors	Untreated fiber	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chlorine treated
Temperature (^{0}C)	179.31	178.11	178.72	177.31	178.11
Time (Mins)	5.0	5.00	5.0	5.0	5.0
Pressure (Bar)	49.11	40.3	41.31	39.38	42.11
Desirability	0.721	0.991	0.960	0.889	0.825
Predicted values Tensile strength (MPa)	56.211	78.52	66.00	61.31	58.11

Table 4.39. Optimum conditions for compression molding of *Ampelocissus leonensis* fiber reinforced composites

Factors	Untreated fiber	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chlorine treated
Temperature (^{0}C)	150.00	152.42	164.71	155.17	156.11
Time (Mins)	5.00	5.0	5.00	5.00	5.00
Pressure (Bar)	20.00	20.07	14.66	26.75	120.55
Desirability	0.721	1.00	8.918	0.835	0.705
Predicted values Tensile strength (MPa)	21.00	30.74	28.00	25.31	24.11

4.12.5. Validation of the optimum condition for compression molding of the composite

The optimum conditions above were validated by repeating the experiment at the said optimum conditions. This was necessary to obtain the deviation of the experimental values from the predicted values as percentage error. The magnitude of the error tells us how valid the optimum conditions were.

Tables 4.40 to 4.43 show the verification of the test results. The results showed that the predicted value were in good agreement with the experimental values.

With errors of less than 2.0%, it showed that the optimum conditions were valid and that the developed mathematical models can be used for the production of fiber reinforced HDPE composites for superior properties.

Table 4.40. Validations of optimum conditions for the compression molding of *ampelocissus cavicaulis* fiber reinforced composites

Fiber conditions	Temp. (^{0}C)	Time (Mins)	Pressure (Bar)	Tensile Strength (Mpa)		Error (%)
				Experimental values	Predicted values	
Untreated	171.31	5.0	28.19	23.38	23.59	0.90
NaoH treated	179.83	5.0	26.33	40.00	40.196	0.50
Acetic Anhydride treated	178.31	5.0	29.10	32.21	32.531	1.0
Nitric acid treated	171.32	5.0	29.33	25.34	25.65	1.21
Zinc chloride treated	170.11	5.0	29.12	30.13	30.3706	0.80

Table 4.41. Validation of optimum conditions for the compression molding of *Adenia lobata* fiber reinforced composites

Fiber conditions	Temp. (^{0}C)	Time (Mins)	Pressure (Bar)	Tensile Strength (Mpa)		Error (%)
				Experimental values	Predicted values	
Untreated	175.68	23.02	36.370	36.019	36.370	0.8
NaoH treated	172.89	26.77	41.0073	41.755	42.007	0.60
Acetic Anhydride treated	177.88	25.69	43.463	43.115	43.463	0.8
Nitric acid treated	171.86	28.51	40.83	40.463	40.83	0.9
Zinc chloride treated	171.31	26.34	38.18	37.989	38.18	0.5

Table 4.42. Validation of optimum conditions for the compression molding of *Morinda morindoides* fiber reinforced composites

Fiber conditions	Temp. (^{0}C)	Time (Mins)	Pressure (Bar)	Tensile Strength (Mpa)		Error (%)
				Experimental Values	Predicted values	
Untreated	179.31	5.0	49.11	55.99	56.211	0.4
NaoH treated	178.11	5.0	40.13	78.36	78.52	0.2
Acetic Anhydride treated	178.22	5.0	41.31	65.93	66.00	0.1
Nitric acid treated	177.31	5.0	39.38	60.51	61.31	1.3
Zinc chloride treated	178.11	5.0	42.11	57.65	58.11	0.8

Table 4.43. Validation of optimum conditions for the compression molding of *Ampelocissus leonensis* fiber reinforced composites

Fiber conditions	Temp. (^{0}C)	Time (Mins)	Pressure (Bar)	Tensile Strength (Mpa)		Error (%)
				Experimental values	Predicted values	
Untreated	150.00	5.0	20.0	20.75	21.00	1.2
NaoH treated	152.42	5.0	20.07	30.52	30.74	0.7
Acetic Anhydride treated	164.71	5.0	14.66	27.76	28.00	0.85
Nitric acid treated	155.17	5.0	26.25	25.28	25.31	0.1
Zinc chloride treated	158.11	5.0	12.55	23.89	24.11	0.9

4.13. Microstructural analysis of composites

Surface morphologies of the prepared composites were analyzed using scanning electron microscopy, and the functional groups were studied using Fourier transform infrared spectroscopy (FTIR).

4.13.1. Scanning Electron Microscopy Analysis of the Composites (SEM)

Scanning electron microscopic analysis (SEM) was used to examine the surface morphology of the fiber reinforced composites

Figures 4.51 and 4.52 show the micrograph of the untreated and treated surfaces of the composites samples, respectively. The microstructure of the composites treated with all the chemicals showed that there was reasonably uniform distribution of the fibers on the HDPE matrix. When it comes to using natural fibers as reinforcement in composite materials, many problems occur at the interface due to imperfect bonding. Interfacial properties of fiber/polymer composites are largely determined by the strength and nature of secondary interactions that are established across the phase boundary (Wang, 2004). Interfacial stress transfer is therefore limited to relatively weak dispersion forces (Joseph et al., 2000). A strong fiber matrix interface bond is critical for high mechanical properties of the composites. When manufacturing composite materials, compatibility of the matrix and fiber is also a problem. Therefore, treatment of fibers with chemicals modified the properties of the fiber and improved compatibility as can be seen from Fig.4.52. The untreated fiber composite (Fig. 4.51) presented a very poor dispersion of the fiber which also exhibited some fiber agglomeration, small fiber breakage and very poor adhesion between fiber and matrix. On the other hand, chemically treated fiber composites showed better fiber – matrix interaction. This is as a result of more uniform dispersion of fibers with the polymer matrix. The fibers were less agglomerated showing the presence of some fibers dissociated into a matrix polymer. It is important to mention that dissociation of the fiber into a matrix polymer may increase the surface area of the fiber, which contributes to a greater stress transfer solicitation (Wang, 2004).

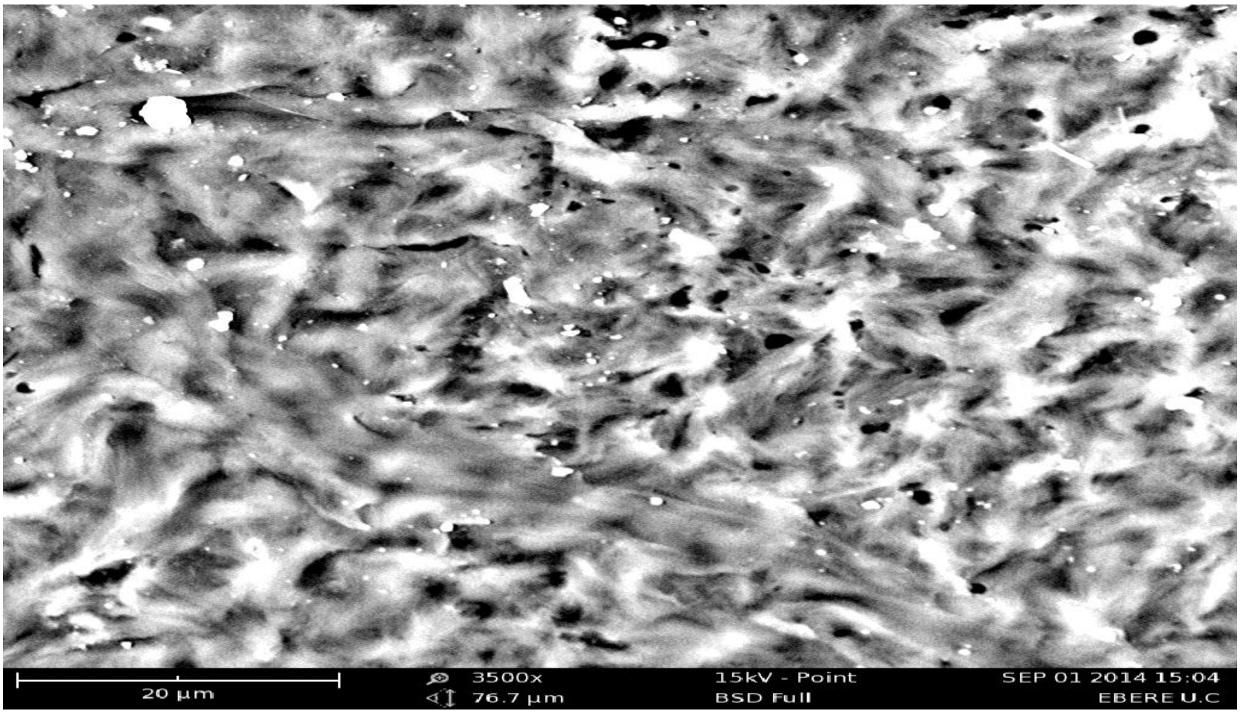
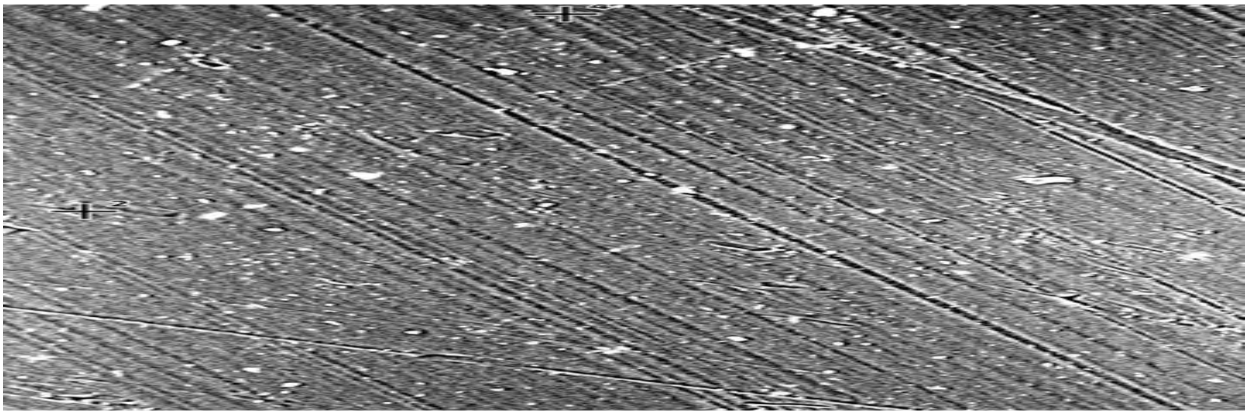


Fig 4.51. SEM of the untreated *ampelocissus cavicaulis* fiber reinforced composite.



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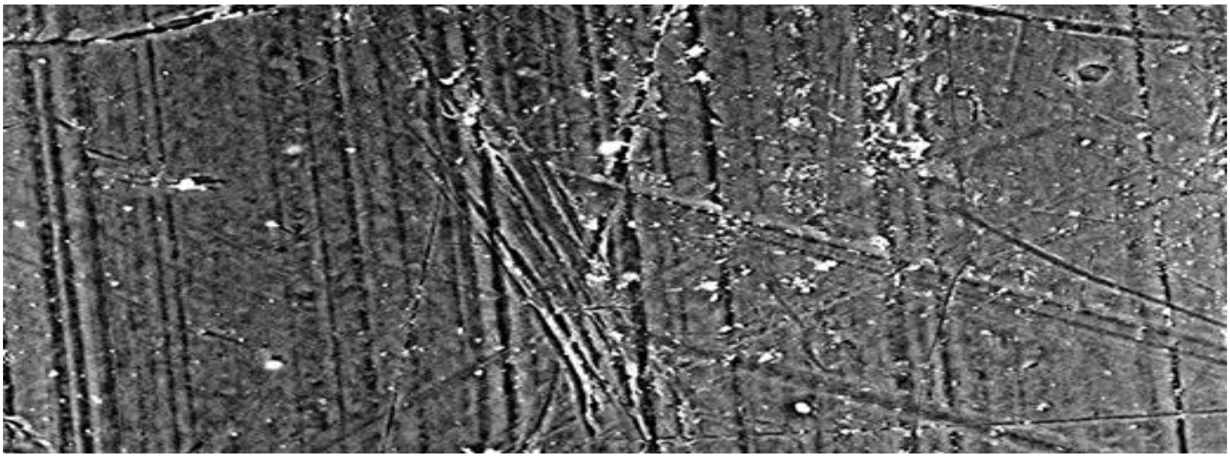

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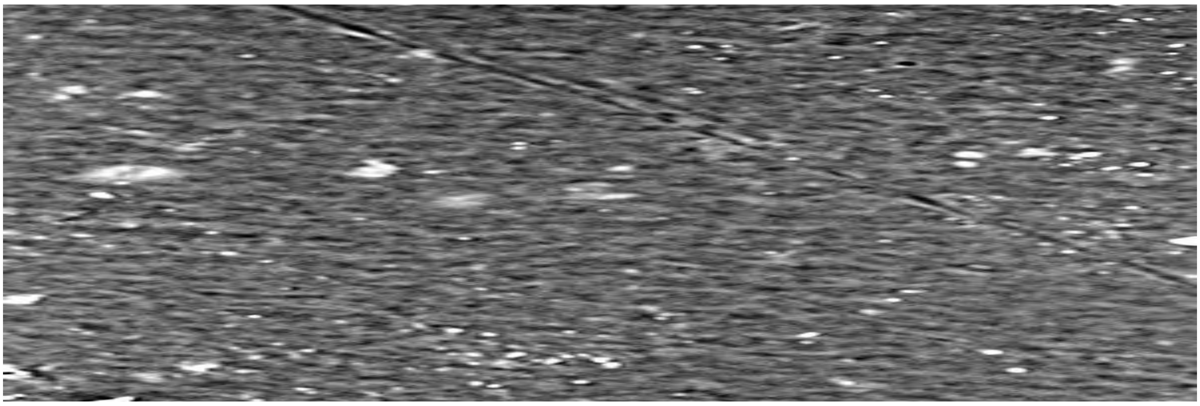
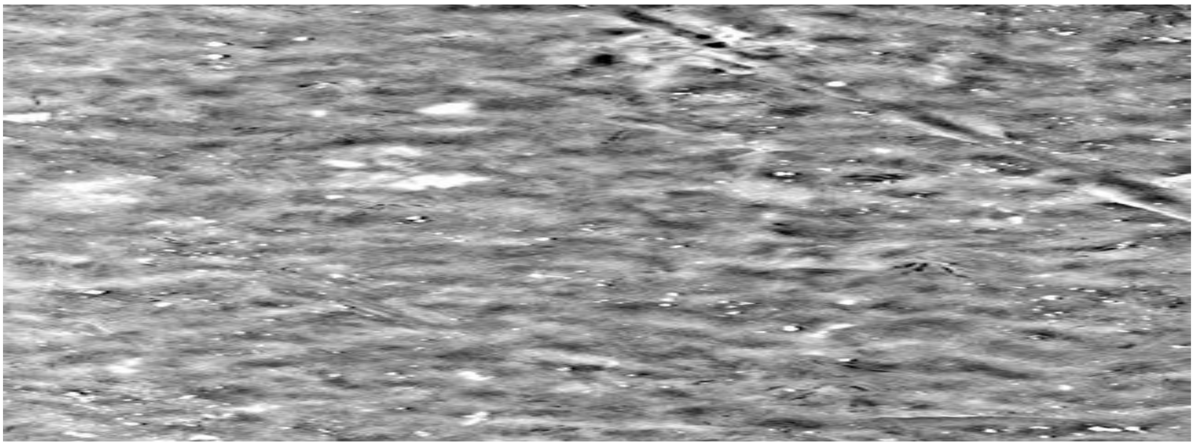


(c)

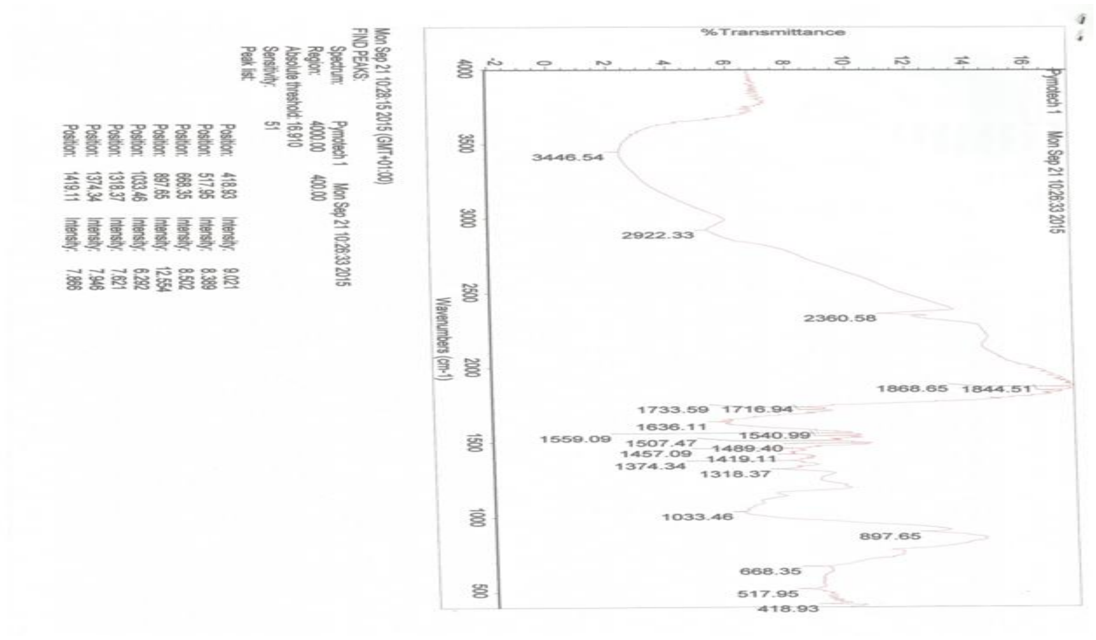


(d)

Fig 4.52. SEM of sodium hydroxide treated fiber reinforced composites (a) *Ampelocissus cavicaulis* (b) *Adenia lobata* (c) *Morinda morindoides* (d) *Ampelocissus leonensis*

4.13.2. Fourier Transformed Infrared Spectroscopy (FTIR)

FTIR analysis was used to characterize the natural fibers used with various treatments as well as the interpretation of the nature of adhesion between the fiber and the high density polyethylene. The FTIR spectra of all the untreated fibers used were shown on Fig. 4.53. All the spectra revealed broad and intense peak at 3400cm^{-1}. This band suggested hydrogen bonded O–H stretching vibration from the cellulose and lignin structure of the fibers (Oh et al., 2005; Kondo 1997). It was equally observed that one major band around 2923cm^{-1} was noticed on all the fibers except untreated *morinda morindoidies* fiber. This band was related to asymmetric and symmetric methyl and methylene stretching groups present in the spectra of all fiber components but most notably in the spectra for cellulose (Polelto et al., 2012; Popescu et al., 2009). This might be attributed to the higher extractive contents in these fibers since some compounds in organic extractives such as fatty acid methyl esters and phenoic acid methyl esters contain methyl and methylene groups (Yokoi et al., 2003; Ishiada et al., 2007; Meszaros et al., 2007). The absorption at 1724cm^{-1} present on all the untreated fibers except untreated *morinda morindoidies* can be assigned to the carbonyl stretching of the acetyl groups of hemicellulose. The peak around 1636cm^{-1} present on all the fibers represents the C = O of the hemicellulose (Yan et al.,, 2009). The bands at 1457cm^{-1}, 1374cm^{-1}, 1418cm^{-1}, 1269cm^{-1} and 1031cm^{-1} were characteristics of C – H, C – O deformation, bonding or stretching vibrations of many groups in lignin and carbohydrates (Yokoi et al., 2003; Ishiada et al., 2007; Meszaros et al., 2007). The peaks around 668cm^{-1}, 517cm^{-1} and 407cm^{-1} can be said to be originated from the β - glucosidic linkages between the sugar units in the hemicelluloses and celluloses (Bledzki et al., 2010).



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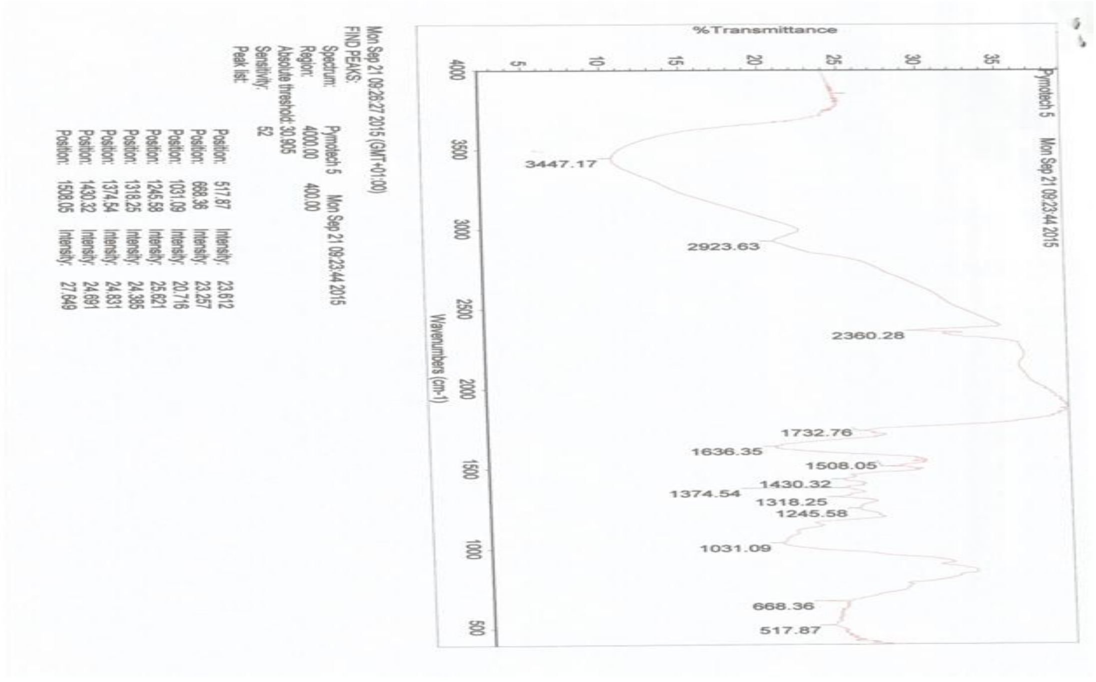
(a)



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                    Position:  1318.25   Intensity:  24.385
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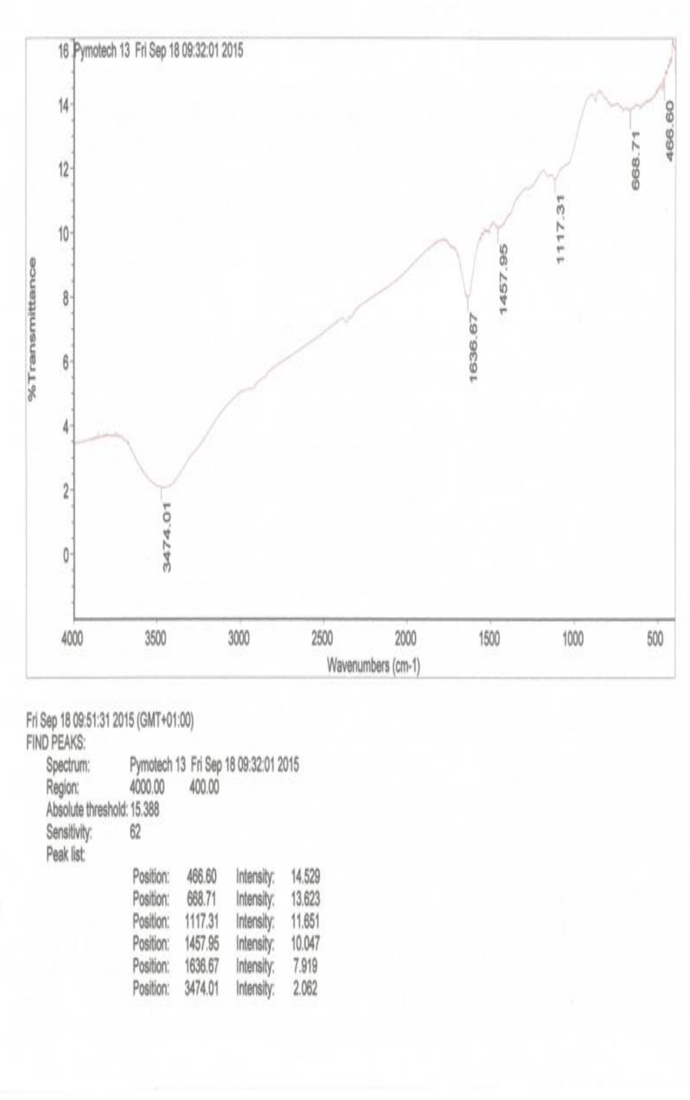
(b)



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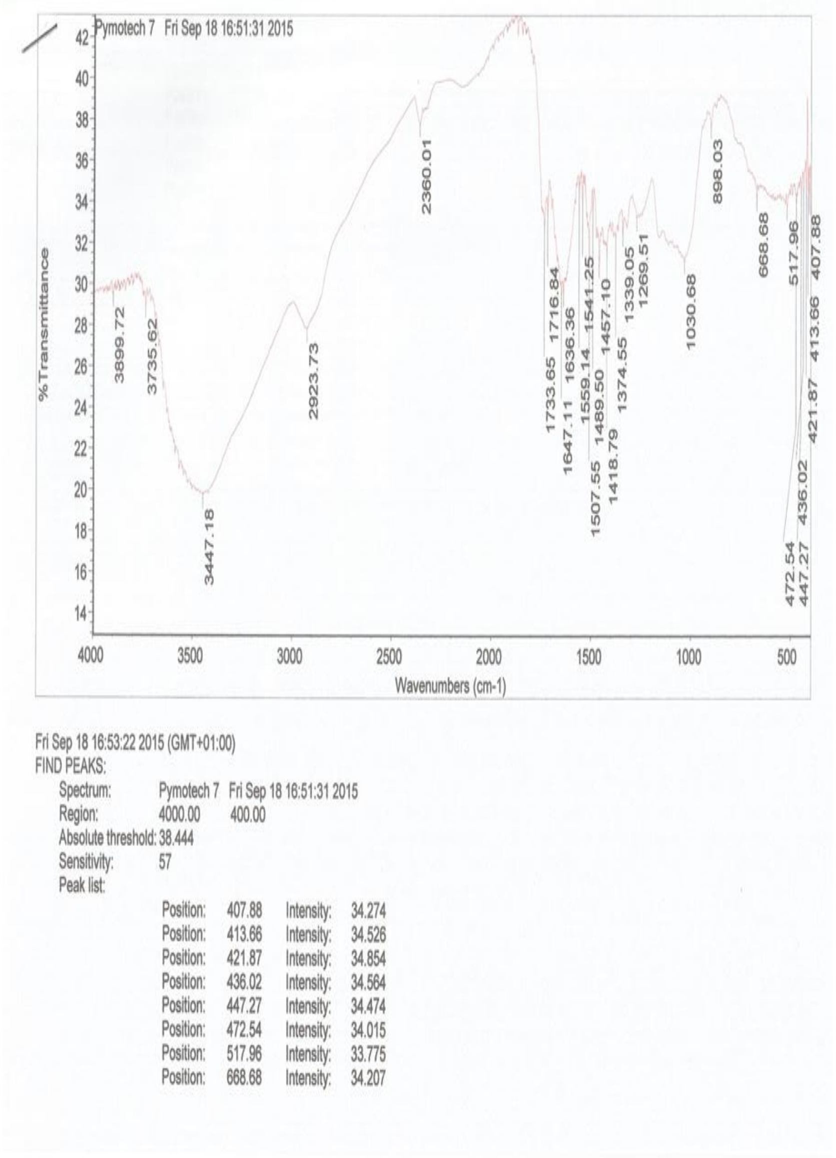
(c)



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                       Position:  447.27  Intensity:  34.474
                       Position:  472.54  Intensity:  34.015
                       Position:  517.96  Intensity:  33.775
                       Position:  668.68  Intensity:  34.207
```

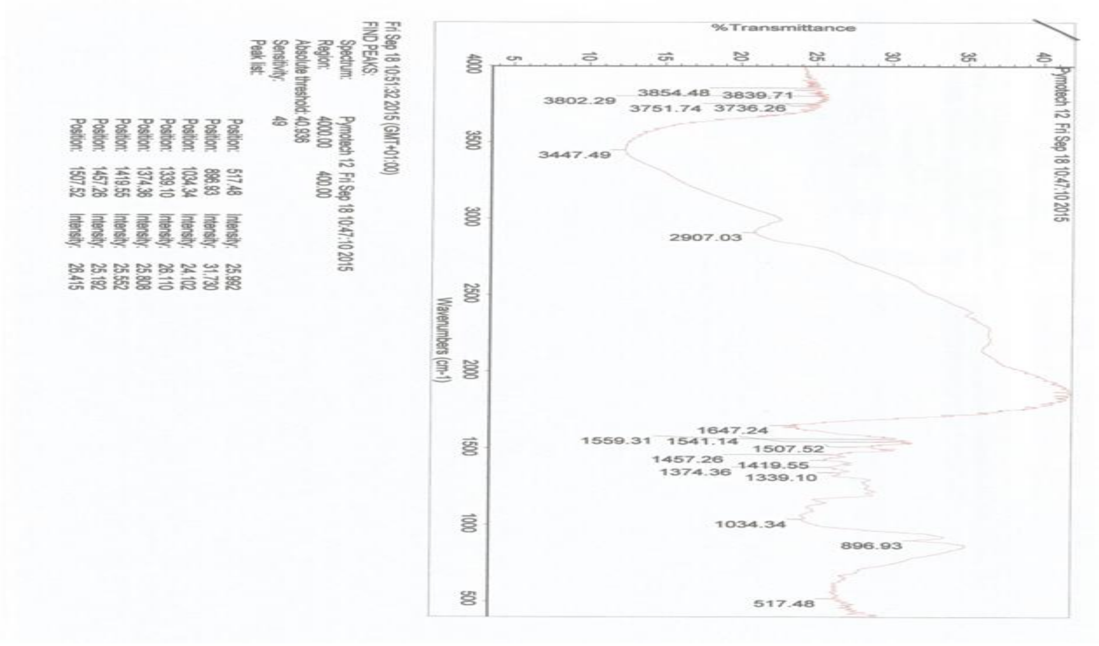
(d)

Fig. 4.53. FTIR analysis of untreated (a) *Ampelocissus cavicaulis (b) Adenia lobata (c) Morinda morindoidies (d) Ampelocissus leonensis* fibers.

After the treatment with sodium hydroxide (Figure 4.54), the bands at 1732cm^{-1} and 1245cm^{-1} attributed to the stretching vibrations of C = O and C – O groups respectively disappeared. These kinds of groups are present in lignin and hemicelluloses structures. The band at 1318cm^{-1} is related to the vibration V(C – O) of esters, ethers and phenols groups attributed mainly to a presence of waxes in the epithermal tissue (Herrera – Franco and Valadarez – Gorizalea 2005) and the disappearance of this band on the treated fibers results from the removal of those waxes.

After acetylation reaction, new acetyl groups were added to the cellulose as indicated by vibration at 1732cm^{-1} and 1734cm^{-1} (-C=O). The spectrum of unmodified cellulose showed an absorption peak at 1374cm^{-1} attributed to the –C-H bending vibration. After esterification, the added contribution of acetyl ($– C – CH_3$) stretching vibration intensified this absorption peak (Mokaloba and Batane, 2004).

The nitric acid used for the treatment tends to oxidize the hydroxyl groups from cellulose in the fiber surface to carboxyl groups giving the fiber a soft cationic potential (Rayes et al., 1998; Shukla and Pai, 2005). This oxidation was confirmed by the spectra of the nitric acid treated fiber at 1732cm^{-1} and 1734cm^{-1}. The carbonyl signal initially present was from lignin and hemicelluloses, after the treatment, the formation of carboxyl group was observed, whose axial vibration of C = O intensified the peak. Between 1370 and 1390cm^{-1}, the absorption peaks referred to a symmetrical and asymmetrical deformation of C – H in cellulose and hemicelluloses groups. In the spectra of the treated fibers, these bands were more accentuated. This can be an indicative of larger exposition of cellulose and hemicelluloses on the fiber surface (Brigida et al., 2011).



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                     Position:  1374.36   Intensity:   25.808
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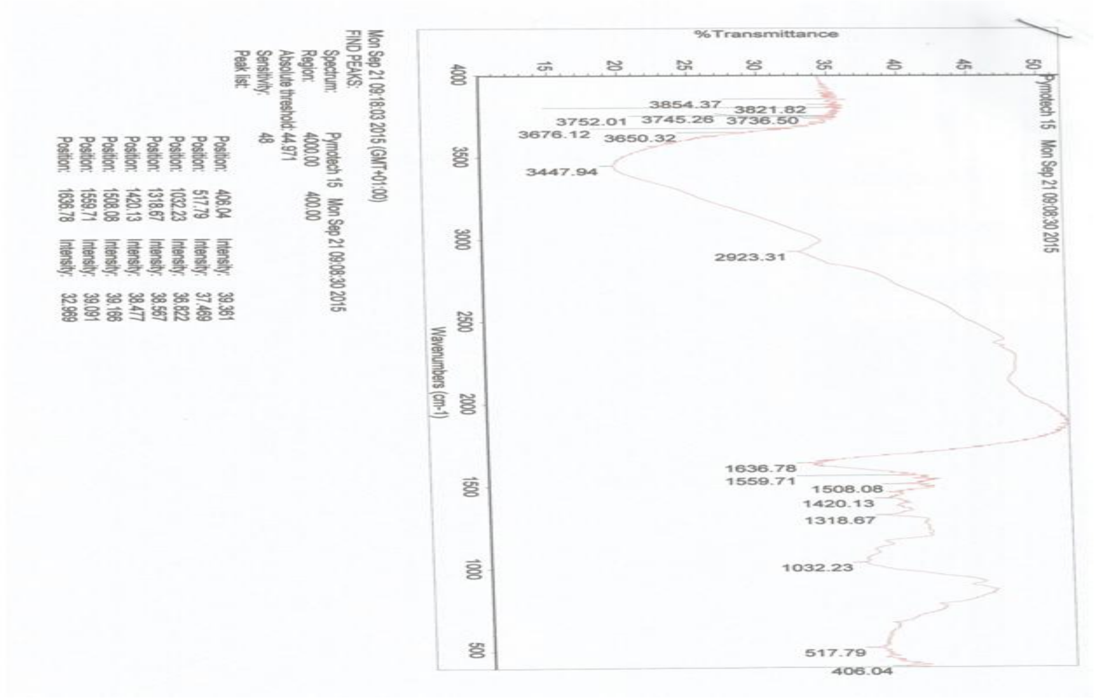
(a)



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                     Position:  1508.08   Intensity:   39.166
                     Position:  1559.71   Intensity:   39.091
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```
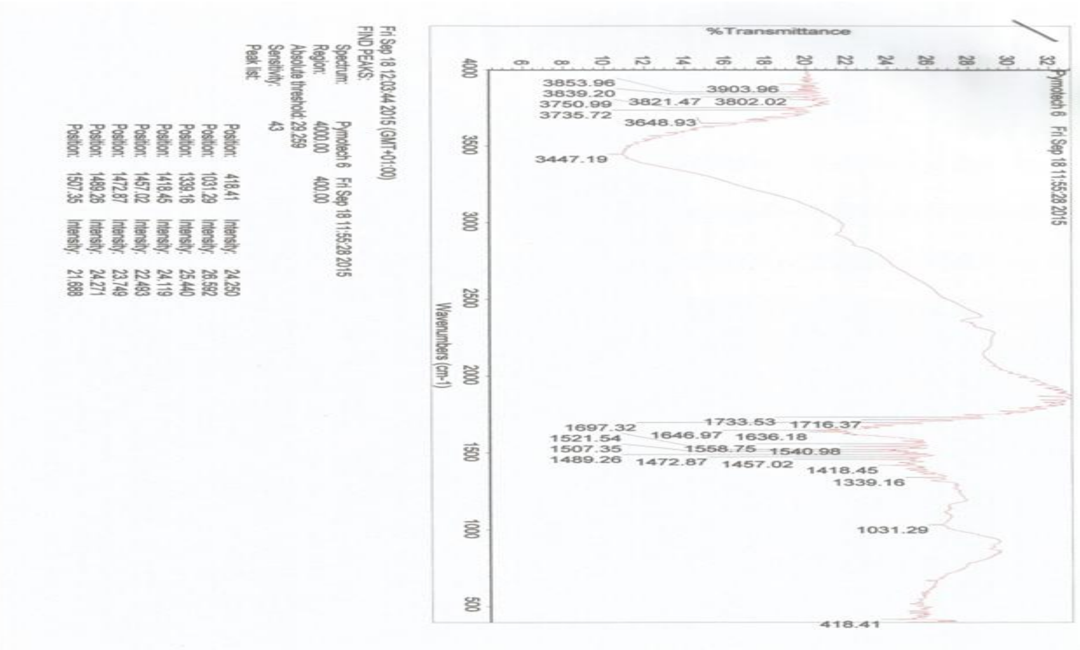
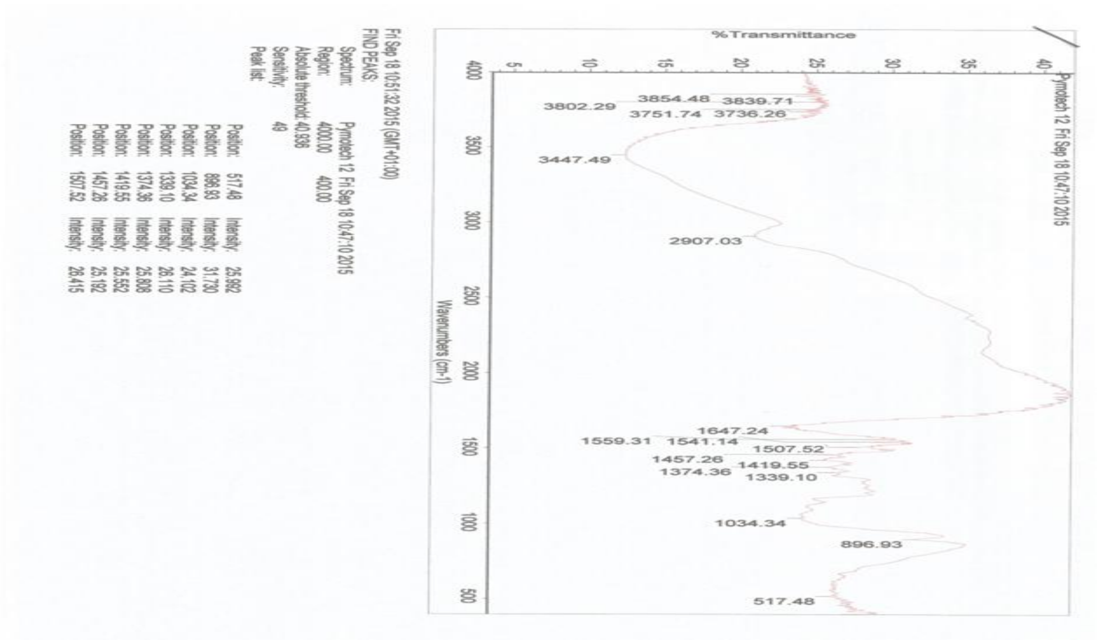

(b)



```
Fri Sep 18 12:03:44 2015 (GMT+01:00)
FIND PEAKS:
  Spectrum:              Pymotech 6  Fri Sep 18 11:55:28 2015
  Region:                4000.00     400.00
  Absolute threshold: 29.259
  Sensitivity:           43
  Peak list:
                         Position:   418.41   Intensity:   24.250
                         Position:  1031.29   Intensity:   26.592
                         Position:  1339.16   Intensity:   25.440
                         Position:  1418.45   Intensity:   24.119
                         Position:  1457.02   Intensity:   22.493
                         Position:  1472.87   Intensity:   23.749
                         Position:  1489.26   Intensity:   24.271
                         Position:  1507.35   Intensity:   21.688
```

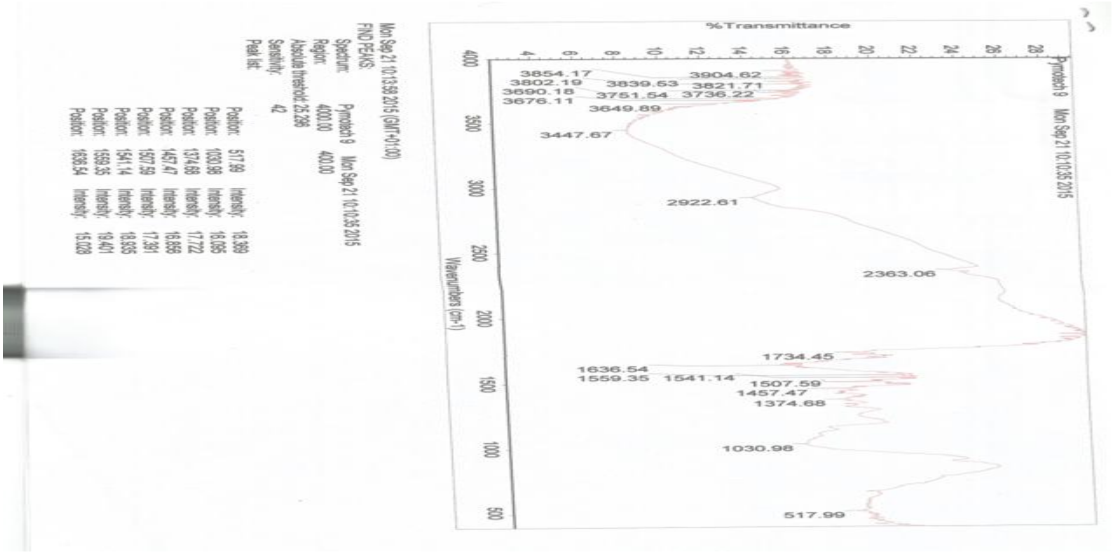
(c)



```
Fri Sep 18 10:51:32 2015 (GMT+01:00)
FIND PEAKS:
  Spectrum:              Pymotech 12  Fri Sep 18 10:47:10 2015
  Region:                4000.00      400.00
  Absolute threshold: 40.936
  Sensitivity:           49
  Peak list:
                         Position:   517.48   Intensity:   25.992
                         Position:   896.93   Intensity:   31.730
                         Position:  1034.34   Intensity:   24.102
                         Position:  1339.10   Intensity:   26.110
                         Position:  1374.36   Intensity:   25.808
                         Position:  1419.55   Intensity:   25.552
                         Position:  1457.26   Intensity:   25.192
                         Position:  1507.52   Intensity:   26.415
```

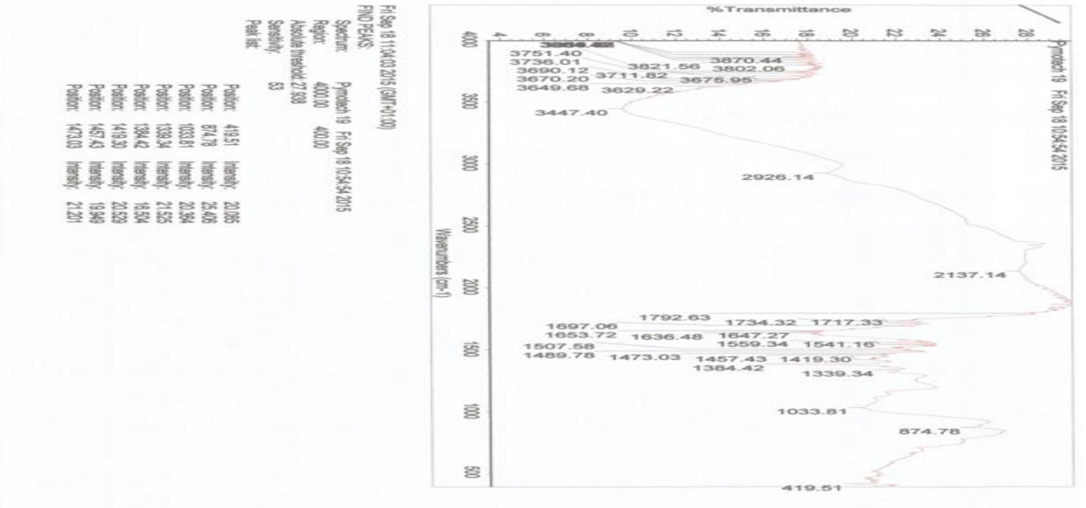
(d)

Fig. 4.54. FTIR analysis of sodium hydroxide treated fibers (a) *Ampelocissus cavicaulis, (b) Adenia lobata (c) Morinda morindoidied (d) Ampelocissus leonensis*



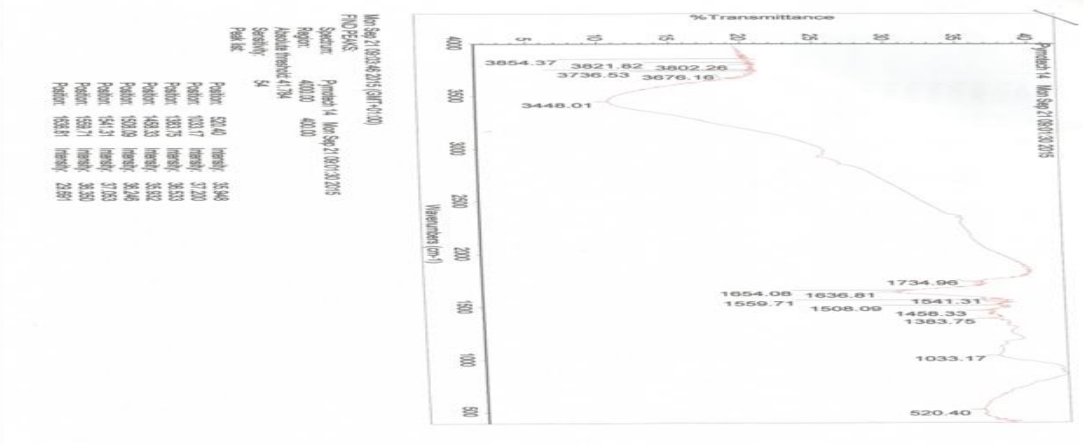
```
Mon Sep 21 10:13:58 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 9  Mon Sep 21 10:10:35 2015
 Region:            4000.00     400.00
 Absolute threshold: 25.296
 Sensitivity:       42
 Peak list:
             Position:  517.99   Intensity:  18.369
             Position:  1030.98  Intensity:  16.095
             Position:  1374.68  Intensity:  17.722
             Position:  1457.47  Intensity:  16.856
             Position:  1507.59  Intensity:  17.391
             Position:  1541.14  Intensity:  18.935
             Position:  1559.35  Intensity:  19.401
             Position:  1636.54  Intensity:  15.028
```

(a)



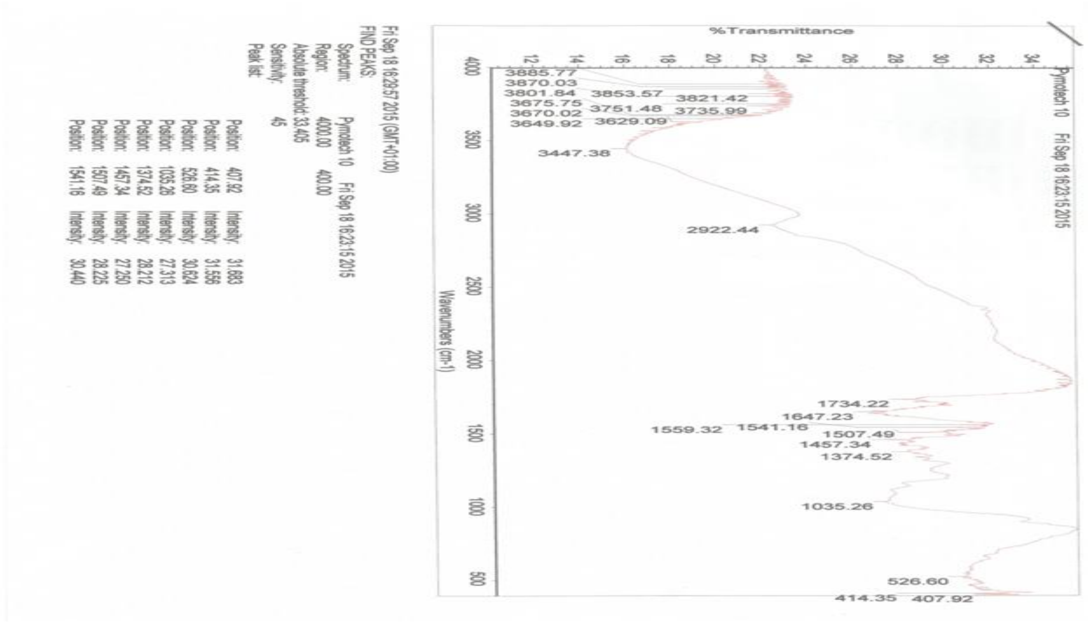
```
Fri Sep 18 11:04:03 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 19  Fri Sep 18 10:54:54 2015
 Region:            4000.00      400.00
 Absolute threshold: 27.938
 Sensitivity:       53
 Peak list:
             Position:  419.51   Intensity:  20.085
             Position:  874.78   Intensity:  25.406
             Position:  1033.81  Intensity:  20.384
             Position:  1339.34  Intensity:  21.525
             Position:  1384.42  Intensity:  16.504
             Position:  1419.30  Intensity:  20.529
             Position:  1457.43  Intensity:  19.949
             Position:  1473.03  Intensity:  21.201
```

(b)



```
Mon Sep 21 09:03:46 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 14  Mon Sep 21 09:01:30 2015
 Region:            4000.00      400.00
 Absolute threshold: 41.784
 Sensitivity:       54
 Peak list:
             Position:  520.40   Intensity:  35.948
             Position:  1033.17  Intensity:  37.200
             Position:  1383.75  Intensity:  36.533
             Position:  1458.33  Intensity:  35.932
             Position:  1508.09  Intensity:  36.246
             Position:  1541.31  Intensity:  37.053
             Position:  1559.71  Intensity:  36.350
             Position:  1636.81  Intensity:  29.691
```

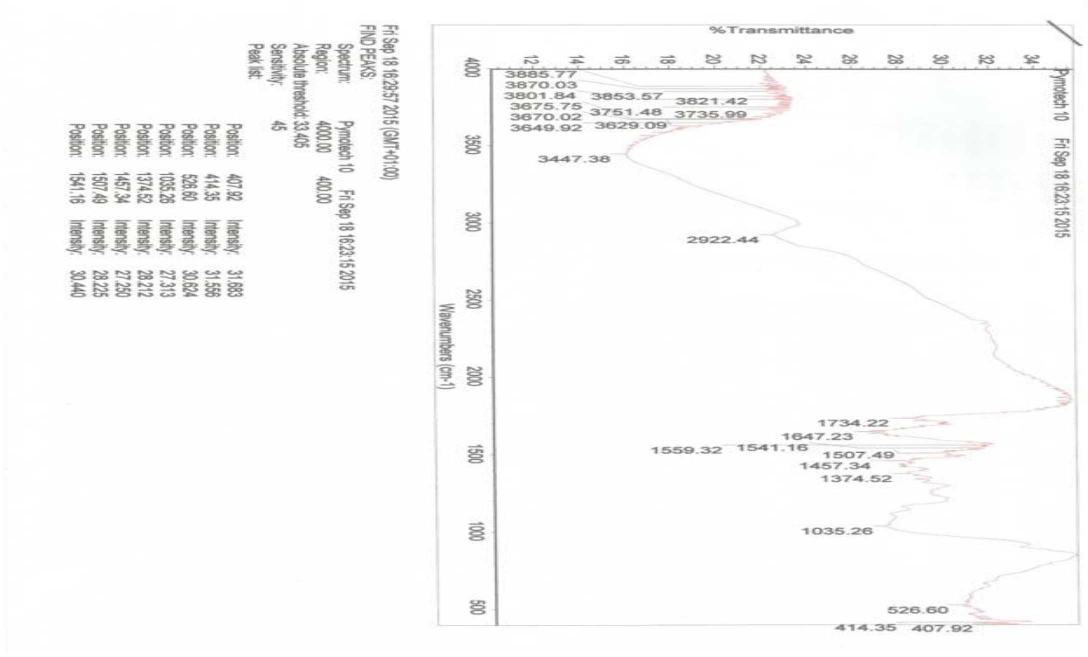
(c)



```
Fri Sep 18 16:29:57 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 10    Fri Sep 18 16:23:15 2015
 Region:            4000.00        400.00
 Absolute threshold: 33.405
 Sensitivity:       45
 Peak list:
                    Position:   407.92   Intensity:  31.683
                    Position:   414.35   Intensity:  31.556
                    Position:   526.60   Intensity:  30.624
                    Position:  1035.26   Intensity:  27.313
                    Position:  1374.52   Intensity:  28.212
                    Position:  1457.34   Intensity:  27.250
                    Position:  1507.49   Intensity:  28.225
                    Position:  1541.16   Intensity:  30.440
```

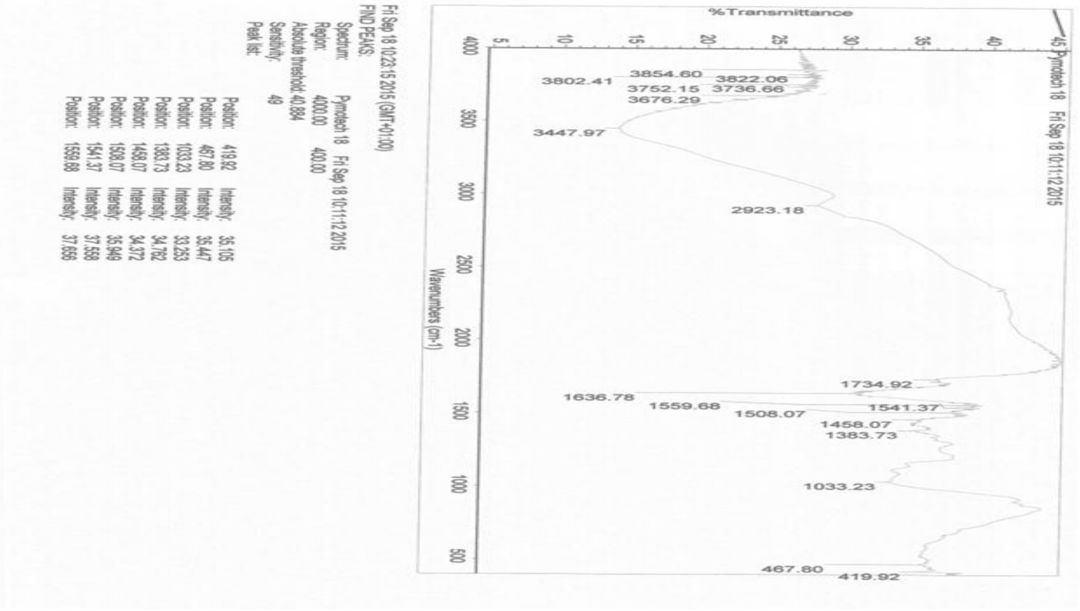
(d)

Fig. 4.55. FTIR analysis of acetic anhydride treated fibers (a) *Ampelocissus cavicaulis (b) Adenia lobata (c) Morinda morindoidies (d) Ampelocissus leonensis*.



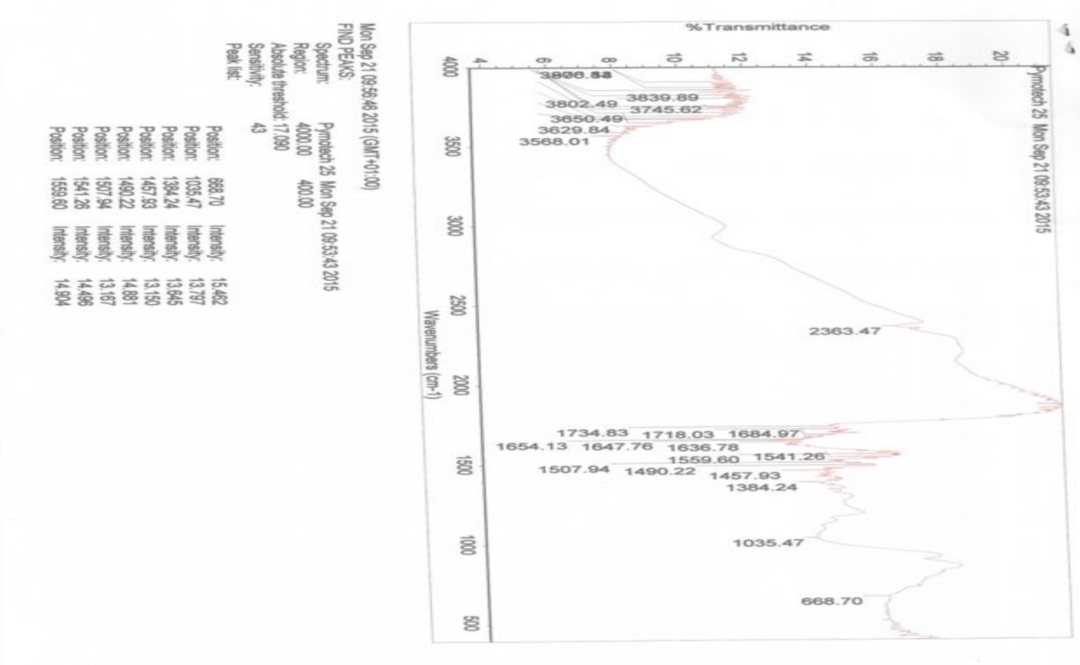
```
Fri Sep 18 16:29:57 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 10    Fri Sep 18 16:23:15 2015
 Region:            4000.00        400.00
 Absolute threshold: 33.405
 Sensitivity:       45
 Peak list:
                    Position:   407.92   Intensity:  31.683
                    Position:   414.35   Intensity:  31.556
                    Position:   526.60   Intensity:  30.624
                    Position:  1035.26   Intensity:  27.313
                    Position:  1374.52   Intensity:  28.212
                    Position:  1457.34   Intensity:  27.250
                    Position:  1507.49   Intensity:  28.225
                    Position:  1541.16   Intensity:  30.440
```

(a)



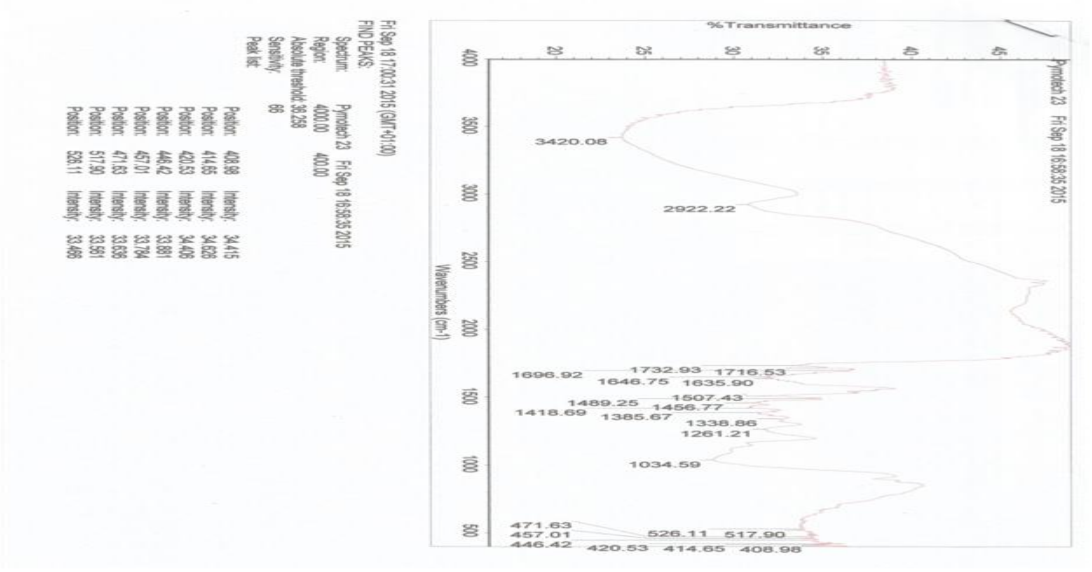
```
Fri Sep 18 10:23:15 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 18  Fri Sep 18 10:11:12 2015
 Region:            4000.00      400.00
 Absolute threshold: 40.884
 Sensitivity:       49
 Peak list:
          Position:   419.92   Intensity:  35.105
          Position:   467.80   Intensity:  35.447
          Position:  1033.23   Intensity:  33.253
          Position:  1383.73   Intensity:  34.762
          Position:  1458.07   Intensity:  34.372
          Position:  1508.07   Intensity:  35.949
          Position:  1541.37   Intensity:  37.558
          Position:  1559.68   Intensity:  37.656
```

(b)



```
Mon Sep 21 09:56:46 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:          Pymotech 25  Mon Sep 21 09:53:43 2015
 Region:            4000.00      400.00
 Absolute threshold: 17.090
 Sensitivity:       43
 Peak list:
          Position:   668.70   Intensity:  15.462
          Position:  1035.47   Intensity:  13.797
          Position:  1384.24   Intensity:  13.645
          Position:  1457.93   Intensity:  13.150
          Position:  1490.22   Intensity:  14.881
          Position:  1507.94   Intensity:  13.167
          Position:  1541.26   Intensity:  14.496
          Position:  1559.60   Intensity:  14.904
```

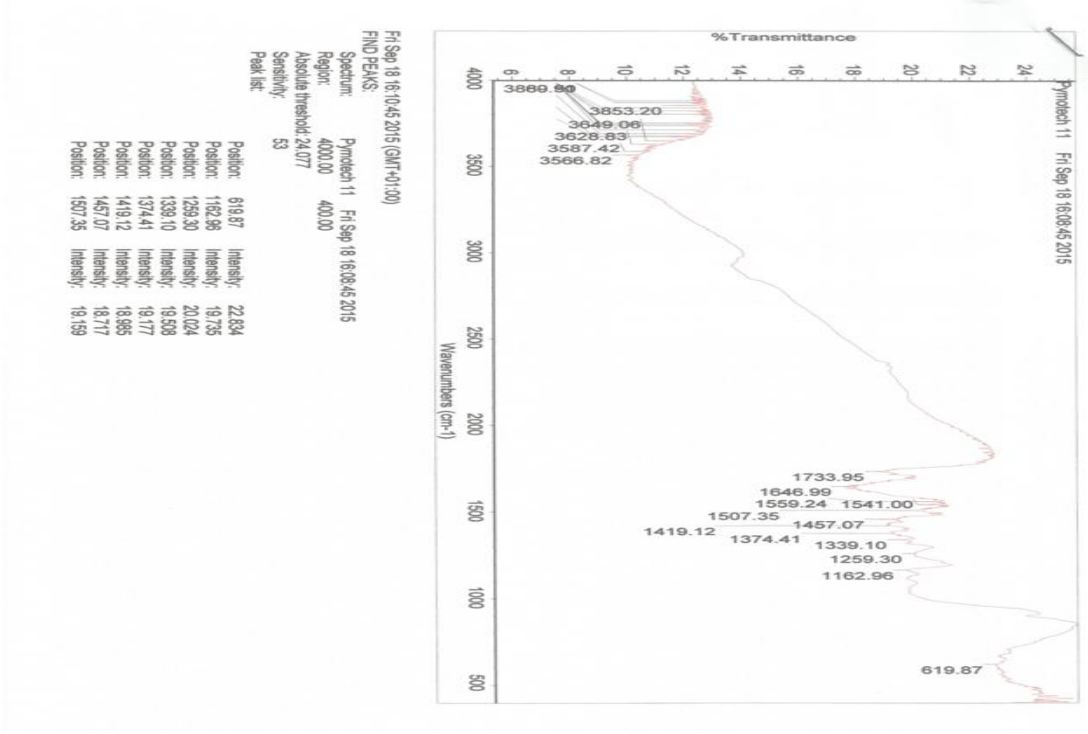
(c)



```
Fri Sep 18 17:00:31 2015 (GMT+01:00)
FIND PEAKS:
   Spectrum:        Pymotech 23   Fri Sep 18 16:58:35 2015
   Region:          4000.00     400.00
   Absolute threshold: 36.258
   Sensitivity:     66
   Peak list:
                    Position:  408.98   Intensity:  34.415
                    Position:  414.65   Intensity:  34.626
                    Position:  420.53   Intensity:  34.406
                    Position:  446.42   Intensity:  33.881
                    Position:  457.01   Intensity:  33.784
                    Position:  471.63   Intensity:  33.636
                    Position:  517.90   Intensity:  33.561
                    Position:  526.11   Intensity:  33.466
```

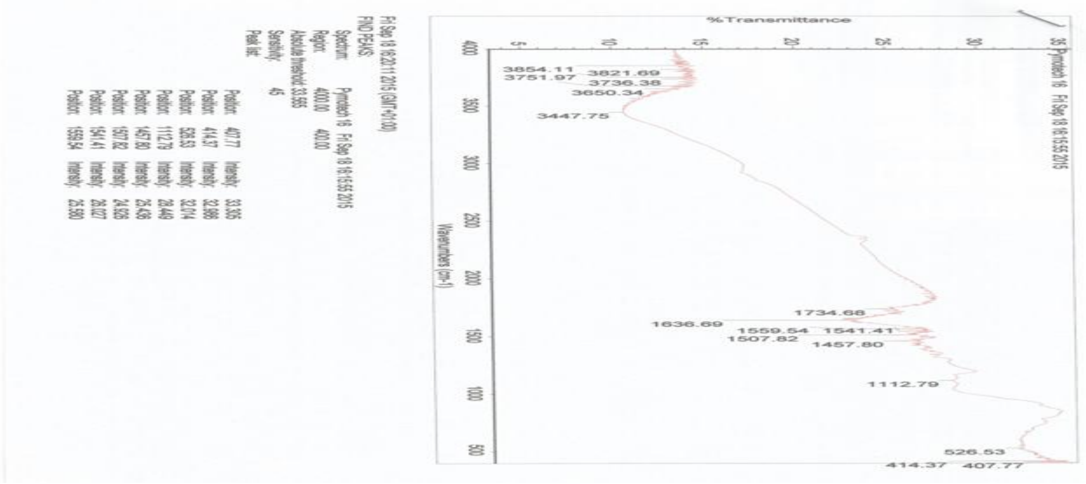
(d)

Fig. 4.56. FTIR analysis of nitric acid treated fibers (a) *Ampelocissus cavicaulis (b) Adenia lobata (c) Morinda morindoidies (d) Ampelocissus leonensis*.

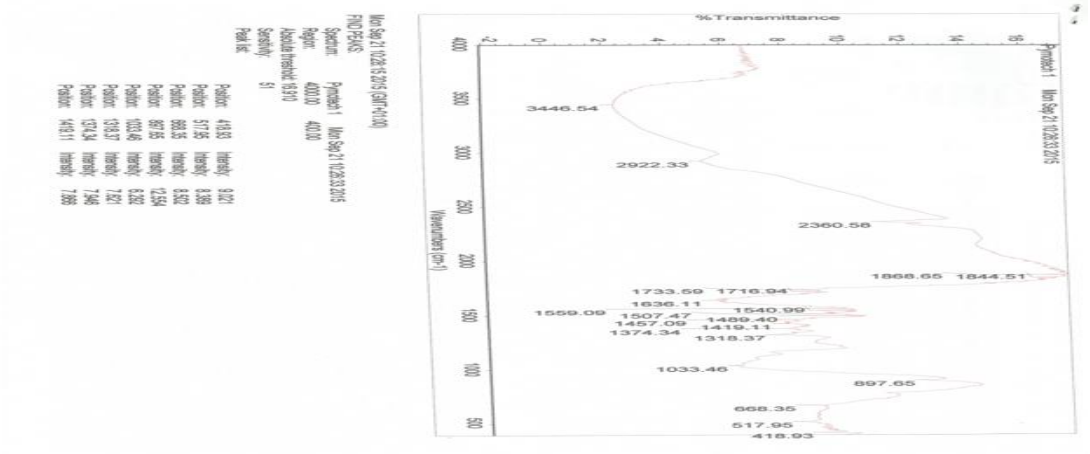


```
Fri Sep 18 16:10:45 2015 (GMT+01:00)
FIND PEAKS:
   Spectrum:        Pymotech 11   Fri Sep 18 16:08:45 2015
   Region:          4000.00     400.00
   Absolute threshold: 24.077
   Sensitivity:     53
   Peak list:
                    Position:  619.87   Intensity:  22.834
                    Position:  1162.96  Intensity:  19.735
                    Position:  1259.30  Intensity:  20.024
                    Position:  1339.10  Intensity:  19.508
                    Position:  1374.41  Intensity:  19.177
                    Position:  1419.12  Intensity:  18.985
                    Position:  1457.07  Intensity:  18.717
                    Position:  1507.35  Intensity:  19.159
```

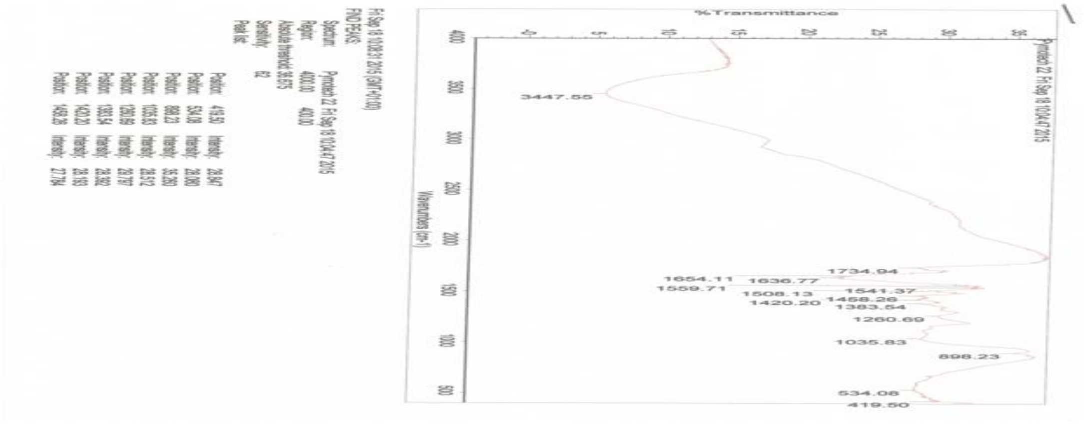
(a)



(b)



(c)



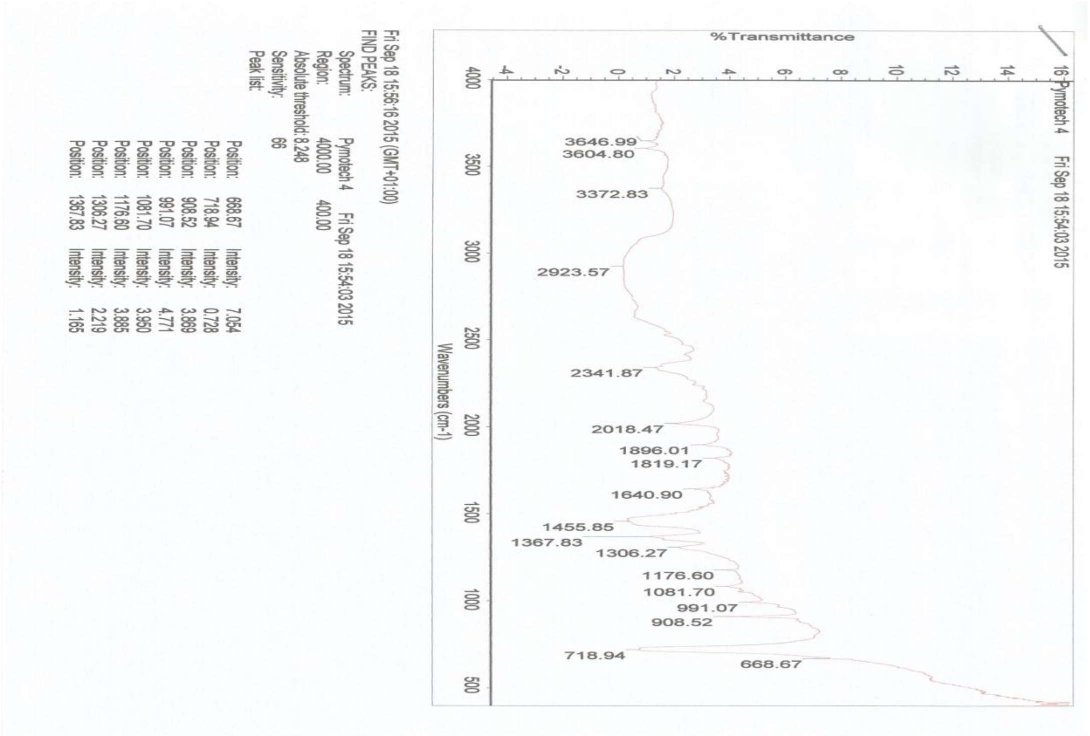
(d)

Fig. 4.57. FTIR analysis of zinc chloride treated fibers (a) *Ampelocissus cavicaulis (b) Adenia lobata (c) Morinda morindoidies (d) Ampelocissus leonensis*.

FTIR was further used to investigate the interfacial properties of natural fiber composites. Figure 4.58 displayed HDPE and composites containing 40% natural fibers. The spectra revealed that the highest absorbance values corresponded to the untreated composites and the lowest value to that of treated fiber reinforced composites. This can be attributed to the removal of lignin and hemicelluloses that absorb at high wavelength as a result of chemical treatments. HDPE spectra (Fig. 4.58(a)) showed peaks ranging from 2923 to 3046.99cm^{-1} and 1455.85cm^{-1} corresponding to CH_2 or CH_3 vibrations (Ismat et al., 2015). The bands at 1081.70cm^{-1} and 1176.60 may be due to stretching of CH – CH_2 and vibration of the rocking of –CH_3 and –CH_2 and due to the isotactic band respectively (Khan et al., 2010; Wachara Wichanant and Siripattanasak 2013; Srinivasa and Manon Mani 2013; Shavanat 2011; Sclavons et al., 2005). The FTIR spectrum of the fiber reinforced composites showed the presence of the characteristic band of the C = C group in the region of 1600 – 1700cm^{-1} and C – O stretching in the region of 1000 – 1300cm^{-1} as the composites contain sodium hydroxide treated fibers as reinforcement.

Absence of carbonyl (C = O) peak (1740 – 1720cm^{-1}) which represents hemicelluloses was the evidence that hemicelluloses were removed from the fiber surfaces during the sodium hydroxide treatment. Peaks observed at 690 – 900cm^{-1} are due to C – H bending. Weak peaks for C-O-H bending due to presence of ester and C = C stretching due to presence of aromatic rings of lignin appear between 1375 and 1600cm^{-1} (Ismat et al., 2015). Bands from 3200 to 3800cm^{-1} were due to the chemically absorbed water and also due to the stretching vibration of hydroxyl groups present in the composite containing cellulose (Sclavons et al., 2000; Begum et al., 2013). Another absorbance peak around 2850 – 3000cm^{-1} is associated with the –CH, -CH_2, or –CH_3 stretching (Syafri et al., 2011).

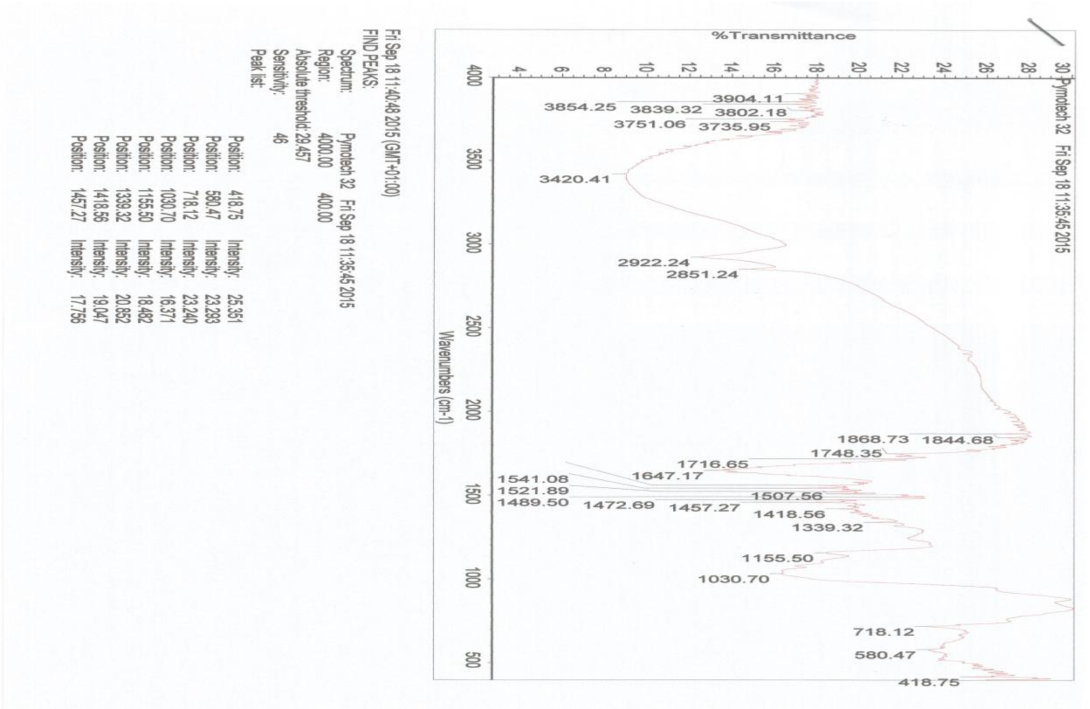
According to the spectra of the composites, the interaction between the fibers and the HDPE matrix could be a physical interaction as there were no new bands or any significant shift compared to the HDPE spectrum (Samira et al., 2012).



Fri Sep 18 15:56:16 2015 (GMT+01:00)
FIND PEAKS:
Spectrum: Pymotech 4 Fri Sep 18 15:54:03 2015
Region: 4000.00 400.00
Absolute threshold: 8.248
Sensitivity: 66
Peak list:

Position:	668.67	Intensity:	7.054
Position:	718.94	Intensity:	0.728
Position:	908.52	Intensity:	3.869
Position:	991.07	Intensity:	4.771
Position:	1081.70	Intensity:	3.950
Position:	1176.60	Intensity:	3.885
Position:	1306.27	Intensity:	2.219
Position:	1367.83	Intensity:	1.165

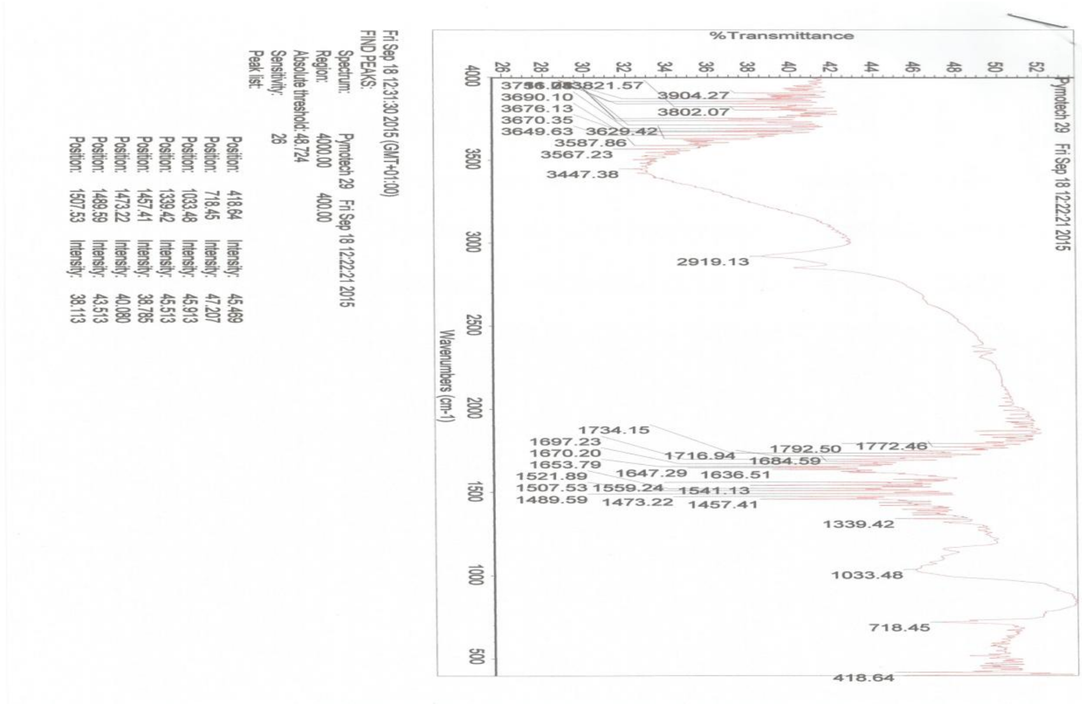
(a)



Fri Sep 18 11:40:48 2015 (GMT+01:00)
FIND PEAKS:
Spectrum: Pymotech 32 Fri Sep 18 11:35:45 2015
Region: 4000.00 400.00
Absolute threshold: 29.457
Sensitivity: 46
Peak list:

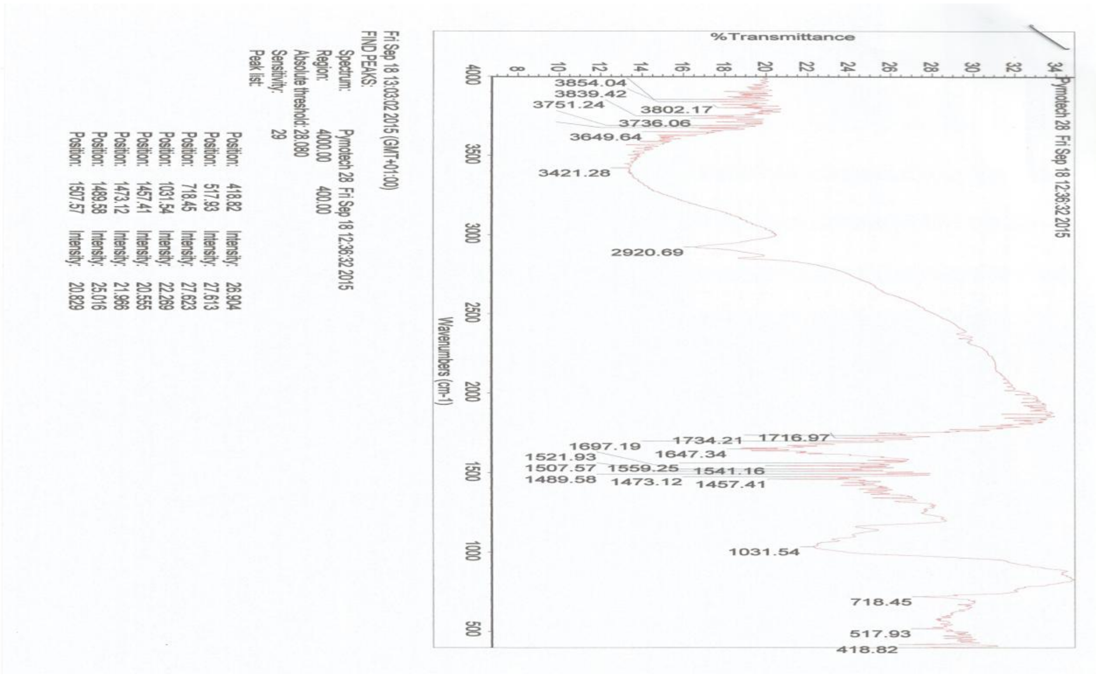
Position:	418.75	Intensity:	25.351
Position:	580.47	Intensity:	23.293
Position:	718.12	Intensity:	23.240
Position:	1030.70	Intensity:	16.371
Position:	1155.50	Intensity:	18.462
Position:	1339.32	Intensity:	20.852
Position:	1418.56	Intensity:	19.047
Position:	1457.27	Intensity:	17.756

(b)



```
Fri Sep 18 12:31:30 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:        Pymotech 29   Fri Sep 18 12:22:21 2015
 Region:          4000.00      400.00
 Absolute threshold: 48.724
 Sensitivity:     26
 Peak list:
            Position:    418.64   Intensity:   45.469
            Position:    718.45   Intensity:   47.207
            Position:   1033.48   Intensity:   45.913
            Position:   1339.42   Intensity:   45.513
            Position:   1457.41   Intensity:   38.785
            Position:   1473.22   Intensity:   40.080
            Position:   1489.59   Intensity:   43.513
            Position:   1507.53   Intensity:   38.113
```

(c)



```
Fri Sep 18 13:03:02 2015 (GMT+01:00)
FIND PEAKS:
 Spectrum:        Pymotech 28   Fri Sep 18 12:36:32 2015
 Region:          4000.00      400.00
 Absolute threshold: 28.080
 Sensitivity:     29
 Peak list:
            Position:    418.82   Intensity:   26.904
            Position:    517.93   Intensity:   27.613
            Position:    718.45   Intensity:   27.623
            Position:   1031.54   Intensity:   22.269
            Position:   1457.41   Intensity:   20.555
            Position:   1473.12   Intensity:   21.966
            Position:   1489.58   Intensity:   25.016
            Position:   1507.57   Intensity:   20.829
```

(d)

Fig. 4.58. FTIR analysis of composites (a) HDPE (b) untreated *Ampelocissus cavicaulis* (c) Sodium hydroxide treated *Ampelocissus cavicaulis* (d) Acetic anhydride treated *Ampelocissus cavicaulis* fiber reinforced composites

CHAPTER FIVE

CONCLUSIONS AND RECOMMENDATION

5.1. Conclusion

The use of natural fibers for reinforcing thermoplastic has been investigated by several researchers. However, no work has been done using the four selected natural fibers. Major works have not been done using natural fibers with thermoplastics to develop compression-molded composites. Thus, the overall goal of this research was to develop compression molded composites using the four natural fibers treated with four different chemicals as reinforcement to HDPE and to optimize the process conditions. This research goal has been achieved.

The conclusions that were drawn from the results are as follows;

- Quadratic model were developed for the chemical treatment process for all the four fibers and the optimum conditions developed were verified with little error of less than 2% for all the fibers.
- It was established that alkaline treatment was more effective in removing the waxy encrusting substances from the surface of the fiber, followed by acetic anhydride, nitric acid and lastly zinc chloride on all the fibers except on *Ampelocissus cavicaulis* fiber where zinc chloride was more effective than nitric acid. The surface morphology observed using scanning electron microscopy (SEM) showed that the chemical treatments were successful in removing surface contaminants as well as waxy substances on the surface of the fibers.
- Quadratic models were developed for all the fibers treated with four chemicals for the compounding process and the optimum conditions established were verified with little error of less than one percent.
- It was established that the tensile strength of compounded composite was affected by increase in temperature, time and rotation speed .
- The developed models for the compression molding process for all the fiber reinforced composites were quadratic models and the optimum conditions derived for compression molding process were validated with error of less than 1.5%.

- It was established that the tensile strengths of the composites were better than the tensile strength of the polymer.
- Increased in temperature, pressure, fiber length, fiber loading and compression time increased the tensile strength of the composite to a point beyond which there was decrease in tensile strength.
- Chemically treated fiber reinforced composites exhibited better mechanical and physical properties compared to the untreated fiber reinforced composite

  In this study, it was found that the natural fibers used can serve as reinforcement for HDPE resin. The results of the HDPE based composites were found promising and can be used as compression molded products for commercial purposes.

5.2. Recommendations

Natural fiber reinforced HDPE composite can be successfully manufactured by compression molding process. Compounding process with combination of compression molding technology can be used to develop uniformly distributed fiber reinforced HDPE composites. Based on this study, several recommendations are made for further studies.

- From the results of these experiments, it is quite evident that natural fibers have a very promising future and should be used as a substitute for synthetic fibers. Surface modifications of hydrophilic natural fibers are recommended so as to achieve superior fiber/matrix interface bonding and ultimately enhance the physio-mechanical properties of composites.
- The use of coupling agents (Malaic Anhydride etc) to promote natural fibre-matrix adhesion is recommended.
- Studies on thermal properties (thermal conductivity) are suggested for further characterization of the compression molded product.
- Only one resin was used in this study, effect of other types of resins can be investigated and compared.
- Comparative studies on the cost effectiveness of natural fiber reinforced high density polyethylene composites are recommended for future to evaluate the commercialization and market possibilities of the developed product.

5.3. Contribution to knowledge

- In carrying out the research, efforts were successfully made to add values to the natural fibers (*Ampelocissus cavicaulis, Adenia lobata, Morinda morindoidies and Ampelocissus leonensis*) by using them to produce composites. These fibers have not been known to be used by any researcher for purposes of reinforcing thermoplastic polymer (s) for composite production, this research successfully achieved that.
- Data bank has been generated for further use in compression molding process.

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APPENDIXES

APPENDIX A

EFFECT OF CHEMICAL TREATMENT ON THE WATER ABSORPTION OF FIBERS

	Ampelocissus leonensis	*Ampelocissus cavicaulis*	*Adenia lobata*	*Morinda morinda*
Untreated	12.5	10.3	9	7.65
Sodium hydroxide treated	6.8	6.3	5	3.9
Acetic anhydride treated	7.1	6.8	6	5.2
Nitric acid treated	8.2	7.5	6.3	6.1
Zinc chloride treated	9.1	8	7.1	6.8

APPENDIX B

EFFECT OF CHEMICAL TREATMENT ON THE MECHANICAL PROPERTIES OF THE FIBER

I. EFFECT OF CHEMICAL STRENGTH

Chemical type	Chemical Strength (%)	Tensile	Strength	(Mpa)	
		Ampelocissus Cavicaulis (Nwogbe)	*Adenia Lobata (Usoro)*	*Morinda Morindoides (Ogbuebo)*	*Ampelocissus Leonensis (Okpaowoko)*
Untreated	0.00	238.28	588.94	1207.79	67.65
NaOH	6.0	365.612	676.29	1623.074	123.346
Acetic anhydride	14.0	341.944	674.16	1522.714	103.745
Nitric acid	6.0	298.756	595.85	1408.48	89.540
Zinc chloride	3.0	330.874	590.73	1277.0	84.590

II. EFFECT OF CONCENTRATION OF NaOH

Chemical concentration (%)	Tensile	Strength	(Mpa)	
	Ampelocissus Cavicalulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
Untreated	238.30	588.94	1201.79	67.65
3.0	350.88	652.30	1321.00	91.422
6.0	365.7.	676.29	1411.09	148.014
9.0	320.168	660.25	1520.28	120.168
12.0	246.64	610.00	1679.20	110.32
15.0	240.08	600.29	1720.12	95.22
18.0	176.136	590.11	1520.19	80.52
21.0	168.784	518.21	1432.27	56.22

III. EFFECT OF NaOH PRETREATMENT TIME

Tensile strength (Mpa)

Time (Minutes)	*Ampelocissus cavicaulis* (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
0.00	238.30	588.94	1201.79	67.65
10.09	254.26	599.32	1352.71	71.422
20.00	281.69	652.11	1581.93	76.392
30.0	314.06	610.11	1599.31	95.35

40.0	330.94	667.32	1687.32	126.19
50.0	365.72	676.29	1767.26	148.01
60.0	332.16	594.72	1513.23	123.74
70.0	272.26	579.32	1384.79	97.2
80.0	201.74	478.32	1211.86	86.3

IV. EFFECT OF CONCENTRATION OF ACETIC ANHYDRIDE

Chemical concentration (%)	Tensile Strength (Mpa)			
	Ampelocissus cavicalulis (Nwogbe)	*Adenia lobat*a (Usoro)	*Morinda morindaoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
Untreated	238.3	588.94	1201.72	67.15
3.0	244.32	591.36	1470.2	115.39
6.0	294.54	600.13	1597.22	124.27
9.0	314.176	615.96	1551.5	120.57
12.0	328.08	634.38	1500.0	109.72
15.0	336.26	664.32	1497.2	88.32
18.0	287.05	612.11	1478.9	72.52
21.0	220.32	570.66	1411.72	46.32

V. EFFECT OF PRETREATMENT TIME OF ACETIC ANHYDRIDE

Time (Minutes)	Tensile Strength (Mpa)			
	Amplelocissus cavicalulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
0.00	238.30	588.94	1201.79	67.15
10.09	240.39	590.72	1292.17	69.31
20.00	252.60	601.32	1321.70	75.99
30.0	258.53	634.00	1439.32	107.67
40.0	296.25	647.32	1491.30	109.16
50.0	303.83	649.15	1570.32	120.19
60.0	329.85	651.30	1592.30	124.43
70.0	336.26	684.57	1600.71	121.51
80.0	341.94	682.32	1528.11	120.102

VI. EFFECT OF CONCENTRATION OF NITRIC ACID

Chemical concentration (%)	Tensile strength (Mpa)			
	Ampelocissus cavicaulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindaoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
Untreated	238.3	588.94	1201.79	67.15
3.0	249.24	590.31	1392.93	69.35
6.0	295.11	595.92	1425.53	82.87
9.0	274.22	593.71	1521.30	91.43
12.0	263.13	582.11	1527.31	106.39
15.0	257.32	500.32	1421.10	102.19

18.0	250.11	492.51	1327.00	91.39
21.0	241.79	490.18	1275.92	24.46

VII. EFFECT OF NITRIC ACID PRETREATMENT TIME

Time (Minutes)		Tensile	Strength	(Mpa)
	Ampelocissus cavicaulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindaoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
0.00	238.3	588.94	1201.79	67.15
10.0	244.42	589.32	1298.11	72.55
20.0	251.32	590.31	1301.92	85.43
30.0	260.44	591.11	1399.20	94.31
40.0	274.32	593.81	1412.50	89.61
50.0	295.71	594.00	1497.77	81.54
60.0	280.70	594.92	1528.94	72.47
70.0	250.36	595.85	1428.32	70.99
80.0	244.16	590.33	1301.32	68.22

VIII. EFFECT OF ZINC CHLORIDE CONCENTRATION

Chemical concentration (%)		Tensile	Strength	(Mpa)
	Ampelocissus cavicaulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
Untreated	238.3	588.94	1201.79	67.15
3.0	320.17	590.55	1271.32	94.31
6.0	316.41	598.10	1282.11	89.42
9.0	281.05	595.32	1279.39	78.99
12.0	256.31	590.90	1257.55	72.54
15.0	240.41	585.36	1242.11	69.33
18.0	231.51	570.68	1231.32	65.32
21.0	200.11	562.32	1211.97	62.11

IX. EFFECT OF ZINC PRETREATMENT TIME

Time (Minutes)		Tensile	Strength	(Mpa)
	Amplelocissus cavicalulis (Nwogbe)	*Adenia lobata* (Usoro)	*Morinda morindaoides* (Ogbuebo)	*Ampelocissus leonensis* (Okpaowoko)
0.00	238.3	588.94	1201.79	62.15
10.00	242.72	592.31	1250.11	72.55
20.00	258.92	598.70	1272.91	85.43
30.00	260.11	595.32	1320.3	94.31
40.00	274.21	590.99	1281.92	89.61
50.00	296.26	588.10	1275.02	81.54
60.00	313.52	572.33	1252.11	77.47

70.00	320.17	551.11	1241.0	70.99
80.00	376.21	550.32	1239.0	68.22

APPENDIX C

OPTIMIZATION OF TENSILE STRENGTH OF THE CHEMICAL TREATED FIBERS

I. . *AMPELOCISSUS CAVICAULIS*

Run Order	Maximum Load (N)	Diameter	Area	Stress
1	244.198	0.97	0.7396	330.176
2	197.217	0.93	0.6194	318.4
3	289.461	1.08	0.9162	315.936
4	157.468	0.78	0.4779	329.52
5	181.826	0.84	0.5542	328.088
6	216.851	0.90	0.6363	340.8
7	35.196	0.38	0.1126	312.576
8	223.044	1.01	0.6794	328.296
9	256.345	0.61	0.8013	319.912
10	121.597	0.83	0.2923	416
11	181.042	0.80	0.5675	319.016
12	153.625	0.98	0.5027	305.6
13	241.565	1.10	0.7544	320.208
14	312.061	0.98	0.9505	328.312
15	247.920	0.87	0.7544	328.632
16	187.967	0.78	0.5945	316.176
17	146.257	0.63	0.4779	306.04
18	63.036	0.76	0.3118	202.168
19	149.823	0.80	0.4537	330.224
20	159.143	0.99	0.5027	316.576
21	252.139	0.01	0.7699	327.496

22	192.645	1.0	0.8013	240.416
23	258.901	0.92	0.7855	329.6
24	136.848		0.6648	205.848
25	246.076	0.91	0.6505	378.288
26	296.554	1.06	0.8826	33.6
27	282.990	1.06	0.8826	320.632
28	173.369	1.00	0.7855	220.712
29	128.881	0.92	0.6648	193.867
30	195.365	0.86	0.5810	336.256
31	311.874	0.97	0.9409	331.464
32	156.234	0.91	0.6505	240.176
33	188.019	0.87	0.5945	316.264
34	199.592	0.89	0.6222	320.784
35	122.751	0.70	0.3849	318.976
36	159.552	0.92	0.6648	240
37	184.089	1.06	0.8826	208.576
38	170.056	1.02	0.8172	208.096
39	127.214	1.63	0.3118	408
40	277.12	1.05	0.8660	320
41	222.612	0.95	0.7089	314.024
42	161.217	0.81	0.5154	312.8
43	126.075	0.81	0.5154	244.616
44	128.840	0.87	0.5945	216.72
45	150.206	0.78	0.4779	314.304
46	217.009	0.90	0.6363	341.048
47	157.132	0.80	0.5027	317.576
48	113.226	0.73	0.4186	270.488
49	158.664	0.92	0.6648	238.664
50	220.099	0.06	0.8826	249.376
51	261.504	1.02	0.8172	320

52	157.552	1.00	0.7855	200.576

II. *ADENIA LOBATA FIBER*

Run	Maximum load (N)	Diameter	Area	Stress
1	554.325	0.97	0.7391	750
2	478.676	0.99	0.7699	609.39
3	463.445	1.00	0.7855	590
4	675.423	1.16	0.0570	639
5	338.429	0.79	0.4902	690.39
6	376.064	0.86	0.5810	647.27
7	324.702	0.81	0.5154	630
8	518.706	1.01	0.8013	647.33
9	391.879	0.99	0.7699	509
10	314.298	0.83	0.5411	580.85
11	184.980	0.62	0.3019	612.72
12	543.649	0.96	0.7239	751
13	565.117	1.05	0.8660	652.56
14	231.025	0.70	0.3849	600.22
15	411.402	0.91	0.6505	632.44
16	362.375	1.00	0.7855	461.33
17	523.368	1.02	0.8172	640.44
18	353.675	1.06	0.8826	400.72
19	226.042	0.76	0.4537	498.22
20	340.500	0.85	0.5675	600
21	360.394	0.86	0.5810	620.3
22	368.451	1.02	0.8172	450.87
23	407.367	0.98	0.744	540
24	278.595	0.79	0.4902	568.33

25	438.843	0.99	0.7699	570
26	350.962	0.98	0.7544	465.22
27	271.698	0.75	0.4418	514.98
28	263.054	0.89	0.6222	422.78
29	633.857	1.20	1.1311	560.39
30	395.395	0.88	0.6083	650
31	475.580	0.93	0.6794	700
32	467.767	0.01	0.8013	584.01
33	565.146	1.06	0.8826	640.32
34	541.021	0.97	0.7391	732
35	509.962	1.0	0.7855	649.22
36	332.52	0.84	0.5542	600
37	828.676	1.36	1.4529	570.36
38	329.591	1.01	0.8013	411.32
39	348.600	0.86	0.5810	600
40	455.841	0.96	0.7855	580.32
41	467.467	1.00	0.7855	595.12
42	482.148	1.02	0.8172	590
43	514.256	1.06	0.8826	582.66
44	352.128	0.87	0.5945	592.31
45	436.601	0.97	0.7391	590.72
46	275.550	0.76	0.4537	607.34
47	511.00	1.05	0.8660	590.07
48	389.426	1.03	0.8333	467.33
49	484.081	1.03	0.8333	580.92
50	302.540	0.81	0.5154	587
51	353.668	1.02	0.8272	432.78
52	447.727	1.00	0.7855	569.99

III. *MORINDA MORINDOIDIES*

Run	Maximum load (N)	Diameter	Area	Stress
1	302.043	0.47	0.1735	1740.88
2	176.282	0.40	0.1257	1402.4
3	49.800	0.23	0.0415	1200
4	169.68	0.43	0.1414	1200
5	485.266	0.60	0.282	1720.8
6	147.610	0.44	0.1521	970.48
7	177.167	0.45	0.1591	113.56
8	45.269	0.24	0.0452	1001.52
9	216.454	0.48	0.1810	1195.88
10	299.160	0.46	0.1662	1800
11	277.939	0.56	0.2464	1128
12	288.384	0.48	0.1810	1593.28
13	162.554	0.36	0.1018	1596.8
14	147.898	0.41	0.1320	1120.44
15	305.614	0.65	0.3319	920.8
16	296.499	0.60	0.2828	1048.44
17	194.177	0.48	0.1810	1072.8
18	23.961	0.25	0.0491	488
19	224.217	0.56	0.2464	912
20	234.344	0.50	0.1964	193.2
21	155.956	0.43	0.1452	1074.08
22	232.563	0.58	0.2643	979.92
23	163.759	0.51	0.2043	801.56
24	236.169	0.56	0.2464	988.48
25	537.56	0.62	0.3020	1780
26	157.140	0.40	0.1257	1250.12
27	370.242	0.58	0.2643	1400.84

28	227.134	0.60	0.2828	803.16
29	349.600	0.22	0.380	920
30	228.201	0.55	0.2376	900.44
31	144.144	0.42	0.1386	1040
32	122.699	0.38	0.1134	1082
33	74.387	0.26	0.0531	1400.88
34	116.160	0.29	0.0660	1760
35	154.493	0.41	0.1320	1170.4
36	50.240	0.20	0.0314	1600
37	68.375	0.30	0.0707	967.12
38	38.603	0.27	0.0572	674.88
39	260.965	0.43	0.1452	1797.28
40	217.200	0.48	0.1810	1200
41	163.609	0.42	0.1386	1180.44
42	422.88	0.58	0.2643	1600
43	59.897	0.26	0.0531	1128
44	80.850	0.35	0.0962	840.44
45	59.136	0.28	0.0616	960
46	290.106	0.51	0.2043	1420
47	267.559	0.48	0.2304	1161.28
48	96.480	0.32	0.0804	1200
49	61.664	0.26	0.0531	1161.28
50	136.038	0.40	0.1257	1082.24
51	180.187	0.43	0.1452	1240.96
52	68.070	0.30	0.0707	962.8

IV. *AMPELOCISSUS LEONENSIS FIBER*

Run	Maximum load (N)	Diameter	Area	Stress
1	20.260	0.52	0.2124	95.385
2	28.863	0.60	0.2879	100.255
3	29.369	0.73	0.4186	70.16
4	89.565	1.10	0.9507	94.21
5	41.434	0.70	0.3849	107.63
6	26.118	0.63	0.3118	83.765
7	35.640	0.55	0.2376	150
8	25.922	0.61	0.2875	90.165
9	57.875	0.1	0.7857	73.66
10	17.026	0.52	0.2124	80.16
11	32.470	0.70	0.3849	84.36
12	16.620	0.50	0.1964	84.625
13	36.554	0.61	0.2923	125.055
14	62.925	0.93	0.6795	92.605
15	77.665	1.10	0.9505	81.71
16	25.922	0.72	0.4072	63.66
17	610.187	0.90	0.6363	96.16
18	12.194	0.63	0.3118	39.11
19	18.641	0.65	0.3319	56.165
20	29.15	0.63	0.3318	87.86
21	56.510	0.87	0.5945	95.055
22	16.445	0.61	0.2923	56.26
23	28.764	0.68	0.3632	79.195
24	26.34	0.65	0.3319	79.385
25	23.699	0.62	0.3019	78.5
26	11.544	0.54	0.2291	50.39
27	51.107	0.81	0.5154	99.16

28	1.966	0.60	0.2828	49.385
29	35.162	0.76	0.4537	77.5
30	22.572	0.59	0.2734	82.56
31	18.077	0.52	0.2124	85.11
32	24.428	0.60	0.2828	89.915
33	35.630	0.56	0.2463	144.66
34	59.062	1.0	0.7855	75.19
35	30.382	0.67	0.3526	86.165
36	43.500	0.81	0.5154	84.4
37	64.054	1.01	0.8015	81.165
38	11.641	0.60	0.2828	41.165
39	33.131	0.71	0.3960	83.665
40	26.537	0.63	0.3118	85.11
41	29.922	0.65	0.3319	90.155
42	44.792	0.72	0.4072	110
43	36.00	0.73	0.4186	86
44	14.399	0.61	0.2923	49.26
45	31.584	0.65	0.3319	95.16
46	32.086	0.75	0.4419	72.61
47	28.149	0.64	0.3217	87.5
48	37.109	0.88	0.6083	61.005
49	26.459	0.62	0.3019	87.64
50	99.677	1.22	1.1691	85.26
51	14.974	0.52	0.2124	70.5
52	49.966	0.92	0.6648	75.16

APPENDIX D.

SUMMARY SEQUENTIAL SUM OF SQUARE FOR CHEMICAL TREATMENTS OF THE FIBERS

I. MODEL SUMMARY TABLE FOR *AMPELOCISSUS CAVICAULIS* (NWOGBE)

Source	Sequential P-Value	Lack of fit P-Value	Adjusted R-Squared	Predicted R-Squared
Linear	0.0001	0.0034	0.3945	0.2902
2FI	0.9384	0.0559	0.6402	0.4691
Quadratic	0.0001	1.3	0.8575	0.7315 suggested
Cubic	0.2707	0.0575	0.6711	-0.4085 aliased

SEQUENTIAL MODEL SUM OF SQUARE

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob>F	
Mean	7.265E+006	1	7.26E+006			
Linear	1.006E+005	5	20114.97	7.65	<0.0001	
2FI	20688.93	2	99.53	0.064	0.9384	
Quadratic	62973.57	9	11398.53	12.20	<0.0001	suggested
Cubic	37157.55	11	1880.81	1.32	0.2707	
Residual	37157.55	26	1429.14			
Total	7.487E+006	52	1.440E+005			

II. MODEL SUMMARY TABLE FOR *ADENIA LOBATA* (USORO)

Source	Sequential P-Value	Lack of fit P-Value	Adjusted R-Square	Predicated R-Square
Linear	<0.0001	0.0394	0.6469	0.5910
2FI	0.1039	0.1597	0.7419	0.6339
Quadratic	0.0001	1.3	0.9233	0.8247 suggested
Cubic	0.1330	0.2913	0.7856	0.3357 aliased

SEQUENTIAL MODEL SUM OF SQUARES

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob<F
Mean	1.784E+007	1	1.78E+007	19.69	
Linear	2.262E+005	5	45241.02	2.81	<0.0001
2FI	35461.78	7	5065.97	2.41	0.0180
Quadratic	2.658E+005	9	29536.957	18.77	0.0001 suggested
Cubic	25672.27	11	2352.02		0.1330 aliased
Residual	36276.39	26	1395.25		
Total	1.817E+007	52	3.495E+005		

III. MODEL SUMMARY TABLE FOR *MORINDA MORINDOIDIES* (OGBUEBO)

Source	Sequential P-Value	Lack of fit P-Value	Adjusted R-Square	Predicted R-Square
Linear	<0.0001	0.1390	0.5045	0.4157
2FI	0.0036	0.4843	0.6572	0.4678
Quadratic	<0.0001	1.09	0.8494	0.6911 suggested
Cubic	0.1193	0.8503	0.7191	0.3768 aliased

SEQUENTIAL MODEL SUM OF SQUARES

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob>F
Means	4.602E+005	1	4.602E+006		
Linear	53816.65	5	33281.59	11.38	<0.0001
2FI	5846.45	7	7688.09	3.72	0.0036
Quadratic	4.670E+005	9	5.122E+005	17.3	<0.0001 suggested
Cubic	43091.70	11	2885.15	1.74	0.1193 aliased
Residual	4.903E+006	26	657.37		
Total		52	94286.71		

IV. MODEL SUMMARY TABLE FOR *AMPELOCISSUS LEONENSIS*

Source	Sequential P-Value	Lack of fit P-Value	Adjusted R-Square	Predicted R-Square
Linar	<0.0001	0.2302	0.6650	0.6090
2FI	0.0107	0.3711	0.9461	0.8578
Quadratic	0.0081	0.6673	0.7937	0.7251 suggested
Cubic	0.4822	0.86735	0.7929	0.5324 aliased

SEQUENTIAL MODEL SUM OF SQUARES

Source	Sum of Squares	df	Mean Square	F Value	P-Value Prob>F
Means	1.443E+006	1	1.443E_006		
Linear	6440991	5	12881.98	21.24	<0.0001
2FI	9969.34	7	1424.19	3.10	0.0107
Quadratic	19205.22	9	2133.91	23.15	0.0001 suggested
Cubic	4069.77	11	369.98	0.99	0.4822 aliased
Residual	9743.45	28	374.75		
Total	1.535E+006	52	29521.74		

APPENDIX E

ACTUAL VALUES, PREDICATED VALUE AND RESIDUALS AGAINST STANDARD ORDER FOR CHEMICAL TREATED FIBERS

I. *AMPELOCISSUS CAVICAULIS* (NWOGBE)

Standard order	Actual Values	Predicated Values	Residuals
1	316.26	316.05	0.21
2	408.00	369.21	38.79
3	320.21	295.92	24.28
4	416.00	349.08	66.92
5	312.58	279.41	33.17
6	378.29	385.73	-7.44
7	328.09	352.70	-24.61
8	315.94	312.44	3.50
9	305.60	332.57	-26.97
10	240.00	332.57	-92.57
11	330.18	332.57	-2.39
12	320.78	332.57	-11.78

13	331.46	332.57	-1.10
14	314.02	298.76	15.27
15	208.58	223.16	-14.58
16	316.58	278.63	37.95
17	205.85	203.03	2.82
18	312.58	326.49	-13.92
19	193.86	175.29	18.57
20	320.00	271.02	48.98
21	200.58	230.76	-30.19
22	238.66	250.89	-12.23
23	244.62	250.89	-6.28
24	240.18	250.89	-10.72
25	216.72	250.89	-34.17
26	249.38	250.89	-1.52
27	318.40	328.30	-9.90
28	328.63	341.94	-13.31
29	320.63	308.17	12.46
30	336.26	321.82	14.44
31	312.80	311.42	1.38
32	340.80	338.70	2.10
33	327.50	345.19	-17.69
34	320.60	304.93	24.67
35	328.30	325.06	3.24
36	306.04	325.06	-19.02
37	328.31	325.06	3.25
38	329.52	325.06	4.46
39	318.98	325.06	-6.08
40	319.02	330.87	-11.86
41	240.42	264.42	-24.00

42	341.05	310.75	30.30
43	208.10	244.29	-36.19
44	314.30	354.04	-39.73
45	220.71	221.13	-0.41
46	319.91	307.71	12.20
47	202.17	267.45	-65.28
48	316.18	287.58	28.59
49	336.00	287.58	48.42
50	270.49	287.58	-17.09
51	320.00	287.58	32.42
52	330.22	287.58	42.64

II. *ADENIA LOBATA* (USORO) FIBER

Standard order	Actual Values	Predicated Values	Residuals
1	640.32	685.20	-44.88
2	600.00	646.53	-46.53
3	652.56	659.18	-6.62
4	580.85	620.51	-39.66
5	630.00	691.53	-61.53
6	570.00	614.18	-44.18
7	690.39	678.87	11.52
8	590.00	626.84	-36.84
9	751.00	652.86	98.14
10	600.0	652.86	-52.86
11	750.00	652.86	97.14
12	732.00	652.86	79.14

13	700.00	652.86	47.14
14	595.12	604.31	-9.19
15	570.36	585.01	-14.65
16	600.00	578.29	21.71
17	568.33	558.99	9.34
18	590.07	600.95	-10.88
19	560.39	562.35	-1.96
20	580.32	607.67	-27.35
21	569.99	555.63	14.36
22	580.92	581.65	-0.23
23	582.66	581.65	1.01
24	584.01	581.65	2.36
25	592.31	581.65	10.66
26	587.00	581.65	5.35
27	609.39	620.21	-10.82
28	632.44	648.98	-16.54
29	614.98	594.19	20.79
30	650.00	622.96	27.04
31	590.00	592.82	-2.82
32	647.27	650.35	-3.08
33	620.30	647.60	-27.30
34	540.00	595.57	-55.57
35	647.33	621.58	25.75
36	640.44	621.58	18.86
37	600.22	621.58	-21.36
38	639.00	621.58	17.42
39	649.22	621.58	27.64
40	612.72	557.77	54.95
41	450.87	442.15	8.72

42	607.34	531.75	75.59
43	411.32	416.13	-4.81
44	590.72	602.58	-11.86
45	422.78	371.33	51.45
46	509.00	512.97	-3.97
47	400.72	460.93	-60.21
48	461.33	486.95	-25.62
49	465.22	486.95	-21.73
50	467.33	486.95	-19.62
51	432.78	486.95	-54.17
52	498.22	486.95	11.27

III. *MORINDA MORINDOIDES* (OGBUEBO) FIBER

Standard order	Actual Values	Predicated Values	Residuals
1	1400.88	1454.06	-53.18
2	1797.28	1776.14	21.14
3	1596.80	1322.85	273.95
4	1800.00	1644.93	155.07
5	1113.56	1227.42	-113.86
6	1780.00	1871.58	-91.58
7	1720.80	1680.71	40.09
8	1200.00	1418.29	-218.29
9	1593.28	1549.50	43.78
10	1600.00	1549.50	50.50
11	1740.88	1549.50	191.38
12	1760.00	1549.50	210.50
13	1040.00	1549.50	-509.50

14	1180.44	1207.56	-27.12
15	967.12	1052.46	-85.34
16	119.20	1076.35	116.85
17	958.48	921.25	37.23
18	1161.28	1219.51	-58.23
19	920.00	909.31	10.69
20	1200.00	1195.61	4.39
21	962.80	933.20	29.60
22	1161.28	1064.41	96.87
23	1128.00	1064.41	63.59
24	1082.00	1064.41	17.59
25	840.44	1064.41	-223.97
26	1082.44	1064.41	17.83
27	1402.40	1377.80	24.60
28	920.80	1014.29	-93.49
29	1400.84	1246.59	154.25
30	960.44	883.09	77.35
31	1600.00	1493.95	106.05
32	970.48	766.94	203.54
33	107.08	1261.65	-187.57
34	801.56	999.23	-197.67
35	1001.52	1130.44	-128.92
36	1072.80	1130.44	-57.64
37	1120.44	1130.44	-10.00
38	1200.00	1130.44	69.56
39	170.40	1130.44	39.96
40	1128.00	1190.00	-62.00
41	879.92	972.19	-92.27
42	1420.00	1058.79	361.21

43	674.88	840.98	-166.10
44	960.00	1233.30	-273.30
45	803.16	797.68	5.48
46	1195.88	1146.70	49.18
47	488.00	884.28	-396.28
48	1048.44	1015.49	32.95
49	1250.12	1015.49	234.63
50	1200.00	1015.49	184.51
51	240.96	1015.49	225.47
52	912.00	1015.49	-103.49

IV. *AMPELOCISSUS LEONENSIS* (OKPAOWOKO) FIBER

Standard order	Actual Values	Predicated Values	Residuals
1	144.66	123.47	21.19
2	83.67	81.99	1.68
3	125.06	113.05	12.00
4	80.16	71.57	8.59
5	150.00	148.62	1.38
6	78.50	65.66	12.84
7	107.65	104.73	2.92
8	70.16	83.90	-13.74
9	84.63	94.31	-9.69
10	84.40	94.31	-9.91
11	95.39	94.31	1.07
12	75.19	94.31	-`9.12
13	85.11	94.31	-9.20

14	90.16	90.26	-0.11
15	81.17	84.02	-2.85
16	87.86	79.85	8.01
17	79.39	73.60	5.78
18	87.50	97.80	-10.30
19	77.50	85.31	-7.81
20	85.11	89.14	-4.03
21	75.16	68.31	6.85
22	87.64	78.73	8.91
23	86.00	78.73	7.27
24	89.92	78.73	11.19
25	49.26	78.73	-29.47
26	85.26	78.73	6.53
27	100.26	104.37	-4.12
28	81.71	89.77	-8.06
29	99.16	93.96	5.20
30	82.56	79.35	3.21
31	110.00	116.08	-6.08
32	83.77	86.88	-3.11
33	95.06	99.07	-4.02
34	79.19	78.24	0.95
35	90.17	88.66	1.51
36	96.16	88.66	7.50
37	92.61	88.66	3.95
38	94.21	88.66	5.55
39	86.17	88.66	-2.49
40	84.36	80.62	3.74
41	56.26	55.43	0.83
42	72.61	70.20	2.41

43	41.16	45.02	-3.85
44	95.16	97.62	-2.46
45	49.38	47.26	2.13
46	73.66	70.03	3.63
47	39.11	49.20	-10.09
48	63.66	59.61	4.05
49	50.39	59.61	-9.22
50	61.01	59.61	1.39
51	70.50	59.61	10.89
52	56.16	59.61	-3.45

APPENDIX F.

DESIGN MATRIX FOR COMPOUNDING OF COMPOSITES

APPENDIX G.

TENSILE STRENGTH FOR THE COMPOUNDING OF COMPOSITES BASED ON RUN ORDER

1. *AMPELOCISSUS CAVICAULIS* FIBER

I. Untreated *Ampelocissus Cavicaulis* Reforced Composite

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1,163.712	60.80	19.14
2	766.08	60.80	12.6
3	1,085.28	60.80	17.85
4	1,292.608	60.80	21.26
5	1,326.657	60.80	21.82
6	1,295.04	60.80	21.3
7	1,269.504	60.80	20.88
8	1,251.264	60.80	20.58
9	1,240.32	60.80	20.4
10	1,075.552	60.80	17.69
11	1,293.824	60.80	21.28
12	1,232.416	60.80	20.27
13	764.256	60.80	12.57
14	1,060.352	60.80	17.44
15	1,258.56	60.80	20.7
16	1,347.936	60.80	22.17
17	1,301.12	60.80	21.4

II. Acetic Anhydride Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1,773.536	60.80	29.17
2	1,366.784	60.80	22.48
3	1,679.904	60.80	27.63
4	1,905.472	60.80	31.34
5	1,939.52	60.80	31.9
6	1,892.704	60.80	31.13
7	1,882.976	60.80	30.97
8	1,869.6	60.80	30.75
9	1,878.112	60.80	30.89
10	1,687.2	60.80	27.75
11	1,915.2	60.80	31.5
12	1,842.24	60.80	30.3
13	1,375.904	60.80	22.63
14	1,685.376	60.80	27.72
15	1,877.504	60.80	30.88
16	1,976	60.80	32.5
17	1,9113.984	60.80	31.48

III. Nitric Acid Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1,630.048	60.80	26.81
2	1,223.904	60.80	20.13

3	1,552.224	60.80	25.53
4	1,763.2	60.80	29
5	1,801.504	60.80	29.63
6	1,765.024	60.80	29.03
7	1,724.288	60.80	28.36
8	1,703.008	60.80	28.01
9	1,725.504	60.80	28.38
10	1,542.496	60.80	25.37
11	1,748	60.80	28.37
12	1,704.224	60.80	28.03
13	1,246.4	60.80	20.5
14	1,527.904	60.80	25.13
15	1,732.8	60.80	28.5
16	1,833.12	60.80	30.15
17	1,771.104	60.80	29.13

IV Zinc Chloride Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1,412.384	60.80	23.23
2	1,001.984	60.80	16.48
3	1,319.36	60.80	21.7
4	1,526.688	60.80	25.11
5	1,575.936	60.80	25.92
6	1,544.32	60.80	25.4
7	1,503.32	60.80	24.73
8	1,504.5	60.80	24.75

9	1,490.208	60.80	24.51
10	1,339.304	60.80	21.88
11	1,550.4	60.80	25.5
12	1,496.896	60.80	24.62
13	1,018.4	60.80	16.75
14	1,315.104	60.80	21.63
15	1,492.64	60.80	24.55
16	1,597.824	60.80	26.28
17	1,550.4	60.80	25.5

V. Sodium chloride treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2,352.352	60.80	38.69
2	1,939.52	60.80	31.9
3	2,273.92	60.80	37.4
4	2,497.664	60.80	41.08
5	2,523.2	60.80	41.5
6	2,498.88	60.80	41.1
7	2,462.4	60.80	40.5
8	22,439.904	60.80	40.13
9	2,432	60.80	40
10	2,264.192	60.80	37.24
11	2,482.856	60.80	40.82
12	2,416.8	60.80	39.75
13	1,968.096	60.80	32.37
14	2,249.6	60.80	37

15	2,447.2	60.80	40.25
16	2,536.576	60.80	41.72
17	2,504.96	60.80	41.2

2. *ADENIA LOBATA*

I. Untreated *Adenia lobata*

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2,314.048	60.80	38.06
2	1,901.824	60.80	31.28
3	2,234.4	60.80	36.75
4	2,458.144	60.80	40.43
5	2,492.192	60.80	40.99
6	2,460.576	60.80	40.47
7	2,419.84	60.80	39.8
8	2,402.208	60.80	39.51
9	2,394.304	60.80	39.38
10	2,234.4	60.80	36.75
11	2,444.16	60.80	40.2
12	2,383.36	60.80	39.2
13	1,915.2	60.80	31.5
14	2,204	60.80	36.25
15	2,408.896	60.80	39.62
16	2,500.704	60.80	41.13
17	2,452.064	60.80	40.33

II. Sodium Hydroxide Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2,712.896	60.80	44.62
2	2,325.6	60.80	38.25
3	2,629.6	60.80	43.25
4	2,857.6	60.80	47
5	2,860.64	60.80	47.05
6	2,690.4	60.80	44.25
7	2,819.296	60.80	46.37
8	2,801.056	60.80	46.07
9	2,805.312	60.80	46.14
10	2,631.424	60.80	43.28
11	2,850.304	60.80	46.88
12	2,782.208	60.80	45.76
13	2,329.248	60.80	38.31
14	2,610.144	60.80	42.93
15	2,819.904	60.80	46.38
16	2,897.728	60.80	47.66
17	2,835.712	60.80	46.64

III. Acetic Anhydride Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2,705.6	60.80	44.5
2	2.307.968	60.80	37.96
3	2,627.168	60.80	43.21

4	2,834.496	60.80	46.62
5	2,868.544	60.80	47.18
6	2,836.928	60.80	46.66
7	2,811.392	60.80	46.24
8	2,793.152	60.80	45.94
9	2,782.208	60.80	45.76
10	2,617.44	60.80	43.05
11	2,835.104	60.80	46.63
12	2,774.304	60.80	45.63
13	2,306.144	60.80	37.93
14	2,663.04	60.80	43.8
15	2,770.048	60.80	45.56
16	2,889.824	60.80	47.53
17	2,843.008	60.80	46.76

IV. Nitric Acid treated *Adenia lobata*

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2,52.632	60.80	41.54
2	2,143.808	60.80	35.26
3	2,436.864	60.80	40.08
4	2660	60.80	43.75
5	2,688.576	60.80	44.22
6	2660	60.80	43.75
7	2,631.424	60.80	43.28
8	2,598.904	60.80	42.74
9	2,602.848	60.80	42.81

10	2,439.904	60.80	40.13
11	2,670.944	60.80	43.93
12	2,594.944	60.80	42.68
13	2.129.824	60.80	35.03
14	2432	60.80	40
15	2,620.48	60.80	43.1
16	2,710.464	60.80	44.58
17	2,678.848	60.80	44.06

V. Zinc chloride treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	2352.352	60.80	38.69
2	1,939.52	60.80	31.9
3	2,121.92	60.80	34.9
4	2,481.248	60.80	40.81
5	2,515.296	60.80	41.37
6	2,468.48	60.80	40.6
7	2,458.144	60.80	40.43
8	2,424.704	60.80	39.88
9	2,445.984	60.80	40.23
10	2,264.192	60.80	37.24
11	2,466.656	60.80	40.57
12	2,436.256	60.80	40.07
13	1,962.896	60.80	32.12
14	2,248.992	60.80	36.99
15	2,432	60.80	40

16	2,506.176	60.80	41.22
17	2,489.76	60.80	40.95

3. *MORINDA MORINDOIDES*

I. Zinc Chloride Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	5,068.896	60.80	83.37
2	5,045.792	60.80	82.99
3	4,541.152	60.80	74.69
4	5,062.208	60.80	83.26
5	4,972.832	60.80	81.79
6	5,019.648	60.80	82.56
7	4,924.8	60.80	81
8	4,710.176	60.80	77.47
9	4,978.304	60.80	81.88
10	5,032.416	60.80	82.77
11	4,684.64	60.80	77.05
12	4,851.84	60.80	79.8
13	4,876.16	60.80	80.2
14	5,040.928	60.80	82.91
15	5,070.112	60.80	83.39
16	4,544.8	60.80	74.75
17	5,102.944	60.80	83.93

II. Sodium hydroxide Treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	6,855.2	60.80	112.75
2	6,806.56	60.80	111.95
3	6,165.728	60.80	101.41
4	6.854.592	60.80	112.74
5	6,916	60.80	113.75
6	6,796.224	60.80	111.78
7	6,716.576	60.80	110.47
8	6,638.752	60.80	109.19
9	6,755.488	60.80	111.11
10	6,794.4	60.80	111.75
11	6,613.216	60.80	108.77
12	6,582.816	60.80	108.27
13	6,809.6	60.80	112
14	6,832.704	60.80	112.38
15	6,855.808	60.80	112.76
16	6,319.552	60.80	103.94
17	6,879.52	60.80	113.15

III. Acetic anhydride treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	6,467.904	60.80	106.38
2	6,536	60.80	107.5
3	5,955.36	60.80	97.95

4	6,475.2	60.80	106.5
5	6,493.44	60.80	106.8
6	6,433.856	60.80	105.82
7	6,353.6	60.80	104.5
8	6,245.984	60.80	102.73
9	6,423.62	60.80	105.65
10	6,431.424	60.80	105.78
11	6,250.848	60.80	102.81
12	6,262.4	60.80	103
13	6,427.168	60.80	105.71
14	6,470.336	60.80	106.42
15	6,317.12	60.80	103.9
16	5,941.984	60.80	97.73
17	6,471.552	60.80	106.44

IV. Nitric acid treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	6,043.52	60.80	99.4
2	6,020.416	60.80	99.02
3	5,515.168	60.80	90.71
4	6,052.032	60.80	99.54
5	6,098.848	60.80	100.31
6	6,009.472	60.80	98.84
7	5,914.016	60.80	97.27
8	5,836.192	60.80	95.99
9	5,983.328	60.80	98.41

10	5,991.232	60.80	98.54
11	5,811.264	60.80	95.58
12	5,826.464	60.80	95.83
13	6,002.176	60.80	98.72
14	6,045.344	60.80	99.43
15	6,044.128	60.80	99.41
16	5,516.992	60.80	90.74
17	6,076.96	60.80	99.95

VI. Untreated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	4843.328	60.80	79.66
2	4843.208	60.80	79.51
3	4516.224	60.80	74.28
4	4845.76	60.80	79.7
5	4870.688	60.80	80.11
6	4844.544	60.80	79.68
7	4745.44	60.80	78.05
8	4707.744	60.80	77.43
9	4791.04	60.80	78.8
10	4820.832	60.80	79.29
11	4688.288	60.80	77.11
12	4696.192	60.80	77.24
13	4821.44	60.80	79.3
14	4845.76	60.80	79.7
15	4843.936	60.80	79.67

16	4560	60.80	75
17	4857.312	60.80	79.89

4. *AMPELOCISSUS LEONESIS*

I. Untreated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1160.064	60.80	19.08
2	1083.456	60.80	17.82
3	676.704	60.80	11.13
4	1185.6	60.80	19.5
5	1279.84	60.80	21.05
6	1127.232	60.80	18.54
7	1075.552	60.80	17.69
8	1006.24	60.80	16.55
9	1129.664	60.80	18.58
10	1025.696	60.80	16.87
11	1002.592	60.80	16.49
12	1003.2	60.80	16.5
13	1139.392	60.80	18.74
14	1161.28	60.80	19.1
15	1192.288	60.80	19.61
16	724.128	60.80	11.91
17	1209.92	60.80	19.9

II. Nitric acid treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1264.032	60.80	20.79
2	1217.824	60.80	20.03
3	737.504	60.80	12.13
4	1272.544	60.80	20.93
5	1375.296	60.80	22.62
6	1199.584	60.80	19.73
7	1134.528	60.80	18.66
8	1056.704	60.80	19.38
9	1203.84	60.80	19.8
10	1160.064	60.80	19.08
11	1031.168	60.80	16.96
12	1046.368	60.80	17.21
13	1222.688	60.80	20.11
14	1265.856	60.80	20.82
15	1265.856	60.80	20.82
16	783.712	60.80	12.89
17	1297.472	60.80	21.34

III. Sodium hydroxide treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1979.04	60.80	32.55
2	1932.832	60.80	31.79
3	1495.68	60.80	24.6
4	1983.296	60.80	32.62

5	2090.304	60.80	34.38
6	1916.416	60.80	31.52
7	1895.136	60.80	31.17
8	1816.704	60.80	29.88
9	1948.64	60.80	32.05
10	1875.072	60.80	30.84
11	1791.776	60.80	29.47
12	1799.072	60.80	29.59
13	1958.368	60.80	32.21
14	1980.256	60.80	32.57
15	1980.864	60.80	32.58
16	1543.104	60.80	25.38
17	1996.672	60.80	32.84

IV. Acetic anhydride treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1721.248	60.80	28.31
2	1675.04	60.80	27.55
3	1207.488	60.80	19.86
4	1725.504	60.80	18.38
5	1854.4	60.80	30.5
6	1658.624	60.80	27.28
7	1644.64	60.80	27.05
8	1589.312	60.80	26.14
9	1690.848	60.80	27.81
10	1617.28	60.80	26.6

11	1553.44	60.80	25.55
12	1541.28	60.80	25.35
13	1705.44	60.80	28.05
14	1735.84	60.80	28.55
15	1723.072	60.80	28.34
16	1315.712	60.80	21.64
17	1738.88	60.80	28.6

V. Zinc chloride treated

Run Order	Maximum Load (N)	Area (mm^2)	Tensile Strength (MPa)
1	1246.4	60.80	20.5
2	1169.792	60.80	19.24
3	790.4	60.80	13
4	1281.056	60.80	21.07
5	1368	60.80	22.5
6	1213.568	60.80	19.96
7	1170.4	60.80	19.25
8	1084.064	60.80	17.83
9	1219.04	60.80	20.05
10	1143.04	60.80	18.8
11	1085.28	60.80	17.85
12	1066.432	60.80	17.54
13	1225.728	60.80	20.16
14	1247.616	60.80	20.52
15	1248.224	60.80	20.53
16	780.064	60.80	12.83
17	1263.423	60.80	20.78

APPENDIX H.

SEQUENTIAL MODEL SUM OF SQUARE FOR COMPOUNDING PROCESS

1. Untreated *ampelocissus cavicaulis*

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0635	0.0053	0.2834	-0.0900	
2FI	0.0756	0.0095	0.5179	-0.0126	
Quadratic	< 0.0001	0.8015	0.9647	0.9310	Suggested
Cubic	0.8015		0.9507		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	6380.67	1	6380.67			
Linear vs Mean	57.35	3	19.12	3.11	0.0635	
2FI vs Linear	38.57	3	12.86	3.11	0.0756	
Quadratic vs 2FI	39.24	3	13.08	43.19	< 0.0001	Suggest
Cubic vs Quadratic	0.43	3	0.14	0.34	0.8015	Aliased
Residual	1.69	4	0.42			
Total	6517.95	17	383.41			

2. *Ampelocissus cavicaulis* treated with sodium hydroxide

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R Squared	
Linear	0.0645	0.0047	0.2814	-0.0806	
2FI	0.0896	0.0078	0.4984	-0.0222	
Quadratic	< 0.0001	0.8109	0.9671	0.9372	Suggested
Cubic	0.8109		0.9536		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**
Mean vs Total	14765.94	1	14765.94		
Linear vs Mean	59.60	3	19.87	3.09	0.0645
2FI vs Linear	38.72	3	12.91	2.87	0.0896

Quadratic vs 2FI	42.84	3	14.28	48.51	< 0.0001
Cubic vs Quadratic	0.40	3	0.13	0.32	0.8109
Residual	1.66	4	0.42		
Total	14909.17	17	877.01		

3. *Ampelocissus cavicaulis* treated with nitric acid

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0498	0.0075	0.3117	-0.0463	
2FI	0.0728	0.0136	0.5408	0.0421	
Quadratic	< 0.0001	0.9099	0.9635	0.9487	Suggested
Cubic	0.9099		0.9434		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**	
Mean vs Total	9343.18	1	9343.18			
Linear vs Mean	61.03	3	20.34	3.42	0.0498	
2FI vs Linear	37.69	3	12.56	3.16	0.0728	
Quadratic vs 2FI	37.53	3	12.51	39.60	< 0.0001	Suggest
Cubic vs Quadratic	0.25	3	0.084	0.17	0.9099	Aliased
Residual	1.96	4	0.49			
Total	9481.65	17	557.74			

4. *Ampelocissus cavicaulis* treated with acetic anhydride

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0651	0.0082	0.2803	-0.0906	
2FI	0.0883	0.0139	0.4993	-0.0530	
Quadratic	0.0002	0.7456	0.9531	0.8962	Suggested
Cubic	0.7456		0.9377		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**

Mean vs Total	12470.88	1	12470.88			
Linear vs Mean	57.44	3	19.15	3.08	0.0651	
2FI vs Linear	37.60	3	12.53	2.90	0.0883	
Quadratic vs 2FI	40.45	3	13.48	33.23	0.0002	Suggested
Cubic vs Quadratic	0.69	3	0.23	0.43	0.7456	Aliased
Residual	2.15	4	0.54			
Total	12609.20	17	741.72			

5. *Ampelocissus cavicaulis* treated with zinc chloride

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0664	0.0047	0.2779	-0.1008	
2FI	0.0759	0.0084	0.5138	-0.0318	
Quadratic	< 0.0001	0.7751	0.9657	0.9287	Suggested
Cubic	0.7751		0.9532		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**	
Mean vs Total	25829.71	1	25829.71			
Linear vs Mean	58.39	3	19.46	3.05	0.0664	
2FI vs Linear	39.97	3	13.32	3.10	0.0759	
Quadratic vs 2FI	40.82	3	13.61	44.92	< 0.0001	Suggested
Cubic vs Quadratic	0.47	3	0.16	0.38	0.7751	Aliased
Residual	1.65	4	0.41			
Total	25971.01	17	1527.71			

6. Untreated *adenia lobata*

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0683	0.0050	0.2746	-0.1070	
2FI	0.0666	0.0095	0.5252	0.0026	
Quadratic	< 0.0001	0.8180	0.9658	0.9358	Suggested
Cubic	0.8180		0.9515		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**	
Mean vs Total	24979.28	1	24979.28			
Linear vs Mean	59.21	3	19.74	3.02	0.0683	
2FI vs Linear	42.21	3	14.07	3.29	0.0666	
Quadratic vs 2FI	40.64	3	13.55	43.96	< 0.0001	Suggested
Cubic vs Quadratic	0.41	3	0.14	0.31	0.8180	Aliased
Residual	1.75	4	0.44			
Total	25123.50	17	1477.85			

7. *Adenia lobata* treated with sodium hydroxide

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.1117	0.0035	0.2116	-0.1721	
2FI	0.2188	0.0039	0.3287	-0.4539	
Quadratic	0.0001	0.3214	0.9449	0.7723	Suggested
Cubic	0.3214		0.9563		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > **F**	
Mean vs Total	34051.62	1	34051.62			
Linear vs Mean	46.23	3	15.41	2.43	0.1117	
2FI vs Linear	28.43	3	9.48	1.76	0.2188	
Quadratic vs 2FI	50.86	3	16.95	38.29	0.0001	Suggested
Cubic vs Quadratic	1.69	3	0.56	1.61	0.3214	Aliased
Residual	1.41	4	0.35			
Total	34180.24	17	2010.60			

8. *Adenia lobata* treated with acetic anhydride

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0598	0.0055	0.2905	-0.0733	
2FI	0.0879	0.0092	0.5067	-0.0289	
Quadratic	< 0.0001	0.9288	0.9685	0.9592	Suggested
Cubic	0.9288		0.9503		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	33972.89	1	33972.89			
Linear vs Mean	57.66	3	19.22	3.18	0.0598	
2FI vs Linear	36.52	3	12.17	2.90	0.0879	
Quadratic vs 2FI	40.10	3	13.37	49.94	< 0.0001	Suggested
Cubic vs Quadratic	0.18	3	0.061	0.14	0.9288	Aliased
Residual	1.69	4	0.42			
Total	34109.04	17	2006.41			

9. *Adenia lobata* treated with nitric acid

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0551	0.0064	0.3001	-0.0554	
2FI	0.0877	0.0108	0.5138	-0.0001	
Quadratic	< 0.0001	0.8389	0.9633	0.9348	Suggested
Cubic	0.8389		0.9469		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	29731.51	1	29731.51			
Linear vs Mean	58.25	3	19.42	3.29	0.0551	
2FI vs Linear	35.76	3	11.92	2.90	0.0877	
Quadratic vs 2FI	38.87	3	12.96	41.82	< 0.0001	Suggested
Cubic vs Quadratic	0.37	3	0.12	0.28	0.8389	Aliased
Residual	1.79	4	0.45			
Total	29866.56	17	1756.86			

10. *Adenia lobata* treated with zinc chloride

Source	Sequential p-value	Lack of Fit p-value	R-Squared	Adjusted R-Squared	Predicted
Linear	0.0322	0.0007	0.3595	0.0368	
2FI	0.0618	0.0013	0.5875	0.2000	

Quadratic	< 0.0001	0.4770	0.9844	0.9469	Suggested
Cubic	0.4770		0.9844		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	25466.15	1	25466.15			
Linear vs Mean	71.66	3	23.89	3.99	0.0322	
2FI vs Linear	39.24	3	13.08	3.40	0.0618	
Quadratic vs 2FI	37.50	3	12.50	85.76	< 0.0001	Suggested
Cubic vs Quadratic	0.44	3	0.15	1.01	0.4770	Aliased
Residual	0.58	4	0.15			
Total	25615.57	17	1506.80			

11. Untreated *morinda morindoidies*

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0976	0.0040	0.2295	-0.1624	
2FI	0.0829	0.0070	0.4712	-0.0826	
Quadratic	0.0002	0.3624	0.9456	0.7861	Suggested
Cubic	0.3624		0.9538		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	1.110E+005	1	1.110E+005			
Linear vs Mean	55.36	3	18.45	2.59	0.0976	
2FI vs Linear	43.75	3	14.58	2.98	0.0829	
Quadratic vs 2FI	45.39	3	15.13	30.07	0.0002	Suggested
Cubic vs Quadratic	1.81	3	0.60	1.41	0.3624	Aliased
Residual	1.71	4	0.43			
Total	1.112E+005	17	6539.34			

12. *morinda morindoidies* treated with sodium hydroxide

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared

Linear	0.0613	0.0068	0.2876	-0.0784	
2FI	0.0893	0.0115	0.5031	-0.0419	
Quadratic	0.0002	0.5700	0.9496	0.8495	Suggested
Cubic	0.5700		0.9440		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	2.075E+005	1	2.075E+005			
Linear vs Mean	75.63	3	25.21	3.15	0.0613	
2FI vs Linear	48.17	3	16.06	2.88	0.0893	
Quadratic vs 2FI	51.81	3	17.273	0.54	0.0002	Suggested
Cubic vs Quadratic	1.44	3	0.48	0.77	0.5700	Aliased
Residual	2.52	4	0.63			
Total	2.077E+005	17	12216.52			

13. *morinda morindoidies* treated with acetic anhydride

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0574	0.0945	0.2953	-0.0485	
2FI	0.0802	0.1618	0.5198	0.0818	
Quadratic	0.0082	0.9451	0.8601	0.8327	Suggested
Cubic	0.9451		0.7750		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	1.855E+005	1	1.855E+005			
Linear vs Mean	56.73	3	18.91	3.23	0.0574	
2FI vs Linear	36.17	3	12.06	3.03	0.0802	
Quadratic vs 2FI	31.71	3	10.57	9.11	0.0082	Suggested
Cubic vs Quadratic	0.66	3	0.22	0.12	0.9451	Aliased
Residual	7.47	4	1.87			
Total	1.856E+005	17	10917.24			

14. *morinda morindoidies*treated with nitric acid

	Sequential		Lack of Fit	Adjusted	Predicted
Source	p-value	p-value	R-Squared	R-Squared	
Linear	0.0634	0.0053	0.2835	-0.0896	
2FI	0.0758	0.0095	0.5177	-0.0125	
Quadratic	< 0.0001	0.8019	0.9647	0.9311	Suggested
Cubic	0.8019		0.9507		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	1.616E+005	1	1.616E+005			
Linear vs Mean	57.31	3	19.103.11	0.0634		
2FI vs Linear	38.50	3	12.83	3.10	0.0758	
Quadratic vs 2FI	39.22	3	13.07	43.22	< 0.0001	Suggested
Cubic vs Quadratic	0.43	3	0.14	0.34	0.8019	Aliased
Residual	1.69	4	0.42			
Total	1.618E+005	17	9516.50			

15. *morinda morindoidies* treated with zinc chloride

	Sequential	Lack of Fit		Adjusted	Predicted
Source	p-value	p-value	R-Squared	R-Squared	
Linear	0.0743	0.0018	0.2642	-0.1130	
2FI	0.0623	0.0036	0.5253	0.0511	
Quadratic	< 0.0001	0.6213	0.9748	0.9304	Suggested
Cubic	0.6213		0.9704		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	1.047E+005	1	1.047E+005			
Linear vs Mean	19.63	3	6.54	2.92	0.0743	
2FI vs Linear	14.70	3	4.90	3.38	0.0623	
Quadratic vs 2FI	13.94	3	4.65	60.42	< 0.0001	Suggested
Cubic vs Quadratic	0.18	3	0.059	0.65	0.6213	Aliased
Residual	0.36	4	0.090			
Total	1.048E+005	17	6164.38			

16. untreated *ampelocissus leonensis*

	Sequential	Lack of	Fit	Adjusted	Predicted
Source	p-value	p-value	R-Squared	R-Squared	
Linear	0.0557	0.2270	0.2987	-0.0289	
2FI	0.1072	0.3418	0.4910	0.0464	
Quadratic	0.0366	0.9572	0.7687	0.7421	Suggested
Cubic	0.9572		0.6229		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	5260.99	1	5260.99			
Linear vs Mean	47.67	3	15.89	3.27	0.0557	
2FI vs Linear	27.88	3	9.29	2.64	0.1072	
Quadratic vs 2FI	24.04	3	8.01	5.00	0.0366	Suggested
Cubic vs Quadratic	0.77	3	0.26	0.098	0.9572	Aliased
Residual	10.45	4	2.61			
Total	5371.79	17	315.99			

17. *ampelocissus leonensis*treated with nitric acid

	Sequential	Lack of	Fit	Adjusted	Predicted
Source	p-value	p-value	R-Squared	R-Squared	
Linear	0.0652	0.0794	0.2802	-0.0581	
2FI	0.1132	0.1196	0.4712	-0.0074	
Quadratic	0.0037	0.9899	0.8778	0.8966	Suggested
Cubic	0.9899		0.7916		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	6072.57	1	6072.57			
Linear vs Mean	54.00	3	18.00	3.08	0.0652	
2FI vs Linear	33.08	3	11.03	2.57	0.1132	
Quadratic vs 2FI	36.03	3	12.01	12.09	0.0037	Suggested
Cubic vs Quadratic	0.18	3	0.060	0.035	0.9899	Aliased
Residual	6.78	4	1.69			
Total	6202.64	17	364.86			

18. *Ampelocissus leonensis* treated with sodium hydroxide

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0595	0.1162	0.2911	-0.0444	
2FI	0.1160	0.1726	0.4764	-0.0109	
Quadratic	0.0104	0.9153	0.8365	0.7751	Suggested
Cubic	0.9153		0.7452		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	16277.53	1	16277.53			
Linear vs Mean	45.11	3	15.04	3.19	0.0595	
2FI vs Linear	26.46	3	8.82	2.53	0.1160	
Quadratic vs 2FI	27.21	3	9.07	8.34	0.0104	Suggested
Cubic vs Quadratic	0.83	3	0.28	0.16	0.9153	Aliased
Residual	6.78	4	1.69			
Total	16383.92	17	963.76			

19. *Ampelocissus leonensis*treated with acetic anhydride

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	0.0585	0.1565	0.2931	-0.0352	
2FI	0.1301	0.2221	0.4645	-0.0249	
Quadratic	0.0223	0.8359	0.7903	0.6245	Suggested
Cubic	0.8359		0.6973		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	12207.94	1	12207.94			
Linear vs Mean	46.61	3	15.54	3.21	0.0585	
2FI vs Linear	26.24	3	8.75	2.39	0.1301	
Quadratic vs 2FI	26.61	3	8.87	6.18	0.0223	Suggested
Cubic vs Quadratic	1.76	3	0.59	0.28	0.8359	Aliased
Residual	8.29	4	2.07			
Total	12317.44	17	724.56			

20. *ampelocissus leonensis* treated with zinc chloride

	Sequential	Lack of Fit		Adjusted	Predicted
Source	p-value	p-value	R-Squared	R-Squared	
Linear	0.0515	0.1640	0.3079	-0.0232	
2FI	0.1166	0.2420	0.4882	-0.0035	
Quadratic	0.0177	0.9557	0.8127	0.7893	Suggested
Cubic	0.9557		0.6952		Aliased

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	6114.60	1	6114.60			
Linear vs Mean	47.71	3	15.90	3.37	0.0515	
2FI vs Linear	26.43	3	8.81	2.53	0.1166	
Quadratic vs 2FI	25.94	3	8.65	6.78	0.0177	Suggested
Cubic vs Quadratic	0.62	3	0.21	0.10	0.9557	Aliased
Residual	8.31	4	2.08			
Total	6223.61	17	366.09			

APPENDIX I.

ANALYSIS OF VARIANCE TABLES FOR THE COMPOUNDING PROCESS

ANOVA Ttable for compounding of untreated *Adenia lobata* (Usoro) fiber.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	142.08	8	17.76	65.78	<0.0001
A-Rotation Speed	25.63	1	25.63	94.93	<0.0001
B-Temperature	33.13	1	33.13	122.70	<0.0001
C-Time	1.45	1	1.45	5.37	0.0484
AB	17.77	1	17.77	65.81	<0.0001
BC	23.47	1	23.47	86.93	<0.0001
B^2	32.74	1	32.74	121.26	<0.0001
C^2	6.18	1	6.18	22.89	0.0023
Residual	2.14	8	0.27		
Lack of fit	1.39	4	0.35	1.20	0.6875
Pure error	0.75	4	0.19		

R-squared = 0.9782, adj. R-square = 0.9612, pred. R- squared = 0.9386, adeq. precision = 23.488

ANOVA Table for compounding of *Adenia lobata* (Usoro) treated with NaOH

Source	Sum of Squares	Df	Mean Squares	F Value	P-value Prob>F
Model	124.43	7	17.78	38.18	<0.0001
A-Rotation Speed	23.05	1	23.05	49.52	<0.0001
B-Temperature	20.45	1	20.45	43.92	<0.0001
C-Time	2.73	1	2.73	5.86	0.0386
AB	17.56	1	17.56	37.71	<0.0002
BC	10.08	1	10.08	21.65	<0.0012
B^2	36.30	1	36.30	77.98	<0.0001
C^2	11.81	1	11.81	25.36	0.0007
Residual	4.19	9	0.47		
Lack of fit	2.78	5	0.56	1.58	0.3383
Pure error	1.41	4	0.35		
Cor Total	128.62	16			

R-squared = 0.9624, pred. R-squared = 0.8563 Adj. R-squared = 0.9420, Adeq. Precision = 19.11.

ANOVATtable for compounding of *Adenia lobata* (Usoro) treated with acetic anhydride.

Source	Sum of Squares	Df	Mean Squares	F Value	P-value Prob>F
Model	134.54	8	16.82	84.09	<0.0001
A-Rotation Speed	22.95	1	22.95	114.75	<0.0001
B-Temperature	34.16	1	34.16	170.8	<0.0001
C-Time	1.55	1	1.55	7.75	0.009
AB	15.76	1	15.76	78.8	<0.0001
BC	20.03	1	20.03	100.15	<0.0001
B^2	32.93	1	32.93	113.80	<0.0001
C^2	5.54	1	5.54	27.7	0.0018
Residual	1.60	8	0.20		
Lack of fit	0.91	4	0.23	1.15	0.8092
Pure error	0.69	4	0.17		

R-Squared = 0.9809, Pred R-Squared = 0.9565, Adj. R-squared = 0.9660, adeq. Precision = 25.333.

ANOVA Table for compounding of *Adenia lobata* (Usoro) treated with nitric Acid

Source	Sum of Squares	Df	Mean Squares	F Value	P-value Prob>F
Model	132.21	8	18.89	59.98	<0.0001
A-Rotation Speed	26.90	1	26.90	85.43	<0.0001
B-Temperature	30.62	1	30.62	97.23	<0.0001
C-Time	0.73	1	0.73	12.32	0.0161
AB	16.52	1	16.52	52.48	<0.0001
BC	18.58	1	18.58	59.00	<0.0001
B^2	31.67	1	31.67	100.57	<0.0001
C^2	5.60	1	5.60	17.77	0.0023
Residual	2.83	9	0.31		
Lack of fit	1.04	5	0.21	0.46	0.7893
Pure error	1.79	4	0.45		
Cor Total	135.05	16			

R-Squared = 0.9790, Pred R-Squared = 0.9437, Adj. R-squared = 0.9627, adeq. Precision = 24.799.

ANOVA Table for compounding of *Adenia lobata* (Usoro) treated with zinc chloride

Source	Sum of Squares	Df	Mean Squares	F Value	P-value Prob>F
Model	148.28	8	18.54	129.88	<0.0001
A-Rotation Speed	35.28	1	35.28	247.21	<0.0001
B-Temperature	33.13	1	33.13	232.14	<0.0001
C-Time	3.25	1	3.25	22.78	0.0014
AB	14.78	1	14.78	103.59	<0.0001
AC	4.43	1	4.43	31.05	0.0005
BC	20.03	1	20.03	140.32	<0.0001
B^2	25.84	1	25.84	181.04	<0.0001
C^2	9.67	1	9.67	67.79	<0.0001
Residual	1.14	8	0.14		
Lack of fit	0.56	4	0.14	0.96	0.5142
Pure error	0.58	4	0.15		
Cor Total	149.43	16			

R-Squared = 0.9924, Pred R-Squared = 0.9584, Adj. R-squared = 0.9847, adeq. Precision = 36.012.

ANOVA Table for compounding of untreated *Morinda morindoides* (Ogbuebo)

Source	Sum of Squares	Df	Mean Squares	F Value	P-value Prob>F
Model	145.43	8	18.18	56.81	<0.0001
A-Rotation Speed	41.72	1	41.72	130.34	<0.0001
B-Temperature	13.11	1	13.11	40.97	0.0006
C-Time	1.53	1	1.53	4.78	0.0410
AB	9.58	1	9.58	29.94	0.0017
AC	4.41	1	4.41	13.78	0.0139
BC	29.76	1	29.76	93.00	<0.0001
B^2	35.87	1	35.87	112.09	<0.0001
C^2	7.50	1	7.50	23.44	0.0035
Residual	2.58	8	0.32		
Lack of fit	1.87	4	0.47	1.5	0.4660
Pure error	0.71	4	0.18		
Cor Total					

R-Squared = 0.9758, Pred R-Squared = 0.8575, Adj. R-squared = 0.9516, adeq. Precision = 21.059.

ANOVA Table for compounding of *Morinda morindoides* (ogbuebo) treated with NaOH.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	175.67	8	21.96	44.81	<0.0001
A-Rotation Speed	30.85	1	30.85	62.90	<0.0001
B-Temperature	44.46	1	44.46	90.73	<0.0001
C-Time	1.32	1	1.32	2.69	0.0346
AB	31.70	1	31.70	64.69	<0.0001
BC	15.76	1	15.76	32.16	0.0004
B^2	45.83	1	45.83	93.53	<0.0001
C^2	4.07	1	4.07	8.31	<0.0231
Residual	3.90	8	0.49		
Lack of fit	3.39	4	0.85	1.72	0.6220
Pure error	0.51	4	0.13		

R-Squared = 0.9727, Pred R-Squared = 0.9044, Adj. R-squared = 0.9514, adeq. Precision = 22.929.

ANOVA Table for compounding of *Morinda morindoides* (Ogbuebo) treated with acetic anhydride

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	125.80	8	15.73	18.07	<0.0001
A-Rotation Speed	22.85	1	22.85	26.26	<0.0010
B-Temperature	32.36	1	32.36	37.20	<0.0003
C-Time	3.52	1	3.52	4.05	0.0468
AB	15.72	1	15.72	18.07	<0.0032
BC	19.71	1	19.71	22.66	0.0016
B^2	24.61	1	24.61	28.29	<0.0008
C^2	5.62	1	5.62	6.46	<0.0412
Residual	6.93	8	0.87		
Lack of fit	1.46	4	0.37	0.42	0.9665
Pure error	5.47	4	1.37		

R-Squared = 0.9327, Pred R-Squared = 0.8646, Adj. R-squared = 0.8804, adeq. Precision = 13.476.

ANOVA Table for compounding of *Morinda morindoides* (Ogbuebo) treated with nitric Acid.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	135.31	8	16.91	73.52	<0.0001
A-Rotation Speed	24.68	1	24.68	107.30	<0.0001
B-Temperature	32.08	1	32.08	139.48	<0.0001
C-Time	1.56	1	1.56	6.78	0.0317
AB	17.81	1	17.81	77.43	<0.0001
BC	19.98	1	19.98	86.87	0.0001
B^2	31.37	1	31.37	136.39	<0.0001
C^2	6.17	1	6.17	26.83	<0.0017
Residual	1.85	8	0.23		
Lack of fit	1.16	4	0.29	1.26	0.7390
Pure error	0.69	4	0.17		

R-Squared = 0.9792, Pred R-Squared = 0.9428, Adj. R-squared = 0.9631, adeq. Precision = 24.374.

ANOVA Table for compounding of *Morinda morindoides* (Ogbuebo) treated with zinc chloride

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	48.09	8	6.87	85.91	<0.0001
A-Rotation Speed	10.15	1	10.15	126.89	<0.0001
B-Temperature	9.48	1	9.48	118.58	<0.0001
C-Time	1.8	1	1.8	10.1	0.0422
AB	8.70	1	8.70	108.82	<0.0001
BC	5.83	1	5.83	72.93	0.00001
B^2	10.44	1	10.44	130.53	<0.0001
C^2	2.84	1	2.84	35.49	<0.0002
Residual	0.72	8	0.080		
Lack of fit	0.36	4	0.072	0.79	0.6053
Pure error	0.36	4	0.090		

R-Squared = 0.9853, Pred. R-Squared = 0.9513, Adj. R-squared = 0.9738, adeq. Precision = 28.758

ANOVA Table for compounding of untreated *Ampelocissus leonensis* (Okpaowoko).

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	105.37	7	15.05	16.01	0.0004
A-Rotation Speed	19.25	1	19.25	20.48	0.0036
B-Temperature	27.49	1	27.49	29.24	0.0011
C-Time	5.92	1	5.92	6.3	0.0259
AB	14.33	1	14.33	15.244	0.0085
BC	12.96	1	12.96	10.27	0.0111
B^2	22.42	1	22.42	23.85	0.0022
Residual	8.43	9	0.94		
Lack of fit	2.98	5	0.6	0.64	0.9635
Pure error	5.45	4	1.36		

R-Squared = 0.8788, Pred R-Squared = 0.7535, Adj. R-squared = 0.8061, adeq. Precision = 12.763.

ANOVA Table for compounding of *Ampelocissus leonensis* (Okpaowoko) treated with NaOH.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	100.36	7	14.34	21.40	0.0002
A-Rotation Speed	15.60	1	15.60	23.28	0.0037
B-Temperature	28.92	1	28.92	43.16	0.0004
C-Time	5.59	1	5.59	8.34	0.0418
AB	12.53	1	12.53	18.70	0.0071
BC	13.36	1	13.36	19.94	0.0059
B^2	24.36	1	24.36	36.36	0.0008
Residual	6.02	9	0.67		
Lack of fit	4.25	5	0.85	1.27	0.8374
Pure error	1.77	4	0.44		

R-Squared = 0.8964, Pred R-Squared = 0.7450, Adj. R-squared = 0.8342, adeq. Precision = 13.558.

ANOVA Table for compounding of *Ampelocissus leonensis* (Okpaowoko) treated with nitric Acid.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	124.39	7	17.77	28.21	0.0001
A-Rotation Speed	22.85	1	22.85	36.27	0.0006
B-Temperature	30.93	1	30.93	49.10	0.0002
C-Time	2.22	1	2.22	3.52	0.0481
AB	15.64	1	15.64	24.83	0.0020
BC	16.73	1	16.73	25.56	0.0017
B^2	30.09	1	30.09	47.76	0.0002
C^2	4.52	1	4.52	7.17	0.0459
Residual	5.68	9	0.63		
Lack of fit	0.91	5	0.182	0.29	0.9846
Pure error	4.78	4	1.2		

R-Squared = 0.9409, Pred R-Squared = 0.8992, Adj. R-squared = 0.8950, adeq. Precision = 14.396.

ANOVA Table for compounding of *Ampelocissus leonensis* (Okpaowoko) treated with acetic anhydride.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	99.54	7	14.22	12.81	0.0003
A-Rotation Speed	15.04	1	15.04	13.55	0.005
B-Temperature	31.32	1	31.32	28.22	0.0001
C-Time	2.24	1	2.24	2.02	0.0460
AB	14.67	1	14.67	13.22	0.0057
BC	11.26	1	11.26	10.14	0.0111
B^2	25.01	1	25.01	22.53	0.0010
Residual	9.97	9	1.11		
Lack of fit	3.68	5	0.74	0.66	0.910
Pure error	6.29	4	1.57		

R-Squared = 0.8907, Pred R-Squared = 0.7636, Adj. R-squared = 0.8252, adeq. Precision = 13.165.

ANOVA Table for compounding of *Ampelocissus leonensis* (Okpaowoko) treated with zinc chloride.

Source	Sum of Squares	df	Mean Squares	F Value	P-value Prob>F
Model	98.55	7	14.08	12.14	0.0003
A-Rotation Speed	16.88	1	16.88	14.55	0.0004
B-Temperature	29.07	1	29.07	25.06	0.0007
C-Time	3.76	1	3.76	3.24	0.0426
AB	12.50	1	12.50	10.78	0.0100
BC	13.62	1	13.62	11.74	0.0079
B^2	22.73	1	22.73	19.59	0.0016
Residual	10.45	9	1.16		
Lack of fit	4.15	5	0.83	0.72	0.889
Pure error	6.31	4	1.58		

R-Squared = 0.8857, Pred R-Squared = 0.6942, Adj. R-squared = 0.8172, adeq. Precision = 13.077.

APPENDIX J.

EFFECT OF COMPOUNDING CONDITIONS ON THE TENSILE STRENGTH OF THE COMPOSITES

a. EFFECT OF ROTATION SPEED

I. *AMPELOCISSUS CAVICAULIS* FIBER REINFORCED COMPOSITE

Rotation Speed	Untreated	NaOH treated	Acetic anhydr	Nitric acid	Zinc chloride
60	19.5	39	29.5	23.5	27.2
70	21.6	40.6	31.1	24	28.1
80	22	41.5	32.5	25.6	29.8
90	22.5	42	33	26.3	30.5
100	23.8	42.8	33.5	27	31.8

II. *ADENIA LOBATA* FIBER REINFORCED COMPOSITE

Rotation speed	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	zinc chloride treated
60	38.4	45.5	44.9	41.8	38.9
70	39.5	46	45.1	42.5	40

80	40.2	46.9	46.5	43.7	41
90	41.5	47.5	47.5	44.8	42.2
100	42	48.8	48.5	45.3	43

III. *MORINDA MORINDOIDES* FIBER REINFORCED COMPOSITES

Rotation speed	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
60	80.5	110.5	104.4	97.7	78.2
70	81.8	111	105	98.2	78.8
80	82.5	112.5	106	99.5	80
90	84	113.9	107.1	100.5	80.8
100	85	114.6	107.9	101	81

IV. *AMPELOCISSUS LEONENSIS* FIBER REINFORCED COMPOSITE

Rotation speed	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
60	17	30.8	26.5	19	18.7
70	17.8	31.1	27.2	19.5	19.3
80	18.2	31.9	27.9	20.6	20
90	19.9	32.8	28.5	21.8	20.8
100	21.5	33.5	29	22.5	21.8

b. EFFECT OF TEMPERATURE

I. *AMPELOCISSUS CAVICAULIS* FIBER REINFORCED COMPOSITE

Temperature	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
165	19	38.5	28.2	27.8	22.2

170	19.8	40	30.8	30	25
175	22	40.6	33	32	27
180	20.6	43	31	30	24.6
185	20	40	30	29.2	24

II. *ADENIA LOBATA* FIBER REINFORCED COMPOSITE

Temperature	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
165	37.8	44.2	44	41	32.8
170	40	46.8	46	43	40.6
175	41.5	48.2	46.9	44.5	42.5
180	39.2	46.5	46	43.1	39.8
185	38.8	45.3	45	42.8	39

III. *MORINDA MORINDOIDES* FIBER REINFORCED COMPOSITE

Temperature	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
165	80.3	109.5	103.8	97	78
170	82.3	112	106	99.1	79.1
175	84	114	108	101	81
180	81.8	111.2	105.8	98.6	79
185	81	110.8	105	98	78.5

IV. *AMPELOCISSUS LEONENSIS* FIBER REINFORCED COMPOSITE

Temperature	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
165	16.2	30.2	25.8	18	17.8

170	18.1	31.8	27	21	19.5
175	18.5	32.2	28	21.6	20.1
180	18	31	27.2	20.8	19.6
185	17.6	30.3	26.8	20.2	19

c. EFFECT OF DISPERION TIME

I. *AMPELOCISSUS CAVICAULIS* FIBER REINFORCED COMPOSITE

Dispersion time	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	19.9	39.2	29.8	23.8	27.8
10	20.2	39.4	29.9	23.9	27.8
15	20.3	39.4	30.1	24	27.9
20	20.4	39.8	30.2	24.2	28
25	20	39.5	30	24	27.8

II. *ADENIA LOBATA* FIBER REINFORCED COMPOSITE

Dispersion time	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	38.8	44.6	45	42	38.2
10	39.2	46	45.8	43	39.7
15	40	46.2	46	43.8	39.9
20	41	46.8	46.1	43.5	40
25	40	46.1	45.8	43	39.8

III. *MORINDA MORINDOIDES* FIBER REINFORCED COMPOSITE

Dispersion time	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated

5	81.3	111.3	104.8	97.8	78.8
10	81.8	111.5	105	98	79
15	82	112	105.3	98.3	79.2
20	82	112	105.8	98.5	79.5
25	81.5	111.8	105.2	98.2	79.3

IV. *AMPELOCISSUS LEONENSIS* FIBER REINFORCED COMPOSITE

Dispersion time	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	18.2	32	27.8	19.6	19.8
10	18.3	32	27.8	19.8	19.8
15	18.3	32	27.8	19.8	19.9
20	18.3	32.1	27.8	20	20
25	18.4	32.2	27.9	19.8	20.2

APPENDIX K

DESIGN MATRIX FOR COMPRESSION MOLDING PROCESS

APPENDIX L.

SEQUENTIAL SUM OF SQUARE FOR COMPRESSION MOLDING PROCESS

I. *AMPELOCISSUS CAVICAULIS*

Source	Sequential p-value	Lack of Fit p-value	Adjusted R-Squared	Predicted R-Squared	
Linear	< 0.0001	0.0001	0.8578	0.8428	
2FI	0.9817	< 0.0001	0.8379	0.7792	
Quadratic	< 0.0001	0.0809	0.9432	0.9069	Suggested
Cubic	0.0044	0.7761	0.9677	0.8998	Aliased

Sequential Model Sum of Squares

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Mean vs Total	68310.74	1	68310.74			
Linear vs Mean	2510.05	7	358.58	73.36	< 0.0001	
2FI vs Linear	31.03	15	2.07	0.37	0.9817	
Quadratic vs 2FI	230.16	3	76.72	39.31	< 0.0001	Suggested
Cubic vs Quadratic	79.62	27	2.95	2.66	0.0044	Aliased
Residual	35.53	32	1.11			
Total	71197.14	85	837.61			

APPENDIX M

TENSILE STRENGTH FOR COMPRESSION MOLDING PROCESS BASED ON RUN ORDER

AMPELOCISSUS CAVICAULIS

RUN	MAXIMUM LOAD (N)	AREA (mm^2)	TENSILE STRENGHT (MPa)
1	1974.78	60.80	32.48
2	1785.69	60.80	29.37
3	1422.11	60.80	23.39
4	1824	60.80	30.0
5	2128	60.80	35.0
6	2451.45	60.80	40.32

7	1426.97	60.80	23.47
8	1785.69	60.80	29.37
9	1737.66	60.80	28.58
10	2056.86	60.80	33.83
11	2188.19	60.80	35.99
12	1488.99	60.80	24.49
13	1347.93	60.80	22.17
14	1368	60.80	22.5
15	2059.90	60.80	33.88
16	2242.30	60.80	36.88
17	1426.36	60.80	23.46
18	2016.73	60.80	33.17
19	1785.69	60.80	29.37
20	1307.2	60.80	21.5
21	1462.84	60.80	24.06
22	1692.06	60.80	27.83
23	1458.59	60.80	23.99
24	2110.97	60.80	34.72
25	1580.8	60.80	26.0
26	2481.24	60.80	40.81
27	2487.32	60.80	40.91
28	1591.13	60.80	26.17
29	1447.04	60.80	23.8
30	2512.25	60.80	41.32
31	1706.65	60.80	28.07
32	1324.22	60.80	21.78
33	2597.37	60.80	42.72
34	1983.90	60.80	32.63
35	1580.8	60.80	26.0

36	1435.48	60.80	23.61
37	1383.80	60.80	22.76
38	1388.06	60.80	22.83
39	1239.10	60.80	20.38
40	2006.4	60.80	33.0
41	1879.93	60.80	30.92
42	1692.06	60.80	27.83
43	1896.96	60.80	31.2
44	1361.92	60.80	22.4
45	1234.24	60.80	20.3
46	1915.2	60.80	31.5
47	1672	60.80	27.5
48	1783.26	60.80	29.33
49	1556.48	60.80	25.6
50	1692.06	60.80	27.83
51	1639.77	60.80	26.97
52	2303.10	60.80	37.88
53	1347.93	60.80	22.17
54	2465.44	60.80	40.55
55	1249.44	60.80	20.55
56	1983.90	60.80	32.63
57	2149.88	60.80	35.36
58	2006.4	60.80	33.0
59	1564.38	60.80	25.73
60	1451.90	60.80	23.88
61	1556.48	60.80	25.6
62	1383.80	60.80	22.76
63	1674.43	60.80	27.54
64	1631.26	60.80	26.83

65	2432.00	60.80	40.00
66	1419.68	60.80	23.35
67	1446.43	60.80	23.79
68	1470.75	60.80	24.19
69	1536.41	60.80	25.27
70	1379.55	60.80	22.69
71	2426.52	60.80	39.91
72	1475.00	60.80	24.26
73	1834.33	60.80	30.17
74	2487.93	60.80	40.92
75	1361.31	60.80	22.39
76	1702.4	60.80	28.0
77	1971.13	60.80	32.42
78	1289.56	60.80	21.21
79	1537.02	60.80	25.28
80	1341.24	60.80	22.06
81	1805.15	60.80	29.69
82	1785.08	60.80	29.36
83	1630.65	60.80	26.82
84	1289.56	60.80	21.21
85	1288.35	60.80	21.19

ADENIA LOBATA

RUN	MAXIMUM LOAD (N)	AREA (mm^2)	TENSILE STRENGHT (MPa)
1	2633.24	60.80	43.31
2	2231.96	60.80	36.71
3	2370.59	60.80	38.99

4	2231.96	60.80	36.71
5	2006.4	60.80	33.0
6	2451.45	60.80	40.32
7	2377.88	60.80	39.11
8	2231.96	60.80	36.71
9	2317.08	60.80	38.11
10	2742.68	60.80	45.11
11	2188.19	60.80	35.99
12	2127.39	60.80	34.99
13	2073.88	60.80	34.11
14	2104.89	60.80	34.62
15	2059.90	60.80	33.88
16	2242.30	60.80	36.88
17	2038.01	60.80	33.52
18	2688.57	60.80	44.22
19	2231.96	60.80	36.71
20	2013.08	60.80	33.11
21	2438.68	60.80	40.11
22	2256.28	60.80	37.11
23	2431.39	60.80	39.99
24	2110.97	60.80	34.72
25	1976.60	60.80	32.51
26	2481.24	60.80	40.81
27	2487.32	60.80	40.91
28	1988.76	60.80	32.71
29	2067.2	60.80	34.00
30	2512.25	60.80	41.32
31	2275.13	60.80	37.42
32	1891.48	60.80	31.11

33	2147.45	60.80	35.32
34	2645.40	60.80	43.51
35	1976.60	60.80	32.51
36	2208.25	60.80	36.32
37	1976.60	60.80	32.51
38	2256.28	60.80	37.11
39	1769.88	60.80	29.11
40	2006.4	60.80	33.00
41	2506.17	60.80	41.22
42	2256.28	60.80	37.11
43	2371.2	60.80	39.00
44	1945.6	60.80	32.0
45	1763.2	60.80	29.0
46	2553.6	60.80	42.0
47	2110.36	60.80	34.71
48	2377.88	60.80	89.11
49	1945.6	60.80	32.0
50	2256.28	60.80	37.11
51	2049.56	60.80	33.71
52	2303.10	60.80	37.88
53	2073.88	60.80	34.11
54	2465.44	60.80	40.55
55	1921.88	60.80	31.61
56	2644.8	60.80	43.5
57	2149.88	60.80	35.36
58	2006.4	60.80	33.0
59	2086.04	60.80	34.31
60	2073.88	60.80	34.11
61	1945.6	60.80	32.0

62	1976.60	60.80	32.51
63	2232.56	60.80	36.72
64	2718.36	60.80	44.71
65	2432	60.80	40.0
66	2366.33	60.80	38.92
67	2066.59	60.80	33.99
68	2451.45	60.80	40.32
69	2560.28	60.80	42.11
70	2122.52	60.80	34.91
71	2426.52	60.80	39.91
72	1843.45	60.80	30.32
73	2292.76	60.80	37.71
74	2487.93	60.80	40.92
75	2269.05	60.80	37.32
76	2432	60.80	40.0
77	2596.65	60.80	43.22
78	1842.24	60.80	30.3
79	2195.48	60.80	36.11
80	1916.41	60.80	31.52
81	2256.28	60.80	37.11
82	2231.36	60.80	36.7
83	2038.01	60.80	33.52
84	1842.24	60.80	30.3
85	2147.45	60.80	35.32

MORINDA MORIDODIS

RUN	MAXIMUM LOAD (N)	AREA (mm^2)	TENSILE STRENGHT (MPa)
1	1635.52	60.80	26.9

2	1369.21	60.80	22.52
3	1623.96	60.80	26.71
4	1465.88	60.80	24.11
5	1830.08	60.80	30.1
6	1866.56	60.80	30.7
7	1520	60.80	25.0
8	1368	60.80	22.5
9	1675.64	60.80	27.56
10	1635.52	60.80	26.9
11	1459.2	60.80	24.0
12	1155.2	60.80	19.0
13	1216	60.80	20.0
14	1198.97	60.80	19.72
15	1580.8	60.80	26.0
16	1652.54	60.80	27.18
17	870.65	60.80	14.32
18	1641.6	60.80	27.0
19	1234.84	60.80	20.31
20	1575.93	60.80	25.92
21	1526.08	60.80	25.1
22	1380.16	60.80	22.7
23	1550.4	60.80	25.5
24	1800.89	60.80	29.62
25	1094.4	60.80	18.0
26	1763.2	60.80	29.0
27	1774.14	60.80	29.18
28	1270.72	60.80	20.9
29	1094.4	60.80	18.0
30	1702.4	60.80	28.0

31	1337.6	60.80	22.0
32	854.24	60.80	14.05
33	1130.88	60.80	18.6
34	1702.4	60.80	28.0
35	1337.6	60.80	22.0
36	1285.92	60.80	21.15
37	729.6	60.80	12.0
38	1429.40	60.80	23.51
39	851.2	60.80	14.0
40	1526.08	60.80	25.1
41	1459.2	60.80	24.0
42	1520	60.80	25.0
43	1308.41	60.80	21.52
44	1101.08	60.80	18.11
45	811.07	60.80	13.34
46	1641.6	60.80	27.0
47	1216	60.80	20.0
48	1441.56	60.80	23.71
49	1457.98	60.80	23.98
50	1697.53	60.80	27.92
51	1186.20	60.80	19.51
52	1551.00	60.80	25.51
53	1589.92	60.80	26.15
54	1612.41	60.80	26.52
55	1526.08	60.80	25.1
56	1520	60.80	25.0
57	1490.20	60.80	24.51
58	1507.84	60.80	24.8
59	1398.4	60.80	23.0

60	1094.4	60.80	18.0
61	1276.8	60.80	21.0
62	870.65	60.80	14.32
63	1348.54	60.80	22.18
64	1465.88	60.80	24.11
65	1709.08	60.80	28.11
66	1459.2	60.80	24.0
67	1275.58	60.80	20.98
68	1410.56	60.80	23.2
69	1587.48	60.80	29.11
70	1166.14	60.80	19.18
71	1709.08	60.80	28.11
72	1160.06	60.80	19.08
73	1380.76	60.80	22.71
74	1709.08	60.80	28.11
75	1314.49	60.80	21.62
76	1064.60	60.80	17.51
77	1711.52	60.80	28.15
78	797.08	60.80	13.11
79	972.8	60.80	16.0
80	972.8	60.80	16.0
81	1337.6	60.80	22.0
82	1276.8	60.80	21.0
83	1113.85	60.80	18.32
84	1233.02	60.80	20.28
85	1389.88	60.80	22.86

AMPELOCISSUS LEONENSIS

RUN	MAXIMUM LOAD (N)	AREA (mm^2)	TENSILE STRENGHT

			(MPa)
1	1465.28	60.80	24.1
2	1398.4	60.80	23
3	1589.92	60.80	26.15
4	1457.98	60.80	23.98
5	1774.14	60.80	29.18
6	1830.08	60.80	30.1
7	1496.28	60.80	24.61
8	1343.68	60.80	22.1
9	1500.54	60.80	24.68
10	1591.13	60.80	26.17
11	1702.4	60.80	28
12	991.64	60.80	16.31
13	1520	60.80	25.0
14	1623.96	60.80	26.71
15	1551.00	60.80	25.51
16	1612.41	60.80	26.52
17	972.8	60.80	16.0
18	1550.4	60.80	25.5
19	1270.72	60.80	20.9
20	1369.21	60.80	22.52
21	1520	60.80	25.0
22	1635.52	60.80	26.9
23	1405.08	60.80	23.11
24	1630.04	60.80	26.81
25	1296.25	60.80	21.32
26	1740.09	60.80	28.62
27	1679.29	60.80	27.62
28	1234.84	60.80	20.31

29	1094.4	60.80	18.0
30	1702.4	60.80	28
31	1575.93	60.80	25.92
32	851.2	60.80	14
33	984.96	60.80	16.2
34	1647.68	60.80	27.1
35	1246.4	60.80	20.5
36	1313.28	60.80	21.6
37	1065.21	60.80	17.52
38	1441.56	60.80	23.71
39	854.24	60.80	14.05
40	1800.89	60.80	29.62
41	1520	60.80	25.0
42	1459.2	60.80	24.0
43	1526.08	60.80	25.1
44	922.94	60.80	15.18
45	1161.88	60.80	19.11
46	1520	60.80	25.0
47	1186.20	60.80	19.51
48	1429.40	60.80	23.51
49	1380.76	60.80	22.71
50	1641.6	60.80	27.0
51	1216	60.80	20.0
52	1580.8	60.80	26.0
53	1550.4	60.80	25.5
54	1652.54	60.80	27.18
55	1800.89	60.80	29.62
56	1532.16	60.80	25.2
57	1866.56	60.80	30.7

58	1763.2	60.80	29.0
59	1675.64	60.80	27.56
60	918.08	60.80	15.1
61	1369.21	60.80	22.52
62	1397.18	60.80	22.98
63	1711.52	60.80	28.15
64	1587.48	60.80	26.11
65	1526.68	60.80	25.11
66	1389.88	60.80	22.86
67	1233.02	60.80	20.28
68	1539.45	60.80	25.32
69	1337.6	60.80	22.0
70	1464.06	60.80	24.08
71	1660.44	60.80	27.31
72	1398.4	60.80	23
73	1276.8	60.80	21
74	1591.13	60.80	26.17
75	1285.92	60.80	21.15
76	1124.8	60.80	18.5
77	1697.53	60.80	27.92
78	1130.88	60.80	18.6
79	991.64	60.80	16.31
80	870.65	60.80	14.32
81	1526.68	60.80	25.11
82	1216	60.80	20.0
83	1465.88	60.80	24.11
84	1155.2	60.80	19
85	1337.6	60.80	22

APPENDIX N

EFFECT OF PROCESS CONDITIONS ON COMPRESSION MOLDING PROCESS

A. EFFECT OF FIBER LENGTH

1. *AMPELOCISSUS CAVICAULIS*

Fiber Length(mm)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0.5	19.91	25.31	23.31	20.1	21.32
1	20.41	29.42	25.1	20.92	23
2	21.59	32.71	27.5	21.5	25.21
3	22.1	35.22	28.31	22.31	26.51
4	21.31	33.11	27.59	22	25.32
5	20.77	30.31	25.32	21.91	23
6	20.51	25.2	23	20.97	22.11

2. *ADENIA LOBATA*

Fiber length (mm)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0.5	28.32	36.51	38.62	35.31	29.31
1	31.33	39.32	42.11	38.32	35.33
2	35	42.92	44.91	40.32	37.11
3	33.71	40.91	43.31	39.11	36.71
4	32.32	39.52	41.32	37.32	34.11
5	30.11	37.31	39.1	35	32.11
6	29.98	35.31	37.32	33.71	30.52

3. *MORINDA MORINDOIDES*

Fiber length (mm)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0.5	38.11	58.77	50	45	42.71
1	48.72	69.32	63.75	58.53	55.31
2	58.31	77.71	68.31	62.33	60.71
3	56.31	76.11	65	60.1	58.55
4	53.11	72.31	62.31	58.22	55.31
5	48	68.72	58.9	55.31	52.4
6	46.32	65.31	55.39	52	50.33

4. *AMPELOCISSUS LEONESIS*

Fiber length (mm)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0.5	19.5	22.31	21.11	20.15	20.51
1	20.1	24	22.72	20.81	21
2	20.3	26.32	24.32	21.3	21.51
3	20.5	28.11	25.62	21.5	21.8
4	20.4	27.79	25	21.32	21.72
5	20.3	25.32	24.72	20.8	21.32
6	20.1	23.11	23.11	20.5	20.8

B. EFFECT OF TEMPERATURE AT 10MINS

1. *AMPELOCISSUS CAVICAULIS*

Temperature (°C)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	21.17	35.31	28	22.31	25.33
150	21.78	37.72	29.15	24.62	28.62
160	22.75	39.18	31.33	26.11	30.28
170	22.4	41.98	32.48	26	30
180	21.32	40.22	31.32	25.32	28.32
190	20.11	38.31	30.41	23.11	25.11

2. *ADENIA LOBATA*

Temperature (°C)	untreated	NaoH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	30	34.57	35.31	32.61	31.32
150	33.51	36.99	38.42	35.91	33.51
160	34.71	39.32	41.39	37.79	35.62
170	34	40.91	43.31	39.11	36.71
180	33.62	40.11	42.31	38.91	36.11
190	32.11	39.88	40.11	38.32	35.71

3. *MORINDA MORINDOIDIES*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	48.32	55.72	52.61	50.11	48.71
150	50.72	58.31	55.32	53.61	50.32
160	52.11	65.3	58.51	55.92	55.17
170	56.31	76.11	65.9	60.1	59.1
180	56.11	75.2	65	58.6	58.8
190	55.98	74	63.98	58	58.68

4. *AMPELOCISSUS LEONENSIS*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	19.91	23.96	23.15	21.11	20.31
150	20.5	24.51	24	21.5	21.8
160	20.4	28.11	25.62	21	21
170	20.31	27.62	25	20.91	20.51
180	20.11	27.51	24.62	20.52	20.32
190	20	26.32	24.11	20.38	20.11

C. EFFECT OF TEMPERATURE AT 5MINS

1. *AMPELOCISSUS CAVICAULIS*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	20.99	34.11	27.11	21.11	23.55
150	21.1	35.89	28.32	22.52	26.32
160	21.98	37.32	29.11	25.32	29
170	22.98	39.11	31.31	26.92	31.32
180	22.52	42.62	33.32	26.1	29.11
190	21	40.1	32.11	24.22	28.31

2. *ADENIA LOBATA*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	28.61	32.11	33.61	31.55	30.11
150	32.51	35.36	36.72	34.62	33.52
160	32.98	38.33	40.11	36.11	34.1
170	34.99	41.32	44.22	39.99	37.71
180	35.1	41.5	44.67	40.5	38.2
190	33.52	40.55	41.22	38.92	36.71

3. *MORINDA MORINDODIES*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
140	46.11	54.31	51.32	48.32	47.72
150	48.38	56.71	56.72	50.71	49.55
160	50.17	63.5	57.61	53.91	53.36
170	54.32	71.32	61.32	56.91	55.71
180	58.72	78.11	65.92	62.32	55.98
190	58.11	77.62	64.31	61.32	55.32

4. *AMPELOCISSUS LEONENSIS*

Temperature (^oC)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
14	19.78	22.71	22.78	20.62	20.11
150	19.91	23.62	25.91	21.76	21.84
160	20.96	28.96	26.32	22	21.98
170	20.81	27.86	26.11	21.57	20.91
180	20	27.62	25.31	21	20.52
190	19.81	27	25.1	20.86	20.41

D. EFFECT OF HOLDING TIME AT 140^{o}C ON THE TENSILE STRENGTH OF THE COMPOSITES

1. *AMPELOCISSUS CAVICAULIS*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	18.9	19.8	19.5	19	19.2
10	19.2	20.32	19.8	19.55	19.5
13	19.5	23.72	21.81	20.82	21.33
16	19.98	24.22	23	21.57	22.22
20	20.22	25.11	25.22	23.17	24.51

2. *ADENIA LOBATA*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	19.5	22.1	21.76	20	19.8
10	19.8	22.52	21.96	20.89	20.32
13	20.15	23.15	22	21.52	21.18
16	21.32	23.72	22.98	22.81	22.32
20	22.32	24.98	24.52	24	23.92

3. *MORINDA MORINDOIDIES*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	24.1	27.32	26.31	25.91	25.32
10	25.32	27.92	26.78	26.1	25.51
13	26.66	28	27.11	26.98	26.71
16	27.11	28.52	28.72	27.91	27.72
20	28.62	29.81	29.65	29	28.82

4. *AMPELOCISSUS LEONENSIS*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	18.81	20.51	19.57	19.11	18.92
10	18.91	21.32	19.68	19.32	19.11
13	19.11	21.92	19.91	19.51	19.32
16	19.51	22.1	20	19.61	19.55
20	19.72	22.72	20.51	20.1	19.97

E. EFFECT OF TEMPERATURE ON TENSILE STRENGTH OF THE COMPOSITE AT 170°C

1. *AMPELOCISSUS CAVICAULIS*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	22.98	39.11	31.31	26.92	31.32
10	22.4	41.98	32.48	26	30
13	21.2	37.31	31.71	25.32	28.72
16	20.76	30.41	28.11	23.11	26.32
20	20.32	26.2	25.32	22.21	24.57

2. *ADENIA LOBATA*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	34.99	40.91	44.22	39.99	37.71
10	34	41.32	43.31	39.11	36.71
13	32.11	38.97	40.1	37	34.31
16	30.32	34.32	37.31	35.32	31.9
20	28.42	31.11	35.22	32.11	29.11

3. *MORINDA MORINDOIDIES*

Time (mins)	Untreated	NaHH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	56.31	70.11	60	56.91	55.71
10	54.32	71.32	61.32	58.1	58.55
13	54.11	60.32	57.11	56.91	55.31
16	52	58.31	55	54.32	53.31
20	50.31	55.11	52.98	52.11	51.91

4. *AMPELOCISSUS LEONENSIS*

Time (mins)	Untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
5	20.81	27.86	26.11	21.51	20.91
10	20.31	27.62	25	20.51	20.51
13	20.11	23.51	22.31	20.62	20.41
16	20	23	22.91	20.35	20.22
20	19.91	22.98	22.52	20.17	20

F. EFFECT OF FIBER LOADING ON THE TENSILE STRENGTH OF COMPOSITES

1. *AMPELOCISSUS CAVICAULIS*

Fiber loading (%)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0	19.5	19.5	19.5	19.5	19.5
5	20.61	27.11	25.32	21.32	22.52
10	21.22	30.32	28.11	24.05	25.51
20	21.98	38.57	30.31	25.22	28.11
30	22.47	41.98	32.48	26.11	30.284
40	22	35.32	30.52	25.72	28.52
50	21.31	30.21	28.11	23.11	26.11

2. *ADENIA LOBATA*

fiber loading (%)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0	19.5	19.5	19.5	19.5	19.5
5	32.72	39.32	36.71	34	33.15
10	36.31	44.71	42.51	40.22	38.72
20	41.32	47.89	47.489	44.8396	41.75
30	38.12	45.81	45.11	42.72	39.15
40	35.33	43.2	42	38.31	36.71
50	30.1	40.11	38.1	35.22	33.11

3. *MORINDA MORINDOIDIES*

Fiber loading (%)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0	19.5	19.5	19.5	19.5	19.5
5	62.81	95.32	93.22	82.31	63.31
10	80.183	113.747	107.396	100.766	84.183
20	63.32	100.1	90.58	84.32	70.31
30	60.15	82.71	70.32	65.18	62.32
40	52.32	78	62.11	58.62	55.11
50	40.72	60.32	50.31	45	41.31

4. *AMPELOCISSUS LEONENSIS*

Fiber loading (%)	untreated	NaOH treated	Acetic anhydride treated	Nitric acid treated	Zinc chloride treated
0	19.5	19.5	19.5	19.5	19.5
5	19.8	22.31	21.9	19.9	20.1
10	20.1	25.72	23.5	20.5	20.8
20	20.5	29.31	25.1	21.8	22
30	20.8	31.22	27	22.1	22.2
40	20.912	33.8707	29.76	22.362	22.38
50	20	30.15	27.22	21	22

APPENDIX O

DENSITY OF COMPOSITES

I. *MORINDA MORINDOIDIES*

fiber loading (%)	Untreated	Sodium hydroxide	Acetric anhydride	Nitric acid	Zinc chloride
0	0.95	0.95	0.95	0.95	0.95
10	0.92	0.78	0.86	0.88	0.8
20	0.88	0.71	0.84	0.86	0.77
30	0.87	0.69	0.83	0.85	0.75
40	0.84	0.68	0.81	0.83	0.74
50	0.82	0.65	0.78	0.8	0.71

II. *AMPELOCISSUS CAVICAULIS*

fiber loading (%)	Untreated	Sodium hydroxide	Acetric anhydride	Nitric acid	Zinc chloride
0	0.95	0.95	0.95	0.95	0.95
10	0.9	0.75	0.85	0.88	0.77
20	0.88	0.69	0.82	0.86	0.76
30	0.86	0.67	0.81	0.85	0.74

40	0.85	0.66	0.79	0.83	0.73
50	0.82	0.62	0.75	0.8	0.7

III. ADENIA LOBATA

fiber loading (%)	Untreated	Sodium hydroxide	Acetric anhydride	Nitric acid	Zinc chloride
0	0.95	0.95	0.95	0.95	0.95
10	0.93	0.77	0.86	0.91	0.79
20	0.91	0.71	0.84	0.9	0.77
30	0.89	0.69	0.82	0.87	0.76
40	0.87	0.68	0.81	0.85	0.75
50	0.83	0.65	0.78	0.8	0.7

IV. AMPELOCISSUS LEONENSIS

fiber loading (%)	Untreated	Sodium hydroxide	Acetric anhydride	Nitric acid	Zinc chloride
0	0.95	0.95	0.95	0.95	0.95
10	0.89	0.75	0.85	0.87	0.78
20	0.87	0.7	0.83	0.85	0.76
30	0.85	0.68	0.81	0.84	0.74
40	0.83	0.66	0.8	0.82	0.72
50	0.79	0.57	0.69	0.75	0.6

APPENDIX P
CHARACTERISATION OF COMPOSITES

I. *AMPELOCIESIS CAVICAULIS* FIBER REINFORCED COMPOSITES

	Matrix	Untreated	NaOH	Acetic Anhydride Treated	Nitric Acid Treated	Zinc Chloride Treated
Area (mm^2)	60.80	60.80	60.80	60.80	60.80	60.80
Slope length (Px)	260.93	422.86	3143.42	654.76	641.44	614.75
Initial length (mm)	100.0	100.0	100.0	100.0	100.0	100.0
Final length	115.2	105.2	106.8	106	105.2	105
Bending force (N)	1240.32	1592.96	2723.84	2121.92	1909.12	1866.56
Span length (mm)	19.00	19.00	19.00	19.00	19.00	19.00
Depth of Composite	3.2	3.2	3.2	3.2	3.2	3.2
Width of the composite (mn)	300	300.00	300.00	300.00	300.00	300.00
Brinnel bulb diameter D (mm)	10.00	10.00	10.00	10.00	10.00	10.00
Constant axial load (N)	72.45	72.45	72.45	72.45	72.45	72.45
Tensile Modulus (MPa)	429.16	695.5	5170.1	1076.90	1055.0	1011.1
Flexural Strength (MPa)	20.4	26.2	44.8	34.9	31.4	30.7
Flexural Modulus (MPa)	494.8	920	3101.8	1251.5	1106.6	1265.1
Elongation at break (%)	15.2	5.2	6.8	6.0	5.2	5.0
Hardness no	2.5	3.2	4.0	3.8	3.5	3.2
Maximum force (N)	1185.60	1421.50	2432	1958.37	1540.6	1831.90

II. *AMPELOCIESIS LEONENSIS* REINFORCED COMPOSITE

	Matrix	Untreated	NaOH	Acetic Anhydride Treated	Nitric Acid Treated	Zinc Chloride Treated
Area (mm^2)	60.80	60.80	60.80	60.80	60.80	60.80
Slope length (PX)	260.87	300.49	1010.34	1131.06	875.62	522.80
Initial length (mm)	100.0	100.0	100.0	100.0	100.0	100.0
Final length	115.2	106.8	108.1	107	106.8	105.5
Bending force (N)	1240.32	1343.68	2121.92	1538.24	1933.44	1507.84
Span length (mm)	19.00	19.00	19.00	19.00	19.00	19.00
Depth of Composite	3.2	3.2	3.2	3.2	3.2	3.2
Width of the composite (mm)	300	300.00	300.00	300.00	300.00	300.00
Brinnel bulb diameter D (mm)	10.00	10.00	10.00	10.00	10.00	10.00
Constant axial load (N)	72.45	72.45	72.45	72.45	72.45	72.45
Tensile Modulus (MPa)	429.16	494.3	1662.0	1860.6	1440.4	860.0
Tensile Strength	19.5	20.75	30.52	27.76	25.28	23.89
Flexural Strength (MPa)	20.4	22.1	34.9	25.3	31.8	24.8
Flexural Modulus (MPa)	494.8	531.1	1251.5	1200.1	1106.7	950.0
Elongation at break (%)	15.2	6.8	8.1	7.0	6.8	5.5
Hardness no	2.5	3.0	3.5	3.3	3.2	3.0
Maximum force (N)	1185.60	1261.6	1855.6	1687.8	1537.0	1452.5

III. *ADENIA LOBATA* FIBER REINFORCED COMPOSITES

	Matrix	Untreated	NaOH	Acetic Anhydride Treated	Nitric Acid Treated	Zinc Chloride Treated
Area (mm^2)	60.80	60.80	60.80	60.80	60.80	60.80
Slope length (Px)	260.95	1201.70	1346.69	1421.46	1339.21	1257.63
Initial length (mm)	100.00	100.00	100.00	100.00	100.00	100.00
Final length	115.2	105	105.8	105.7	103.8	105.2
Bending force (N)	1240.32	3064.32	3289.28	3423.04	3271.04	3173.76
Span length (mm)	19.00	19.00	19.00	19.00	19.00	19.00
Depth of Composite	3.20	3.20	3.20	3.20	3.20	3.20
Width of the composite (m)	300	300.00	300.00	300.00	300.00	300.00
Brinnel bulb diameter D (mm)	10.00	10.00	10.00	10.00	10.00	10.00
Constant axial load (N)	72.45	72.45	72.45	72.45	72.45	72.45
Tensile Strength (MPa)	19.5	36.30	40.68	42.94	40.46	37.99
Tensile Modulus (MPa)	429.16	1976.8	2215.3	2338.3	2203.0	2068.8
Flexural Strength (MPa)	20.4	50.4	54.1	56.3	53.8	52.2
Flexural Modulus (MPa)	494.8	3054.0	3278.8	3400.0	3260.0	3163.6
Elongation at break (%)	15.2	5.0	5.8	5.7	3.8	5.2
Hardness no	2.5	3.5	4.5	4.0	3.8	3.5
Maximum force (N)	1185.60	2207.04	2473.34	2610.75	2459.97	2309.79

IV. *MORINDA MORINDODIES* FIBER REINFORCED COMPOSITES

	Matrix	Untreated	NaOH	Acetic Anhydride Treated	Nitric Acid Treated	Zinc Chloride Treated
Area (mm^2)	60.80	60.80	60.80	60.80	60.80	60.80
Slope length (Px)	260.95	3945.68	4451.17	4176	4082.72	4026.6
Initial length (mm)	100.00	100.00	100.00	100.00	100.00	100.00
Final length	115.2	102	104	103.5	103.2	103
Bending force (N)	1240.32	3587.2	5016	4918.72	3872.96	3690.56
Span length (mm)	19.00	19.00	19.00	19.00	19.00	19.00
Depth of Composite	3.20	3.20	3.20	3.20	3.20	3.20
Width of the composite (mn)	300	300.00	300.00	300.00	300.00	300.00
Brinnel bulb diameter D (mm)	10.00	10.00	10.00	10.00	10.00	10.00
Constant axial load (N)	72.45	72.45	72.45	72.45	72.45	72.45
Tensile Strength (MPa)	19.5	55.99	78.36	65.93	60.51	57.65
Flexural Strength (MPa)	20.4	59.0	82.5	80.9	63.7	60.7
Flexural Modulus (MPa)	494.8	3600.0	6100.0	5800.0	4100.0	3821.0
Elongation at break (%)	15.2	2.0	4.0	3.5	3.2	3.0
Hardness no	2.5	4.0	5.5	5.2	5.0	4.5
Maximum force (N)	1185.60	3404.19	4764.29	4008.544	3679.01	13505.12
Tensile Modulus (MPa)	429.2	6489.6	7321.0	6870.0	6715.0	6622.7

APPENDIX Q

MECHANICAL PROPERTIES OF FIBER REINFORCED COMPOSITES

1. *AMPELOCISSUS CAVICAULIS*

Treatment Methods	Tensile Strength (Mpa)	Tensile Modulus (Mpa)	Flexural Strength (Mpa)	Flexural Modulus (Mpa)	Impact Strength (J/m)	Elongation At break (%)	Hardness number	Bendi at break (mm)
Matrix	19.5	429.16	20.4	494.8	40.0	15.2	2.5	16.2
Untreated	23.38	695.5	26.2	920	45.2	5.2	3.2	13.1
NaOH	40.00	5170.1	44.8	3101.8	54.0	6.8	4.0	14.2
Acetic acid	32.21	1076.90	34.9	1251.5	53.0	6.0	3.8	13.8
Nitric acid	25.34	1055.0	31.4	1106.6	52.6	5.2	3.5	13.0
Zinc Chloride	30.13	1011.1	30.7	1265.1	50.1	5.0	3.2	12.8

2. *MORINDA MORINDOIDES*

Treatment Methods	Tensile Strength (Mpa)	Tensile Modulus (Mpa)	Flexural Strength (Mpa)	Flexural Modulus (Mpa)	Impact Strength (J/m)	Elongation At break (%)	Hardness (Mpa)	Bendi at break (mm)
Matrix	19.5	429.2	20.4	494.8	40.0	15.2	2.5	16.2
Untreated	55.99	6489.6	59.0	3600.0	50.6	2.0	4.0	12.2
NaOH	78.36	7321.0	82.5	6100.0	67.1	4.0	5.5	13.5
Acetic acid	65.93	6870.0	80.9	5800.0	63.3	3.5	5.2	13.0
Nitric acid	60.51	6715.0	63.7	4100.0	62.1	3.2	5.0	12.8
Zinc Chloride	57.65	6622.7	60.7	3821.0	60.0	3.0	4.5	12.0

3. *AMPELOCISSUS LEONENSIS*

Treatment Methods	Tensile Strength (Mpa)	Tensile Modulus (Mpa)	Flexural Strength (Mpa)	Flexural Modulus (Mpa)	Impact Strength (J/m)	Elongation At break (%)	Hardness Number	Bendi at break (mm)
Matrix	19.5	429.16	20.4	494.8	40.0	15.2	2.5	16.2
Untreated	20.75	494.3	22.1	531.1	42.1	6.8	3.0	13.1
NaOH	30.52	1662.0	34.9	1251.5	47.3	8.1	3.5	15.4
Acetic acid	27.76	1860.6	25.3	1200.1	45.0	7.0	3.3	14.5
Nitric acid	25.28	1440.4	31.8	1106.7	43.1	6.8	3.2	13.1
Zinc Chloride	23.89	860.0	24.8	950.0	42.8	5.5	3.0	13.0

4. *ADENIA LOBATA*

Treatment Methods	Tensile Strength (Mpa)	Tensile Modulus (Mpa)	Flexural Strength (Mpa)	Flexural Modulus (Mpa)	Impact Strength (J/m)	Elongation At break (%)	Hardness (Mpa)	Bendi at break (mm)
Matrix	19.5	429.16	20.4	494.8	40.0	15.2	2.5	16.2
Untreated	36.30	1976.8	50.4	3054.0	48.0	5.0	3.5	12.7
NaOH	40.68	2215.3	54.1	3278.8	55.1	5.8	4.5	13.8
Acetic acid	42.94	2338.3	56.3	3400.0	54.2	5.7	4.0	13.0
Nitric acid	40.46	2203.0	53.8	3260.0	52.1	3.8	3.8	13.0
Zinc Chloride	37.99	2068.8	52.2	3163.6	50.3	5.2	3.5	12.5

APPENDIX R

CALCULATIONS FOR THE MECHANICAL PROPERTIES OF COMPOSITES

A. CALCULATION INVOLVING *AMPELOCISSUS CAVICAULIS*

1. TENSILE STRENGTH

Matrix

$$Tensile\ Strength = \frac{maximum\ force}{Area}$$

$$Tensile\ Strength = \frac{1185.6}{60.8} = 19.5\ mPa$$

Untreated

$$Tensile\ Strength = \frac{501421}{60.8} = 23.38\ mPa$$

NaOH – Treatment

$$Tensile\ Strength = \frac{2432}{60.8} = 40\ mPa$$

Acetic Anhydride – Treatment

$$Tensile\ Strength = \frac{1958.37}{60.8} = 32.21\ mPa$$

Nitric Acid– Treatment

$$Tensile\ Strength = \frac{1540.67}{60.8} = 25.34\ mPa$$

Zinc Chloride - Treatment

$$Tensile\ Strength = \frac{1831.90}{60.8} = 30.13\ mPa$$

2. TENSILE MODULUS (MPa)

$$tensile\ Modulus = Slope\ of\ (PX)X(\frac{Initial\ Length}{Area})$$

Matrix

$$Tensile\ Modulus = 260.93\ x\ \left(\frac{100}{60.8}\right) = 429.16\ mPa$$

Untreated

$$Tensile\ Modulus = 422.86\ x\ \left(\frac{100}{60.8}\right) = 695.5\ mPa$$

NaOH – Treatment

$$Tensile\ Modulus = 3143.42\ x\ \left(\frac{100}{60.8}\right) = 5170.1\ mPa$$

Acetic Acid – Treatment

$$Tensile\ Modulus = 654.76\ x\ \left(\frac{100}{60.8}\right) = 1076.90\ mPa$$

Nitric Acid – Treatment

$$Tensile\ Modulus = 641.44\ x\ \left(\frac{100}{60.8}\right) = 1055.0\ mPa$$

Zinc Chloride – Treatment

$$Tensile\ Modulus = 614.75\ x\ \left(\frac{100}{60.8}\right) = 1011.1\ mPa$$

3. FLEXURAL STRENGTH (MPa)

$Flexurl\ Strength = \frac{Bending\ \ Force}{Area}$

Matrix

$$Flexural\ Strength = \frac{1240.32}{60.8} = 20.4\ mPa$$

Untreated – Treatment

$$Flexural\ Strength = \frac{2723.84}{60.8} = 44.8\ mPa$$

NaOH – Treatment

$$Flexural\ Strength = \frac{1592.96}{60.8} = 26.2\ mPa$$

Acetic Acid – Treatment

$$Flexural\ Strength = \frac{2121.92}{60.8} = 34.9\ mPa$$

Nitric Acid– Treatment

$$Flexural\ Strength = \frac{1909.12}{60.8} = 31.4\ mPa$$

Zinc Chloride - Treatment

$$Flexural\ Strength = \frac{1866.56}{60.8} = 30.7\ mPa$$

4. ELONGATION AT BREAK (%)

$$Elongation\ at\ break = \frac{Final\ length - Initial\ length}{Initial\ length}\ x\ \frac{100}{1}$$

Matrix

$$Elongation\ at\ break = \frac{115.2 - 100}{100}\ x\ \frac{100}{1} = 15.2\%$$

Untreated – Treatment

$$Elongation\ at\ break = \frac{105.2 - 100}{100}\ x\ \frac{100}{1} = 5.2\%$$

NaOH – Treatment

$$Elongation\ at\ break = \frac{106.8 - 100}{100}\ x\ \frac{100}{1} = 6.8\%$$

Acetic – Treatment

$$Elongation\ at\ break = \frac{106 - 100}{100}\ x\ \frac{100}{1} = 6\%$$

Nitric Acid – Treatment

$$Elongation\ at\ break = \frac{105.2 - 100}{100}\ x\ \frac{100}{1} = 5.2\%$$

Zinc Chloride – Treatment

$$Elongation\ at\ break = \frac{105 - 100}{100}\ x\ \frac{100}{1} = 5\%$$

CALCULATION INVOLVING *MORINDA MORINDOIDES*

1. TENSILE STRENGTH

Matrix

$$Tensile\ Strength = \frac{maximum\ force}{Area}$$

$$Tensile\ Strength = \frac{1185.6}{60.80} = 19.5\ MP$$

Untreated

$$Tensile\ Strength = \frac{3404.190}{60.8} = 55.99\ MPa$$

NaOH – Treatment

$$Tensile\ Strength = \frac{4764.29}{60.8} = 78.36\ MPa$$

Acetic Acid – Treatment

$$Tensile\ Strength = 4008.544 = 65.93\ MPa$$

Nitric Acid– Treatment

$$Tensile\ Strength = \frac{3679.01}{60.8} = 60.51 MPa$$

Zinc Chloride - Treatment

$$Tensile\ Strength = \frac{3505.12}{60.8} = 57.65\ MPa$$

2. TENSILE MODULUS (MPa)

$$tensile\ Modulus = Slope\ of\ (PX)X(\frac{Initial\ Length}{Area})$$

Matrix

$$Tensile\ Modulus = 260.95\ x\ \left(\frac{100}{60.8}\right) = 429.20\ MPa$$

Untreated – Treatment

$$Tensile\ Modulus = 3945.68\ \ x\ \left(\frac{100}{60.8}\right) = 6489.6\ MPa$$

NaOH – Treatment

$$Tensile\ Modulus = 4451.17\ x\ \left(\frac{100}{60.8}\right) = 7321.0\ MPa$$

Acetic Acid – Treatment

$$Tensile\ Modulus = 4176.96\ x\ \left(\frac{100}{60.8}\right) = 6870.0\ MPa$$

Nitric Acid – Treatment

$$Tensile\ Modulus = 4082.72\ x\ \left(\frac{100}{60.8}\right) = 6715.0.0\ MPa$$

Zinc Chloride – Treatment

$$Tensile\ Modulus = 4026.6\ \ x\ \left(\frac{100}{60.8}\right) = 6622.7\ MPa$$

3. FLEXURAL STRENGTH (MPa)

$$Flexural\ Strength = \frac{Bending\ \ Force}{Area}$$

Matrix

$$Flexural\ Strength = \frac{1240.32}{60.8} = 20.4\ MPa$$

Untreated

$$Flexural\ Strength = \frac{3587.2}{60.8} = 59.0\ MPa$$

NaOH – Treatment

$$Flexural\ Strength = \frac{5016}{60.8} = 82.5\ \ MPa$$

Acetic Acid – Treatment

$$Flexural\ Strength = \frac{4918.72}{60.8} = 80.9\ \ MPa$$

Nitric Acid– Treatment

$$Flexural\ Strength = \frac{3872.96}{60.8} = 80.9\ MPa$$

Zinc Chloride - Treatment

$$Flexurall\ Strength = \frac{3690.56}{60.8} = 60.7\ MPa$$

4. ELONGATION AT BREAK (%)

$$Elongation\ at\ break = \frac{Final\ length - Initial\ length}{Initial\ length}\ x\ \frac{100}{1}$$

Matrix

$$Elongation\ at\ break = \frac{115.2 - 100}{100}\ x\ \frac{100}{1} = 15.2\%$$

Untreated

$$Elongation\ at\ break = \frac{102 - 100}{100}\ x\ \frac{100}{1} = 2\%$$

NaOH – Treatment

$$Elongation\ at\ break = \frac{104 - 100}{100}\ x\ \frac{100}{1} = 4\%$$

Acetic – Treatment

$$Elongation\ at\ break = \frac{103.5 - 100}{100}\ x\ \frac{100}{1} = 3.5\%$$

Nitric Acid – Treatment

$$Elongation\ at\ break = \frac{103.2 - 100}{100}\ x\ \frac{100}{1} = 3.2\%$$

Zinc Chloride – Treatment

$$Elongation\ at\ break = \frac{103 - 100}{100}\ x\ \frac{100}{1} = 3\%$$

CALCULATION INVOLVING *AMPELOCISSUS LEONENSIS*

1. TENSILE STRENGTH

Matrix

$$Tensile\ Strength = \frac{maximum\ force}{Area}$$

$$Tensile\ Strength = \frac{1185.6}{60.8} = 19.5\ MPa$$

Untreated

$$Tensile\ Strength = \frac{1261.6}{60.8} = 20.75\ MPa$$

NaOH – Treatment

$$Tensile\ Strength = \frac{1855.6}{60.8} = 30\ MPa$$

Acetic Acid – Treatment

$$Tensile\ Strength = \frac{1687.8.37}{60.8} = 27.76\ MPa$$

Nitric Acid– Treatment

$$Tensile\ Strength = \frac{1537.0}{60.8} = 25.28\ MPa$$

Zinc Chloride - Treatment

$$Tensile\ Strength = \frac{1452.5}{60.8} = 23.89\ mPa$$

2. TENSILE MODULUS (MPa)

$$tensile\ Modulus = Slope\ of\ (PX)X(\frac{Initial\ Length}{Area})$$

Matrix

$$Tensile\ Modulus = 260.87\ x\ \left(\frac{100}{60.8}\right) = 429.9\ mPa$$

Untreated

$$Tensile\ Modulus = 300.49\ x\ \left(\frac{100}{60.8}\right) = 494.3\ mPa$$

NaOH – Treatment

$$Tensile\ Modulus = 1010.34\ x\ \left(\frac{100}{60.8}\right) = 1662.0\ mPa$$

Acetic Acid – Treatment

$$Tensile\ Modulus = 1131.06\ x\ \left(\frac{100}{60.8}\right) = 1860.6\ mPa$$

Nitric Acid – Treatment

$$Tensile\ Modulus = 875.62\ x\ \left(\frac{100}{60.8}\right) = 1440.4\ mPa$$

Zinc Chloride – Treatment

$$Tensile\ Modulus = 522.80\ x\ \left(\frac{100}{60.8}\right) = 860.0\ mPa$$

3. FLEXURAL STRENGTH (MPa)

$Flexurl\ Strength = \frac{Bending\ \ Force}{Area}$

Matrix

$$Flexural\ Strength = \frac{1240.32}{60.8} = 20.4\ mPa$$

Untreated

$$Flexural\ Strength = \frac{1343.68}{60.8} = 22.1\ mPa$$

NaOH – Treatment

$$Flexural\ Strength = \frac{2121.92}{60.8} = 34.9\ mPa$$

Acetic Acid – Treatment

$$Flexural\ Strength = \frac{1538.24}{60.8} = 25.3\ mPa$$

Nitric Acid– Treatment

$$Flexural\ Strength = \frac{133.44}{60.8} = 31.8\ mPa$$

Zinc Chloride - Treatment

$$Flexurall\ Strength = \frac{1507.84}{60.8} = 24.8\ mPa$$

4. ELONGATION AT BREAK (%)

$$Elongation\ at\ break = \frac{Final\ length - Initial\ length}{Initial\ length}\ x\ \frac{100}{1}$$

Matrix

$$Elongation\ at\ break = \frac{115.2 - 100}{100}\ x\ \frac{100}{1} = 15.2\%$$

Untreated

$$Elongation\ at\ break = \frac{106.8 - 100}{100}\ x\ \frac{100}{1} = 6.8\%$$

NaOH – Treatment

$$Elongation\ at\ break = \frac{108.1 - 100}{100}\ x\ \frac{100}{1} = 8.1\%$$

Acetic – Treatment

$$Elongation\ at\ break = \frac{107 - 100}{100}\ x\ \frac{100}{1} = 7.0\%$$

Nitric Acid – Treatment

$$Elongation\ at\ break = \frac{106.8 - 100}{100}\ x\ \frac{100}{1} = 6.8\%$$

Zinc Chloride – Treatment

$$Elongation\ at\ break = \frac{105.5 - 100}{100}\ x\ \frac{100}{1} = 5.5\%$$

CALCULATION INVOLVING *ADENIA LOBATA*

1. TENSILE STRENGTH

Matrix

$$Tensile\ Strength = \frac{maximum\ force}{Area}$$

$$Tensile\ Strength = \frac{1185.6}{60.80} = 19.5\ mPa$$

Untreated

$$Tensile\ Strength = \frac{2207.04}{60.8} = 36.30\ mPa$$

NaOH – Treatment

$$Tensile\ Strength = \frac{2473.34}{60.8} = 40.68\ mPa$$

Acetic Acid – Treatment

$$Tensile\ Strength = \frac{2610.75}{60.8} = 42.9mPa$$

Nitric Acid– Treatment

$$Tensile\ Strength = \frac{2459.97}{60.8} = 40.46MPa$$

Zinc Chloride - Treatment

$$Tensile\ Strength = \frac{2309}{60.8} = 37.99mPa$$

2. TENSILE MODULUS (MPa)

$$tensile\ Modulus = Slope\ of\ (PX)X(\frac{Initial\ Length}{Area})$$

Matrix

$$Tensile\ Modulus = 260.87\ x\ \left(\frac{100}{60.8}\right) = 429.16\ mPa$$

Untreated

$$Tensile\ Modulus = 1201.70\ \ x\ \left(\frac{100}{60.8}\right) = 1976.69\ mPa$$

NaOH – Treatment

$$Tensile\ Modulus = 1346.69\ x\ \left(\frac{100}{60.8}\right) = 2215.3\ mPa$$

Acetic Acid – Treatment

$$Tensile\ Modulus = 1421.46\ x\ \left(\frac{100}{60.8}\right) = 2338.3\ mPa$$

Nitric Acid – Treatment

$$Tensile\ Modulus = 1339.21\ x\ \left(\frac{100}{60.8}\right) = 2203.0\ mPa$$

Zinc Chloride – Treatment

$$Tensile\ Modulus = 1257.63\ \ x\ \left(\frac{100}{60.8}\right) = 2068.80\ mPa$$

3. FLEXURAL STRENGTH (MPa)

$Flexurl\ Strength = \frac{Bending\ \ Force}{Area}$

Matrix

$$Flexural\ Strength = \frac{1240.32}{60.8} = 20.4\ mPa$$

Untreated

$$Flexural\ Strength = \frac{3064.32}{60.8} = 50.4\ mPa$$

NaOH – Treatment

$$Flexural\ Strength = \frac{3289}{60.8} = 54.1\ mPa$$

Acetic Acid – Treatment

$$Flexural\ Strength = \frac{3423.04}{60.8} = 56.3\ mPa$$

Nitric Acid– Treatment

$$Flexural\ Strength = \frac{3271.04}{60.8} = 53.8\ mPa$$

Zinc Chloride - Treatment

$$Flexurall\ Strength = \frac{3173.76}{60.8} = 52.2\ mPa$$

4. ELONGATION AT BREAK (%)

$$Elongation\ at\ break = \frac{Final\ length - Initial\ length}{Initial\ length}\ x\ \frac{100}{1}$$

Matrix

$$Elongation\ at\ break = \frac{115.2 - 100}{100}\ x\ \frac{100}{1} = 15.2\%$$

Untreated

$$Elongation\ at\ break = \frac{105 - 100}{100}\ x\ \frac{100}{1} = 5.0\%$$

NaOH – Treatment

$$Elongation\ at\ break = \frac{105.8 - 100}{100}\ x\ \frac{100}{1} = 5.8\%$$

Acetic – Treatment

$$Elongation\ at\ break = \frac{105.7 - 100}{100}\ x\ \frac{100}{1} = 5.7\%$$

Nitric Acid – Treatment

$$Elongation\ at\ break = \frac{103.8 - 100}{100}\ x\ \frac{100}{1} = 3.82\%$$

Zinc Chloride – Treatment

$$Elongation\ at\ break = \frac{105.2 - 100}{100}\ x\ \frac{100}{1} = 5.2\%$$