PRODUCTION OF WOOD POLYMER COMPOSITE FROM AFRICAN COPAIBA BALSAM (*Daniella oliveri*) WOOD DUST: EFFECTS OF ALKALINE CONCENTRATION AND TIME OF TREATMENT

This work was carried out to produce wood polymer composites (WPCs) based on virgin high density poly-ethylene (vHDPE) and *Daniella oliveri* wood dusts using compression molding technique. Wood dusts were treated with different concentrations of sodium hydroxide (NaOH) at varying time. The surface modification of both untreated and treated wood polymer samples were monitored by Fourier transform infrared (FTIR) spectroscopy while the changes in the structure and the properties of WPCs resulting from these treatments were explored by the scanning electron microscopy (SEM)as well as physical and mechanical tests. The experimental results indicated that the alkali treatment of the wood dust improved the interactions between the wood dust and the vHDPE matrix, and enhanced mechanical properties of the composites. It was concluded that the physical, mechanical and microstructural properties of the WPCs were optimised when wood dusts were treated with 4wt% concentration of NaOH solution for 150 minutes.

CHAPTER ONE

1.0 INTRODUCTION

1.1 Background to the Study

The quest to turn low-quality wood materials into high-quality products and the concern for environmental sanity have attracted researchers and manufacturers' interest in blending wood fibre with thermoplastic resin, which result in wood polymer composites (WPCs). This had resulted in an average yearly growth of 23% in the WPC market between 2003 and 2007 and ten times as fast as the polymer market (Alireza, 2008). Wood fibres are eco-friendly, have high specific strength and modulus, and can be gotten at a cheaper price using manual labour available in the locality. (Shinoj *et al.* 2011). In addition, they can be easily incinerated of at the end of their life cycle by upturning or by the composting of their calorific value in a furnace, which is not possible for glass fibre (Kuo *et al.* 2009). However, wood fibres also show some unwanted characteristics such as high water absorption and low heat resistance (Mylsamy and Rajendran, 2011). Thermoplastic resins, such as polypropylene (PP), polyethylene (PE), polystyrene, and polyvinyl chloride (PVC), soften when heated and harden when cooled. This property allows materials such as wood fibres, to be mixed with the plastic to form a composite product. The resulting WPCs can be easily processed into various shapes and then recycled. However, the hydrophilic wood fibres do not adhere to the hydrophobic polymers as a consequence of the poor interfacial bond between the fibres and the matrix. Therefore, in order to increase the compatibility and adhesion between the fibres and the matrix, the wood fibres are always subjected to various surface modification and treatments.

The properties of WPCs depend on the degree of interfacial bonding between the polymer matrixes e.g. virgin High Density Polyethylene (vHDPE) and the wood fibre. When wood fibres are used as reinforcement in WPCs, problems such as debinding of the wood fibres within the polymer matrix may occur at the interface due to incompatibility (Kuo *et al.* 2009). To optimise WPCs' engineering performance, researchers are currently exploring the effects of various surface modification techniques and their treatment parameters as well as additives and compatibilizers with a view to improving matrix-reinforcement interfacial bonding. Surface modification of the wood fibres by means of cost effective alkaline treatment is one of the major areas of current research to develop compatibility and interfacial bond strength in the WPCs (Kuo *et al.* 2009; Mylsamy & Rajendran, 2011).

This study makes use of wood dust from a locally available soft wood, African Copaiba Balsam wood *(Daniella-oliveri)* waste materials (from saw-mill) causing environmental and health hazards to the Nigerian society; compounded with virgin high density polyethylene (vHDPE) to produce WPCs for structural applications. *Daniella-oliveri* is widely available in Kotangora, Niger state, Nigeria.

1.2 Statement of the Problem

Wood polymer composites have gained a wide range of utilisation most especially in the construction and automobiles industries (McKeever, 1999). However, in Nigeria most of the WPC products are still imported while there are large deposits of raw materials that can be utilised in WPC production. Wood wastes generated as waste product in our saw mills create a large chunk of environmental problems because they are not usually disposed properly. Judicious use of these waste wood dusts through the production of WPCs for household structural applications will ensure that they no longer constitute environmental hazards while they are also turned to wealth.

Furthermore, by virtue of Nigeria's oil deposit, polymeric materials which are by-product of the refineries exist in large deposit as well. Therefore there is availability of major materials for the production of WPC however, before the actualization of qualitative WPC there is a need for proper treatment of the wood dust.

1.3 Significance of the Study

This work is significant as it is expected to provide most appropriate surface modification treatment parameters using NaOH for optimising the properties of WPCs. This in essence will ensure a straight forward formulation of the wood and polymer for the production of household structural engineering component (photo frame, name plate, phone casing) made of WPC by existing and emerging industries. This is a source of economic boost for Nigeria as most imported WPC materials will now be produced locally, hence, a huge saving on Nigeria's foreign exchange earnings. Employment creation, proper waste management which transform to better health status for Nigerians, conversion of waste to wealth, and a more stable economy for Nigeria are among the benefits of carrying out this research.

1.4 Aim and Objectives of Study

This research work aims at investigating the effect of varying concentration and time of NaOH treatment on the physical, mechanical, and microstructural properties of WPCs obtained by reinforcing vHDPE with African Copaiba Balsam (*Daniella-oliveri*) wood saw dust. The specific objectives are to determine:

(a) The effect of the concentration of NaOH on the physical, mechanical and microstructural properties of WPC produced by blending vHDPE and wood dust from *Daniella-oliveri*.

(b) The effect of the time of treatment of NaOH on the physical, mechanical and microstructural properties of WPC produced by blending vHDPE and wood dust from *Daniella-oliveri*.

(c) The most appropriate surface modification NaOH treatment parameters for optimising the properties of the WPC.

1.5 Scope and Limitation of the Study

The research work is based on alkalisation treatment of wood dust and then compounding with virgin high density polyethylene (vHDPE) to produce composite samples using compression technique. The samples produced were subjected to tensile, density, moisture absorption, Fourier Transform Infra Red (FTIR) and Scanning Electron Microscopy (SEM) tests in other to obtain the optimum NaOH concentration and time of treatment.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Definition and Historical Developments of Wood Polymer Composites

Wood polymer composites (WPCs) are thermoplastically processible composites that consist of varying contents of reinforcing wood fibres/dust, polymers, and additives. They are processed by thermoplastic shape forming techniques such as extrusion, injection moulding, rotor moulding or press (Kristiina and Mohini, 2008). WPCs are very promising and reliable green materials that are produced for their durability without containing toxic chemicals.

The historical development of WPCs reveals that they were first produced in Italy in the 1970’s. Since then, WPCs have become popular mostly in Northern America and other parts of the world. This popularity had been influenced by the emerging interest of researchers in the study of WPCs because of its application in the manufacturing of construction, industrial and automotive products. Advanced Environmental Recycling Technology (AERT), a division of Mobil Chemical Company, Virginia (USA), in the early 1990’s also began the production of solid wood polymer composites consisting of approximately 50% wood fibre in polyethylene. The WPCs produced were used largely as check boards, landscape timbers, tables, and floor tiles (Youngquist, 1995). Furthermore, the first international conference on wood polymer composite was convened in 1991 in Madison, Wisconsin, USA, with the intent of bringing researchers and industrial representatives from both the plastic and forest products industries to share ideas and technology on wood polymer composites (Clemons, 1998). The conference led to Anderson Corporation, Bayport, Minnesota beginning the production of wood fibre-reinforced polyvinyl chloride (PVC) used for French doors. This product contains 40%

wood in PVC extruded to net shape (Schut, 1999). Also, an aftermath of the above development led to the recycling of waste from both wood and plastic processing operation for the production of wood polymer composites. Today, a lot of studies have been carried out and much research is still on-going in other to further improve the quality performance of WPCs worldwide. In the North America, WPCs are used mostly for decking products and outdoor floor coverings. Also, profiles from WPCs are processed to produce skirting boards, pipes, window frames and many more appliances. In Europe, the automobile industries make use of about 50,000 tons of WPC as door trims, rear shelves, dash boards, claddings and body. (www.jeluplast.com). A search through the available literature reveals that Nigeria has not explored the benefit of abundant wood forests resources and petroleum-derived polymers from her large crude oil deposit, this is evident as products that could be developed using produce of WPC (floor tiles) are still imported.

In summary, the first generations of WPCs have evolved from the blending of recycled wood chips and flour binders for undemanding applications to the new generations of WPCs that are used for high performance products of complex geometry with high dimensional stability, and good mechanical properties. The new generation WPCs are produced by compounding wood flour or fibre and polymers to produce materials that can be processed like a conventional plastic but have the best features of wood and plastic. It is worthy of note that the developments of WPCs have not been without impediment from the perspectives of forest product and the plastic industries. For instance, wood properties such as its low bulk density, low specific gravity, low thermal stability as well as its tendency to absorb moisture have had adverse effects on the properties of WPCs such that, it absorbs more moisture and cannot withstand heat. However, there was a great turn around as awareness on WPCs increased, thus, bringing about success in the processing of several WPC products via greater understanding of wood fillers, and development in compounding equipment, improvement of additives as well as emerging opportunities in large volume application.

2.2 Classification of Composite Materials

Composites are materials formed from two or more distinct materials, for example, a polymer matrix with wood fibre, to obtain specific properties that are superior to the individual components (www.utem.edu.my).

Composites are commonly classified based on the matrix materials and material structure. The classification based on matrix materials include metal matrix composite, ceramic matrix composite and polymer matrix composite while that of material structure has the particulate composite and fibrous composite (Figure 2.1).

2.2.1 Metal Matrix Composite (MMC)

Metal matrix composite (MMC), as the name implies, consists of particles surrounded by a matrix of metal. MMC produces composites of very high stiffness and strength as well as high temperature resistance. Despite the fact that MMC has other advantage like better abrasion resistance, creep resistance, dimensional stability, non-flammability and resistance to degradation of fluids, it is still limited in application due to its much higher weight and cost of production. Major matrix materials used in MMC are aluminium, titanium, magnesium and copper while silicon carbide and alumina are used as reinforcements. MMCs are commonly manufactured via powder metallurgy processing techniques, vapour deposition, diffusion bonding and infiltration of metal into the fibre bundles under pressure (www.utem.edu.my).

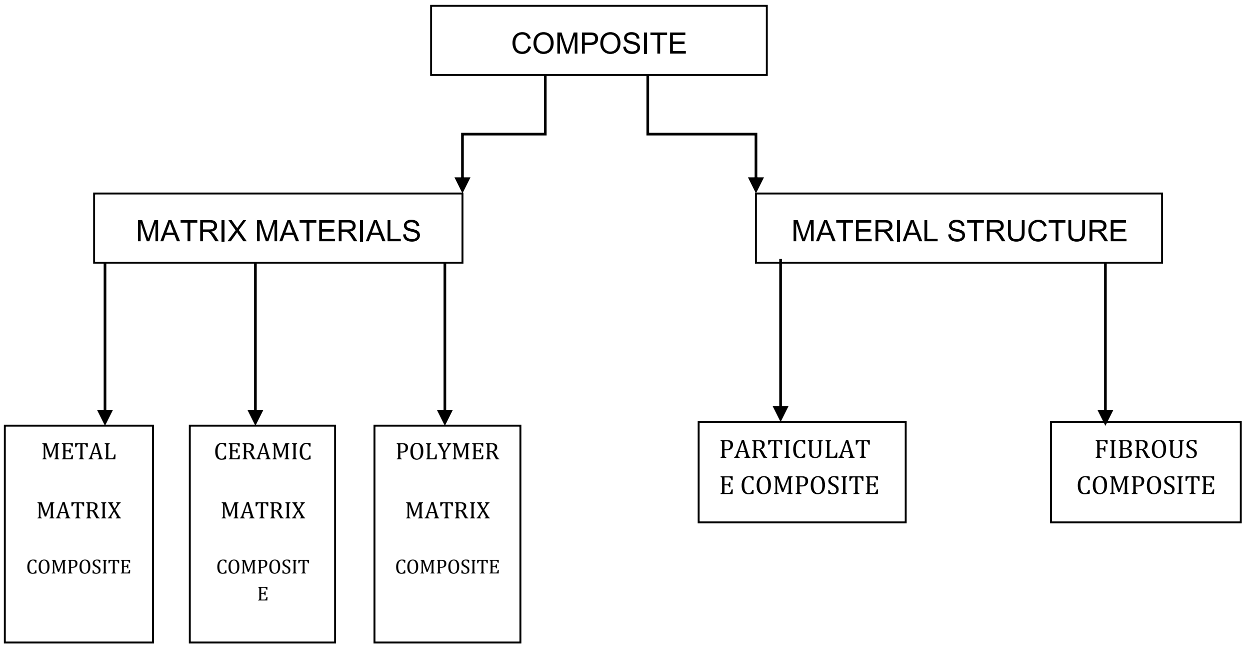


Figure 2.1: Common classification of composites (www.utem.edu.my)

2.2.2 Ceramic Matrix Composite (CMC)

Ceramic matrix composites uses ceramic materials as the binding materials and it has principal advantages over other materials in the area of their resilience to oxidation and deterioration at elevated temperatures, high melting points and high compressive strengths but, unfortunately, ceramic matrix composites are susceptible to brittle fracture hence, they have relatively low values of fracture toughness (www.jeluplast.com).

2.2.3 Particle Reinforced Composites

Particulate reinforced composite is a form of the material structure classified composites with concrete being a good example. The aggregate of coarse rock or gravel is embedded in a matrix of cement. Particle reinforcing in composites is less effective means of strengthening than fibre reinforcement. Particulate reinforced composites achieve gains in stiffness primarily, but also can achieve increases in strength and toughness. In all cases the improvements are less than would be achieved in a fibre reinforced composite. Particulate reinforced composite find applications where high levels of wear resistance are required such as road surfaces. The hardness of cement is increased significantly by adding gravel as reinforcing filler. Particulate reinforced composite has principal advantage in their low cost, ease of production, and forming (www.wikipedia.org).

2.2.4 Fibre Reinforced Composite

Fibre reinforced composites are composed of fibres and matrix (Figure 2.2). The fibres are the reinforcement and the main source of strength while the matrix glues all fibres together in shape and transfer stresses between reinforcing fibres. Fibre reinforced composites are classified based on the structural arrangement of the fibre in the composite mix as either, continuous (long fibres) or discontinuous (short fibres).

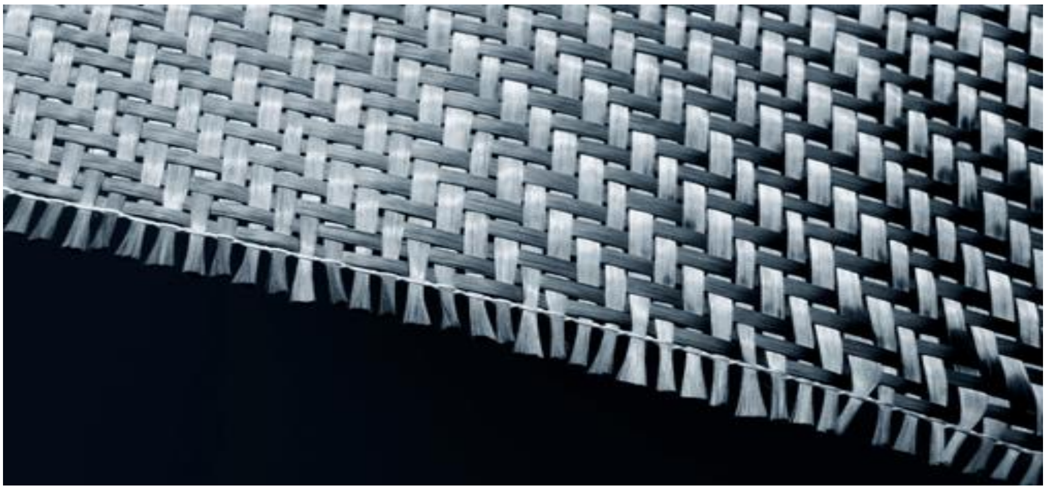


Plate I: Fibre reinforced composite (www.altairenlightening.com/thermoplastic)

Aligned fibres (Plate I) provide maximum strength which occurs only in the direction of alignment. Composite is considerably weaker along the other directions and is therefore highly anisotropic (www.altairenlighten.com).The weakness along the other direction (anisotropy) can be overcome by randomly aligning fibres in all directions. Although, this is less effective strengthening technique but has tremendous advantage of increased formability and reduced cost. Hence, components requiring strength in a particular direction can use aligned fibres while those requiring strength in more than one direction will use randomly oriented fibres (www.altairenlighten.com).

Fibre is the basic element of wood-based composites; WPC composed of many fibres with very large particles. Majority of the fibres used in composites are natural fibres which is hydrophilic in nature as they are lignocellulosic, they contain strongly polarized hydroxyl groups (Cordeiro *et al.*, 2011). These natural fibres are inherently incompatible with hydrophobic polymers such as polyethylene because of their poor interfacial adhesion between the hydrophilic fibres and hydrophobic matrix. Thus, it is important that the natural fibres be subjected to modification and treatments in order to increase the compatibility and adhesion between the fibres and matrix (Jacob *et al.*, 2008).

2.3 Fibre Reinforced Polymer Matrix Composite

Common fibre reinforced composites are composed of fibre and matrix as noted earlier on. The fibres are the reinforcement and the main source of strength while the matrix glues all fibres together in shape and transfer stresses between reinforcing fibres. In a fibre reinforced polymer composites, the matrix material is the polymer (polyethylene, polypropylene, polyvinyl chloride) which acts as the glue that joins the fibre (wood dust) together in order to give strength to the composite.

2.3.1 The Polymer Matrix

Polymers are chemical compounds with large molecules made of many smaller molecules of the same kind which either exist naturally (natural polymers) and others are produced in laboratories and factories (synthetic polymer). The small molecules (monomers) are linked together to form long chains (Figure 2.2) also known as macromolecules (Stolf and Lahr, 2004). The process whereby monomers are joined to form polymers is called polymerization. Polymers are either used as fibres (reinforcement) or as the matrix (gluing agent) in the production of composites, however, this work is considering the polymer as the matrix in a wood (fibre) plastic (polymer) composite.

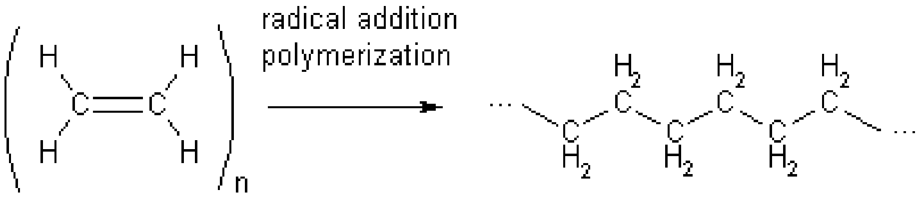


Figure 2.2: Polymerisation of ethene to polyethene (www.google.com)

2.3.2 Types of Polymers

Basically, there two types of polymer, natural and synthetic (Figure 2.3). Synthetic polymer is commonly and widely used for composites, and it is produced as plastics and rubbers. Plastics are rigid materials at service temperature while rubbers are flexible, low modulus materials which exhibit long-range elasticity. Plastics are subdivided into thermoplastics and thermosets. Thermosets are polymers which become irreversibly hard on heating; they are materials in which the long chains are linked together by crosslinks. Thermoplastics on the other hand, are polymeric materials which soften and harden reversibly on heating and cooling respectively. This account for why scrap thermoplastics can be re-used hence, a good justification for the use of thermoplastic for this work.

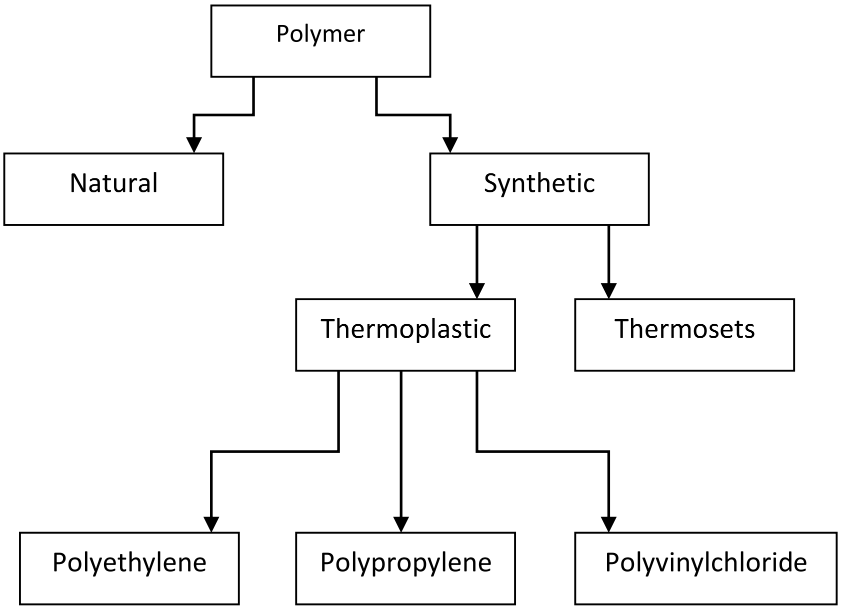


Figure 2.3: Common types of polymer (www.wikipedia.org)

2.3.2.1 Structure and Properties of Polyvinylchloride (PVC)

Figure 2.4 shows a monomer of the polyvinylchloride (PVC) also known as vinyl. It is one of the largest volumes of synthetic thermoplastics in use globally. PVC is a polymer constructed with repeating vinyl groups having one of their hydrogen replaced with a chloride group.

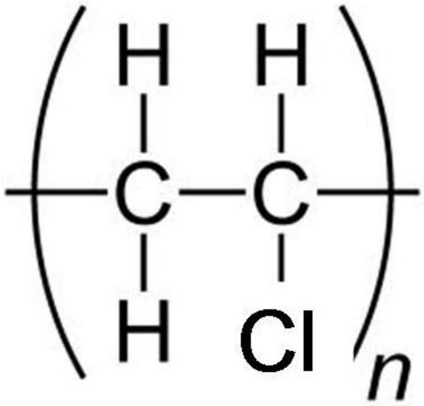


Figure 2.4: Vinyl Chloride monomer structure (C_2H_3Cl) (www.wikipedia.org)

The PVC is used for sewage pipelines and other pipeline application because of its biological and chemical resistance to sewage. Also, because rigid PVC products pass fire standard testing such as UL – 94 V – O and its excellent performance in combustion; it is widely used as insulator for electrical cables, upholstery mattresses, wall linings and floor covering. However, PVC is not without constraint. Figure 2.5 shows the polymerization which led to the production of alternating co-polymer of vinyl chloride called polyvinylchloride peroxide which can detonates upon impact.

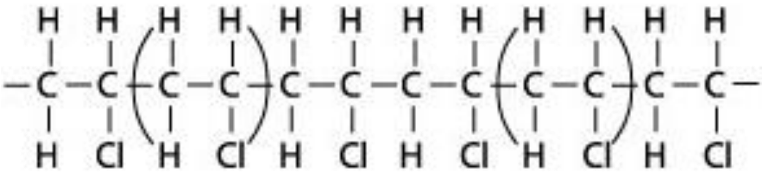


Figure2.5: Polyvinyl chloride structure. (www.wikipedia.org)

2.3.2.2 Structure and Properties of Polypropylene

Polypropylene also known as polypropene is an addition polymer made from a monomer called propylene (Figure 2.6). Polypropylene (PP) is highly resistant to many chemical solvents, bases and acids, hence, its suitability for a variety of application (Shinoj *et al.*, 2011). PP is used mainly in packaging, stationary, laboratory equipment, automobile parts, loudspeakers and polymer banknotes. The melting of polypropylene occurs as a range, thus, the determination of the melting point of PP is by finding the highest temperature obtained in a differential scanning calorimetry chart. The isostatic melting point of polypropylene is 171^0C. The manufacturing process for polypropylene includes molding and extrusion while injection moulding is the most common shaping technique. The physical finishing techniques used for PP are majorly machining and surface treatment.

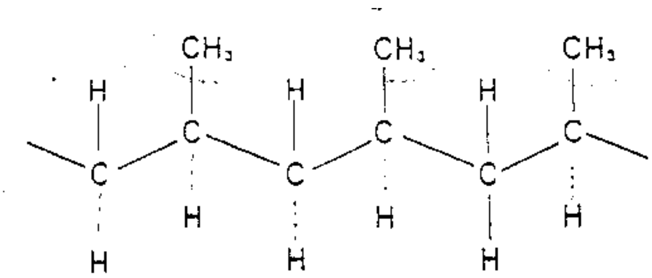


Figure 2.6: Molecular structure of polypropylene (www.wikipedia.org)

2.3.2.3 Structure and Properties of Polyethylene

Polyethylene (PE) also called polythene is the most common plastic polymer. It consists of extended hydrocarbon chains which depend on the crystal-like and the molecular heaviness as shown in Figure 2.7. The melting position and glass conversion may or may not be visible. The most common PE for commercial applications has melting points ranging from 120^0C to 130^0C. It is used widely in the packaging industries. PE has the compound formula $(C_2H_4)_nH_2$ and it is a mix of organic compounds that differ in terms of value of n where n represents the number of monomers. The polymerization of ethylene (C_2H_4) is extremely exothermic, that is, the procedure releases a lot of high temperature, this is because, ethylene (monomer) is a highly steady particle that polymerizes only upon getting in touch with chemical reagent. The common polyethylene includes high density polyethylene (HDPE) and low density polyethylene (LDPE) grades which have excellent chemical resistance. They are not affected by strong acids or strong bases. HDPE and LDPE are resistant to gentle oxidants and reducing agents and are also recyclable. However, polyethylene is not generally biodegradable without some special treatment. About 1kg of HDPE is made from 1.75kg of petroleum in terms of energy and raw materials. It is commonly recyclable with density ranging from 0.93 to 0.97 g/cm^3. HDPE has advantage of no – branching in its nomenclature, thus giving it stronger intermolecular forces and improved tensile strength over LDPE; despite the fact that LDPE has lower marginal density. HDPE has higher specific strength, because, the difference in its strength exceeds the difference in density (www.wikipedia.org).

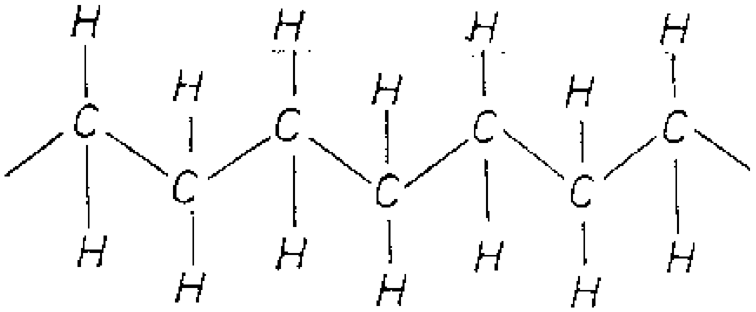


Figure 2.7: Molecular structure of polyethylene polymer (www.google.com)

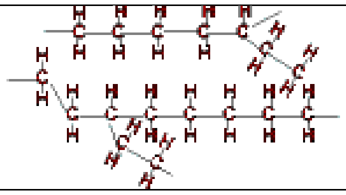
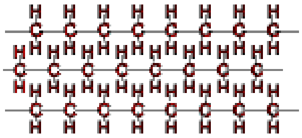
High density polyethylene is harder, more opaque and can withstand higher temperatures, say about 120°C for short periods and 110°C continuously (www.wikipedia.org). Due to the above merits and in addition to its high resistance to different solvents, HDPE is used in a variety of applications including the most important; the production of composites materials. For example, snowboard rails and boxes, toys, automobile parts, packaging materials water pipes, telecom ducts, wood plastic composites etc.

Tables 2.2 and 2.3 summarily compare the properties, merits, and demerits of polymers and the properties of the two types of polyethylene for structural applications respectively. On the basis of the comparative analysis carried out, HDPE is hereby chosen for this study because of the merits discovered in its properties (melting temperature, density and re-usability).

Table 2.1: Comparative analysis of the properties, merits, and demerits of various polymers for structural applications (Source: www.ausetute.com.au)

	PVC	PP	PE
Properties	➢ Melting points ranges from 100^0c to 260^0c ➢ Has superior fire retarding characteristics because of its high ignition temperature (455^0c) ➢ Low heat energy released during burning ➢ Density ranges between 1.1 – 1.45g/cm^3	➢ Isostatic melting point of polypropylene is 171^0C. ➢ Density range 0.855 – 0.946g/cm^3	➢ Melting points range from 120^0C to 130^0C. ➢ Excellent chemical resistance. ➢ HDPE has improved tensile strength over LDPE ➢ Density ranges between 0.91-0.97 g/cm^3
Merits	➢ Used in sewage pipelines and other pipeline application due to its biological and chemical inertness, and fire resistance. ➢ Used as insulator for electrical cables, upholstery mattresses, wall linings and floor covering	➢ Used in packaging, stationary, laboratory equipment, automobile parts, due to its high resistance to chemical solvents, bases and acids. ➢ Used as insulators	➢ Used widely in packaging, toys, automobile parts, and water pipes ➢ Cable insulation and extrusion coating. ➢ Used as liners for tanks and ponds and moisture barriers in construction.
Demerits	➢ Poor impact behavior if polyvinylchloride peroxide is formed during production.	➢ Has tendency to shrink after woven to pattern ➢ Resistance to addition of paint or ink when cooled	➢ They are not biodegradable without some special treatment.

Table 2.2: Properties of Polyethylene (Source: www.ausetute.com.au)

Property	LDPE	HDPE
Crystallinity	Low crystallinity (50-60% crystalline) Main chain contains many side chains of 2-4 carbon atoms leading to irregular packing and low crystallinity (amorphous)	Highly crystalline (>90% crystalline) contains less than 1 side chain per 200 carbon atoms in the main chain leading to long linear chains that result in regular packing and high crystallinity
Flexibility	More flexible than HDPE due to lower crystallinity	More rigid than LDPE due to higher crystallinity
Strength	Not as strong as HDPE due to irregular packing of polymer chains	Stronger as a result of regular packing of polymer chains
Heat Resistance	Retains toughness and pliability over a wide temperature range, but density drops off dramatically above room temperature.	Useful above 100°C
Transparency	Good transparency since it is more amorphous (has non-crystalline regions) than HDPE	Less transparent than LDPE because it is more crystalline
Density	0.91-0.94 g/cm^3 lower density than HDPE	0.95-0.97 g/cm^3 higher density than LDPE
Chemical Properties	Chemically inert Insolvent at room temperature in most solvents. Good resistance to acids and alkalis. Exposure to light and oxygen results in loss of strength and loss of tear resistance.	Chemically inert
Schematic diagram		
Uses	sandwich bags, cling wrap, car covers, squeeze bottles, liners for tanks and ponds, moisture barriers in construction	freezer bags, water pipes, wire and cable insulation, extrusion coating

2.4 Plant Based Fibre

Fibres used in WPC production can be divided into two groups: namely; artificial fibres and natural fibres. Natural fibres present the possibility to convey improved added worth, capability, renewability and lesser price particularly in the auto manufacturing company. Also, the natural fibres are divided into various groups based on their origins e.g., plant based, animal based, and mineral based. The plant fibre, upon which our work is dependent is composed of cellulose and can be divided according to part of the plant they are acquired from (Alireza, 2008). Plant fibre includes stem, soft sclerenchyma fibre, leaf, seed, fruit, root, grassland, cereal, straw and timber (Peijs *et al.* 2002).

The place of wood as filler in the wood polymer blend to form composite materials for structural uses cannot but be discussed. Production of wood packed polymer resources has been a manufacturing process for almost 80 years (Douglas and David, 2004). Most of the initial engineering utilization of wood packed polymers happened in thermosetting resins systems such as phenol formaldehyde resins. Manufacturing utilizations of lignocellulosic packed thermoplastics were announced in the late 1960s (Rowell *et al.*, 2002). A great pact of development was experienced in the 90s, and industrial invention of wood packed thermoplastic matrix became more widely increased. Douglas and David (2004) reiterated that, the biggest concentration of wood packed thermoplastic industries centres on the manufacture of wood polymer composites (WPC) to replace the expensive treated and preserved wood used as decking materials.

2.4.1 The Wood and its Structure

Wood is a hard fibrous structural tissue found in the stem of trees and other woody plants (www.wikipedia.org/wiki/wood). Wood is an organic material that is made of natural composites of cellulose, hemicelluloses and lignin. Just as the wood performs support function for a living tree so also wood fibre performs support or reinforcing function in a wood polymer composite. Wood is commonly classified as soft and hard wood. The soft wood category is obtained from conifers such as pine, cedar and fir and tends to be yellowish or reddish in colour (www.dummies.com). They are relatively cheaper compared to hard woods. Hard woods on the other hand are obtained from dicotyledons meaning broad-leaved trees such as oak, mahogany, cherry and tend to appear in reddish brown and almost white colours.

A typical wood structure is composed of heterogeneous, hygroscopic, cellular and anistropic material. The cells bear in its walls composition of micro-fibrils of cellulose (40% - 50%) and hemicellulose (15% - 25%) as well as lignin (15% - 30%) (www.wikipedia.org). the structure of the softwood is much more uniform in the sense that, the wood cells are mostly of one kind. In hardwoods, the structures are much more complex which requires it to be classified into different division for proper understanding.

Cellulose is an organic compound. It is a structural component of the primary cell wall of green plant. It is hydrophilic, insoluble in water and most organic solvents and biodegradable. Cellulose can be broken down by treating it with concentrated acid at high temperature. Figure 2.8 shows a strand of cellulose revealing the hydrogen bonds within and between the cellulose molecules.

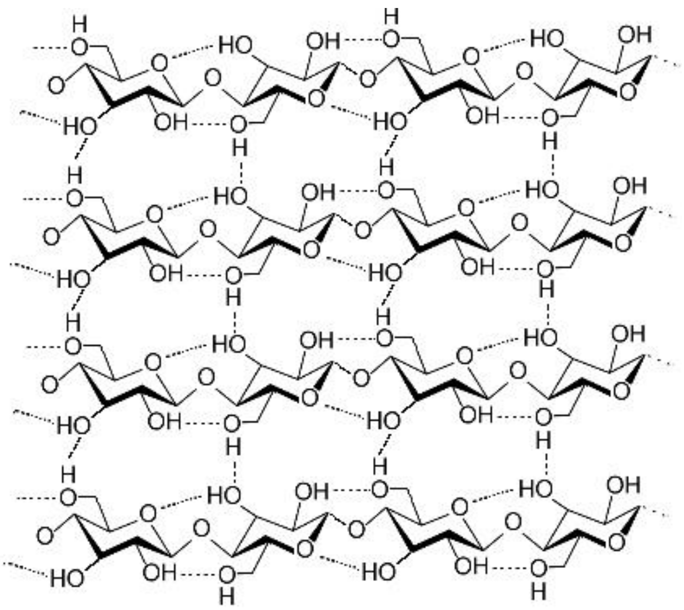


Figure 2.8: A strand of cellulose showing the hydrogen bonds (dashes) within and between cellulose molecules (www.wikipedia.org)

Hemicellulose is present alongside cellulose in virtually all plant cell walls. It has a random, amorphous structure with little strength which can easily be hydrolysed by dilute acid or base. Another important composition of the wood is the lignin. Lignin is a complex chemical compound which is an integral part of the secondary cell wall of a plant. It is hydrophobic and aromatic in nature (www.wikipedia.org/wiki/lignin). The hydrophobic lignin network affects the properties of other network such that, it acts as a coupling agent and increases the stiffness of the cellulose and hemicellulose composition (Jayabal *et al.* 2012).

2.4.2 Factors Affecting the Properties of Wood Polymer Composites

Wood fibre can be obtained from soft wood or hard wood and they have certain properties which makes them preferable for use in WPCs over other reinforced fibres. Due to the quest for more ecologically friendly materials for engineering and other applications, the ecological character of wood fibre stands it out as reinforcement in WPC due to its biodegradability, lower cost, non-abrasive nature, high potential filling level, less power utilization, large specific characteristics, and low mass (Alireza, 2007). However, certain shortcomings such as propensity to form aggregates during the making, low heat steadiness, low opposition to dampness and periodic quality changes greatly decrease the prospective of wood filled fibres to be used as strengthening material in WPCs (Bismarck *et al*. 2006; Kim *et al.* 2006). High water absorption of plant fibre results to bulging and occurrence of voids at the boundary, which leads to poor mechanical characteristics and reduces measurement unsteadiness of composites. Hence, the need for wood fibre treatment.

Another major drawback of the wood fibre is the bad compatibility shown between the fibres and the polymer matrix, which results in non-uniform spreading of fibres inside the matrix and bad mechanical characteristics. Forest product and synthetic polymer do not mix. Most polymers, particularly thermoplastics, are non-polar that is hydrophobic (water repelling) substances which are not well-matched with polar substances that is, hydrophilic (water absorbing), thus, the reason for poor union between the plastic and wood fibre in WPC (Alireza, 2007). However, in order to advance the similarity and bond connecting the wood fibre and polymer matrix in the manufacture of WPC, treatment and modification of the wood fibre is very necessary. That is why this work is considering the modification and treatment of the wood dust for optimum performance. Other factors that affect the properties of WPC include; variation in wood species, variation in wood powder content, variation in wood powder particle size and type of chemical modification or surface treatment.

Jeamtrakull *et al.* (2012) explored the influence of varieties and contents of wood flour namely: Xylia kerrii Craib (XK), Hevea brasiliensis Linn (HB), and Mangifera indica Linn (MI) on the physical, mechanical and deteriorating characteristics of WPCs in which a fixed content of E-chopped strand glass fibre (GF) was included into poly(vinyl chloride) (PVC). The outcome of their study on the mechanical and material characteristics of WPC devoid of and with GF is presented in Table 2.4. It is evident that the disparity in the mechanical characteristics and deteriorating behaviour of WPCs were dependent upon the aspect relation and components of the wood particles, and the preliminary wood hardness standards of the three woods utilized. The hardness of WPVC composites did not change with increasing wood content, whereas the flexural characteristics of WPVC composites tended to amplify with wood substance up to 40 % (Table 2.4) with MI imparting the WPCs with the maximum flexural characteristics.

The wood component with high aspect relation (Xylia kerrii Craib and Mangifera indica Linn.) acted as strengthening by dropping the specific deteriorating rate of PVC. The best of wood substance for maximum deteriorating resistance was established on the constituents of wood, aspect fraction and descending distance. Xylia kerrii Craib at 40 phr (portable hardness Rockwell) gave the minimum specific deteriorating rate for WPVC composites at a descending distance of 2.0 km, whereas Hevea brasiliensis Linn gave the lowest possible definite deteriorating rate for the glass fibre toughened WPVC composites. The inclusion of glass fibre better the flexural modulus and force by 52–129% and 21–93%, in that order, as well as the deterioration resistance. The consequence of glass fibre was more prominent at a descending distance of 2.0 km. The higher the descending distance, the more the definite deteriorating rate of the WPVC composites, for WPVC model both inclusive and exclusive of E-glass fibre.

Furthermore, Leu *et al.* (2012) evaluated the effects of wood flour constituent part; coupling agent quantity; lubricant substance; and the mass relation of wood and recycled polypropylene (RPPs). The outcome of their study confirmed that the use of finer wood flour (smaller than 125µ m) tend to decrease the swelling owing to wetness adsorption while improving the tensile and flexural potency of WPCs (Figure 2.9).

Table 2.3: Results of types and contents of wood dust on the mechanical and physical properties of WPCs exclusive and inclusive of glass fibres (GF) (Jeamtrakull *et al.* 2012)

Wood type	Hardness HV	Flexural Modulus (MPa)	Flexural Strength (MPa)	Roughness (µm)

		Without GF	With GF (10%)	Without GF	With GF (10%)	Without GF	With GF (10%)	Without GF	With GF (10%)
XK	0	78	80	1219	2407	17	39	3.2	3.5
	20	78	81	1765	3253	33	49	3.8	3.8
	40	81	82	2081	3344	34	49	4.1	3.7
	60	81	83	2103	3425	32	51	4.3	4.0
HB	0	78	80	1219	2407	17	39	3.2	3.5
	20	78	80	1755	2896	33	51	4.0	3.5
	40	79	82	1936	3175	32	51	4.4	3.7
	60	80	82	1576	3604	25	48	4.5	3.9
MI	0	78	80	1219	2407	17	39	3.5	3.5
	20	81	80	1778	2798	30	41	4.2	4.2
	40	82	83	2251	3416	38	46	4.0	4.0
	60	82	83	2130	3545	33	45	4.2	4.2

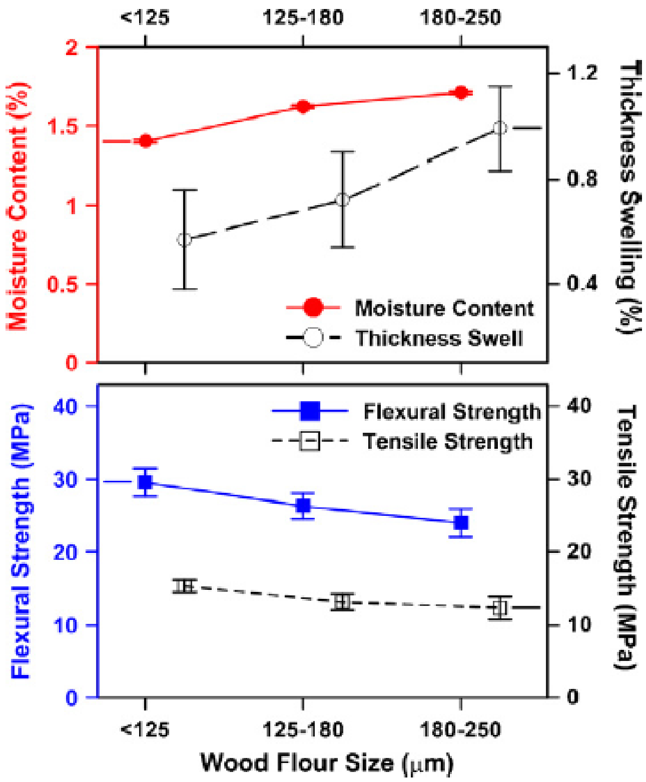


Figure 2.9: Results of varying wood flour sizes on the physical and mechanical characteristics of WPCs (Leu *et al.*,2012).

Moreover, it was proved that the addition of an appropriate quantity of pairing agent (Figure 2.10) can increase the mechanical properties and considerably reduce the bulging, but increasing the lubricant extensively increased bulging and decrease all the mechanical characteristics of the WPCs (Figure 2.11) with the optimal concentrations of the combination agent (Maleic Anhydride Polypropylene) and emollient (Zinc stearate) in WPCs found to be 3%.

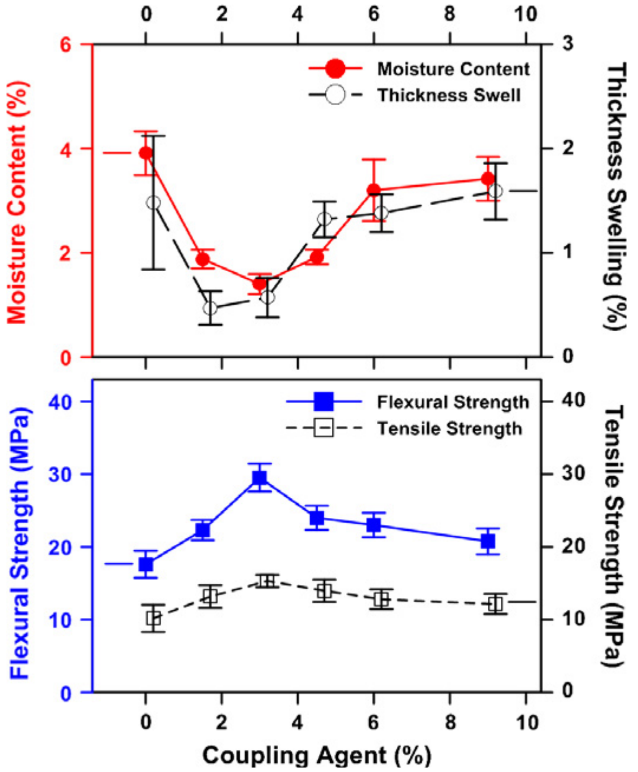


Figure 2.10: Results of varying mass of pairing agent on the physical and mechanical characteristics of WPCs (Leu *et al.*, 2012).

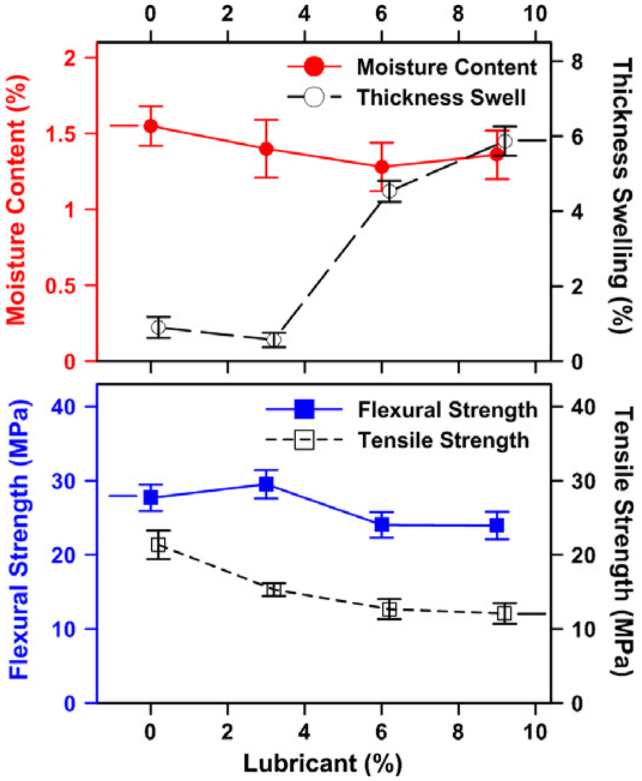


Figure 2.11: Results of varying lubricant mass on the physical and mechanical characteristics of WPCs with extruded moulding (Leu *et al.*, 2012).

Finally, Figure 2.12 reveals that maintaining the wood substance at 50% or a smaller amount produced a superlative mechanical characteristics, while wood substance in the region of 50% resulted in decline of all physical and mechanical characteristics of WPCs. Leu and co-investigators concluded that by dropping thickness of puffiness from water adsorption, or decreasing the contact of wood fibres to ambience, improvement in the mechanical performances of WPCs can be achieved. This finding will be immensely useful in optimising the properties of WPCs to be produced from this study.

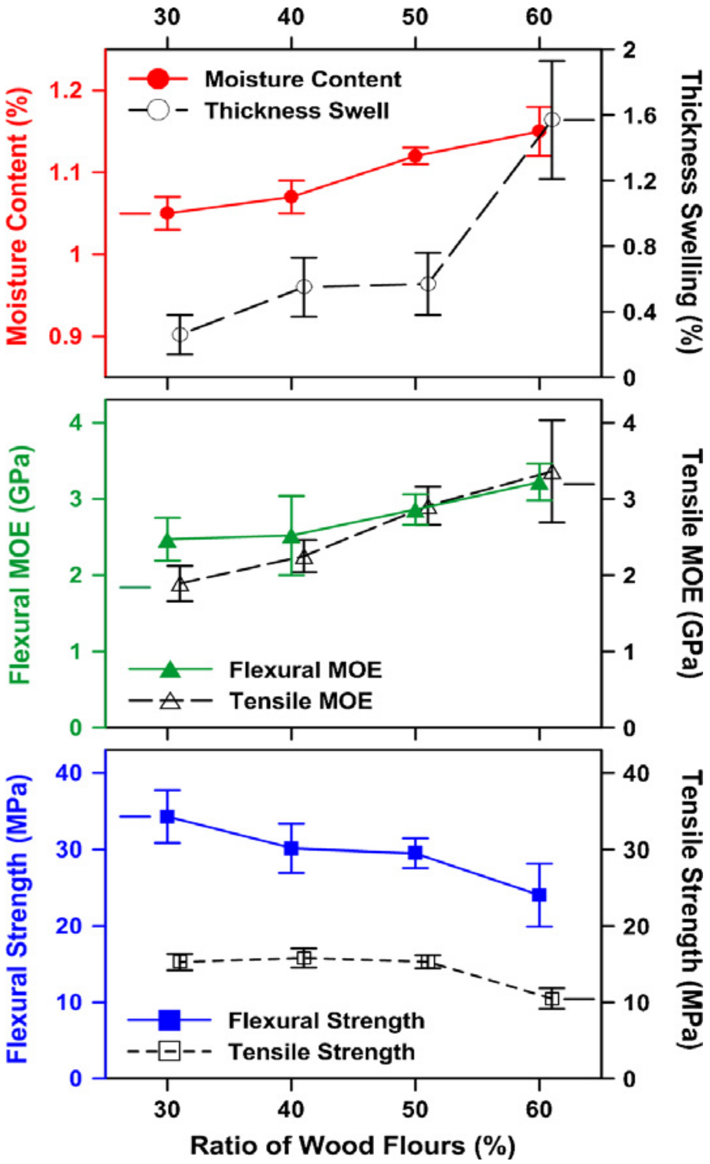


Figure 2.12: Results of varying wood/PP portion to the physical and mechanical characteristics of WPCs with extruded moulding (Leu *et al.*,2012).

2.5 Modification and Surface Treatment in Wood Polymer Composite

2.5.1 Effect of Modification Treatment on the Physical and Mechanical Properties of Wood Polymer Composites

Chemical modification is very necessary for filler materials in wood polymer composites in order to improve the interfacial adhesion between the filler particles and the polymer matrix. However, this is not without additional cost implication.

Previous research reveals that the inclusion of unprocessed filler to a plastic matrix without any treatment might reduce the quality of the physical and mechanical properties in the produced WPC, most especially when there is low length to diameter ratio. Table 2.4 shows the mechanical properties obtained from recycled polyethylene based green composite exclusive of the use of some coupling or chemical variation (LaMantia and Morreale, 2011). The same LaMantia and Morreale, (2011) further established from their research and presented the trends of the major mechanical characteristics upon simple inclusion of 30wt%, less aspect relation natural-organic fillers to a recycled polyethylene (RPE) in Table 2.4. The idea here is to establish that, producing WPC without any adhesion but, taking advantage of the strength of the natural organic filler improves the strength of the produced composite material. However, this is not without adverse effect in terms of swelling of the WPC once the organic filler is increased above 50% (Leu *et al.* 2012), the physical properties are compromised. Although, the wood waste which this research intends to use up, is put into meaningful maximum utilisation and cost of adhesive exempted from the overall production cost. The Table 2.4 clearly shows that the tensile strength of the RPE with coarse wood particles (SDc) as filler is 10.9MPa.This result was compared to the physical and mechanical properties obtained in this research where modification and surface treatment was carried out on the wood dust before the production of WPCs without the use of adhesives.

Table 2.4: Major mechanical characteristics of RPE-based green composites exclusive of the use of any adhesion promoter /chemical variation technique (LaMantia and Morreale, 2011)

Characteristics	RPE	RPE-OS	RPE-SDc	RPE-SDf	RPE-SS
Elastic modulus (MPa)	129	163	264	282	193
Tensile strength (MPa)	12.5	8	10.9	10.4	9.9
Elongation at break (%)	100	36	29	17	45
Heat deflection temperature (^{0}C)	27	35	38	42	37
Impact strength (J/m)	No break	No break	185	99	No break

(RPE=Recycled polyethylene; OS=Olive stone flour; SDc=Coarse wood flour; SDf=Fine wood flour; SS=Sago starch)

2.5.2 Effects of Types of Surface Modification Treatments on the Physical and Mechanical Properties of Wood Polymer Composites

There are different types of surface modification treatments utilised in order to achieve optimal properties in WPCs. Amongst them are, alkalisation, benzylation, acetylation, peroxide treatment, silane treatment, etc (LaMantia and Morreale, 2011). Diverse blending materials have also been used to treat polymeric matrix and develop the interfacial strength and consequently the mechanical characteristics of the WPCs products. Commonly reported coupling agents in the literature include maleic anhydride grafted styrene-ethylene-butylene-styrene (SEBS-g-MA), maleic anhydride grafted high density polyethylene (HDPE-g-MA), maleic anhydride grafted polypropylene (PP-g-MA) and maleic anhydride grafted low density polyethylene (LDPE-g-MA) (Ghasemi and Farsi, 2010). It is therefore very glaring that the modification of wood fibre is targeted on establishing chemical groups such as the cellulose and lignin which are competent of improving the interfacial connections among packing particles and plastic matrix (LaMantia and Morreale, 2011). The common surface modification treatments include the following:

Benzylation: This involves immersing the wood fibre into 10% NaOH and then stirred with benzoyl chloride for 1 hour, filtered, washed and dried, then placed in ethanol for another 1 hour before rinsing with distilled water and finally dried. This technique decreases the hydrophilicity of the wood fibre hence, resulting in better affinity with polymer matrix.

Acetylation: This is a process that stabilizes the cell walls, most importantly in conditions of dampness absorption and measurement deviation. It involves immersing the wood fibres in glacial acetic acid for an hour, then also, immersed in a mix of acetic anhydride and little drops of concentrated sulphuric acid for few minutes after which it is filtered, washed, rinsed and dried.

Silane treatment: The wood fibre is immersed into a mixture of ethanol and water in a ratio of 60/40 for two hours. The pH value of the process is kept within the range of 3.5 to 4 via the addition of a silane based adhesion promoter (pure acetic acid) and then rinsed in distilled water and dried. This technique improves the surface quality of the wood fibre.

Peroxide treatment: This technique imparts significant improvement on the mechanical properties of the WPC products. It involves the immersion of wood fibres into dicumyl peroxide solution in acetone for part of an hour, rinsed and dehydrated in an oven.

Alkalisation: The wood fibres are immersed in known concentrated solution of NaOH for a pre-determined duration and then washed in distilled water. It is further rinsed with a few proportion of acetic acid to eliminate the alkali residue (Ghasemi *et al.,* 2010). The washed fibres are then dried in an oven at 80^0C for 24 hours. This technique allows disrupting fibre and obtains less important and improved quality fibres. It in addition improves fibre dampness with the polymer matrix (LaMantia and Morreale, 2011).

2.5.3 Effects of Duration/Concentration of Alkaline Surface Modification Treatments on the Properties of Wood Polymer Composites

The alkalisation treatment process removes lignin, hemicelluloses and other alkali soluble materials from the surface of the wood fibre thus, increasing the softness of the fibre in order to allow better adhesion with the polymer matrix. Jayabal *et al.* (2012) reported that due to the alkaline treatment, hemicelluloses and lignin are removed thereby, allowing the inter fibril region to be less dense and less rigid and causing them to re-arrange themselves in the direction of loading. Ghasemi and Farsi (2010) concluded that alkali modification improves the fibre-plastic bond due to the elimination of ordinary and non-natural debris and deviations in chemical composition of the fibre by taking away the cementing material like lignin and hemicelluloses.

The mechanisms of functioning in the modification treatment depend on the chemical composition and arrangement of the element. For example, the peroxide induced grafting of polyethylene onto cellulose surfaces via the reaction of peroxy radicals (Ghasemi and Farsi, 2010). In NaOH treatment, the major reaction is between the hydroxyl (OH) groups of the cellulose and the hydrogen of the NaOH at the surface of the wood dust, which increases the numbers of reactive hydroxyl groups on the surface available for bonding. Also, the NaOH treatment makes the wood surface clean by removing waxes, pectin, hemicellulose and part of lignin (Mwaikambo and Ansell, 2002). High solution of concentration and longer duration of NaOH treatment removes components that hinder proper adhesion from the wood fibres thus, providing for improved properties of the WPC.

However, allowing the wood dust to stay too long in the alkali solution will cause the wood dust to produce very large pore spaces; this will reduce the strength of the wood dust in the composite hence, a negative effect on the mechanical properties of the WPC. There are more specifics, according to Jayabal *et al.* (2012), high concentration of alkali solution and shorter soaking time provides better impact strength while low concentration of alkali solution for longer soaking time provides improved flexural and tensile properties as shown in Figure 2.13 to Figure 2.15.The outcome in this research work will indicate optimum concentration and duration of treatment for general applications using *Daniella oliveri* as natural organic filler in WPC.

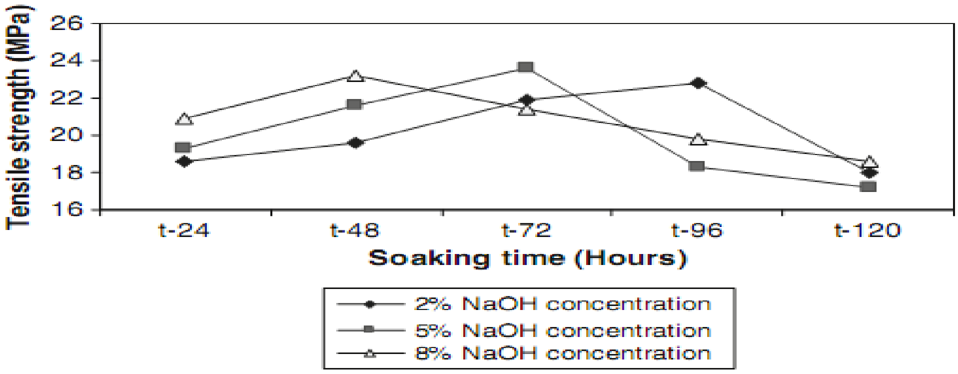


Figure 2.13: Tensile strength value for different level of soaking (Jayabal *et al.*, 2012

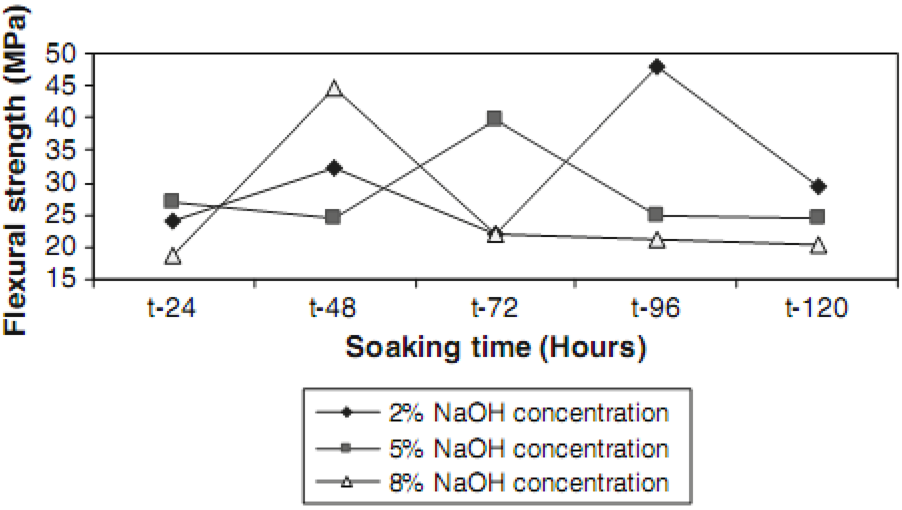


Figure 2.14: Flexural strength value for different level of soaking (Jayabal *et al.*, 2012)

Another very important property to consider in WPC is the moisture absorption capacity; this is because it affects its utilisation. Brugnago et al. (2010) highlights that WPC over time shows an increase in weight with increasing time of immersion. High water absorption is understandable due to the hydrophilic nature of wood substance used as filler material. Also, bigger particle size of organic filler (wood dust) tends to increase the water absorption tendency which correspondingly increases the moisture absorption capacity of the WPC (Leu *et al.*, 2012). In addition to the above, increased water absorption level could be as a result of processing problems such as incomplete curing of thermoset matrix.Mylsamy and Rajendran (2011) also discovered that, when the heat of soaking is improved, the wetness saturation time is considerably reduced. Figure 2.16 shows the graphical representation of treated (CTCEC) and untreated (CUTCEC) continuous epoxy composites at room warmth and at boiling hotness. When immersed in distilled water for 2hours, the moisture absorption capacity of the untreated sample is highest at boiling temperature; this is because at high temperature, the wood particles becomes more soften hence, the tendency to absorb more water content. This confirms that moisture absorption is slower at room temperature due to the fact that the wood dust has high hydroxyl group which has low resistance to moisture.

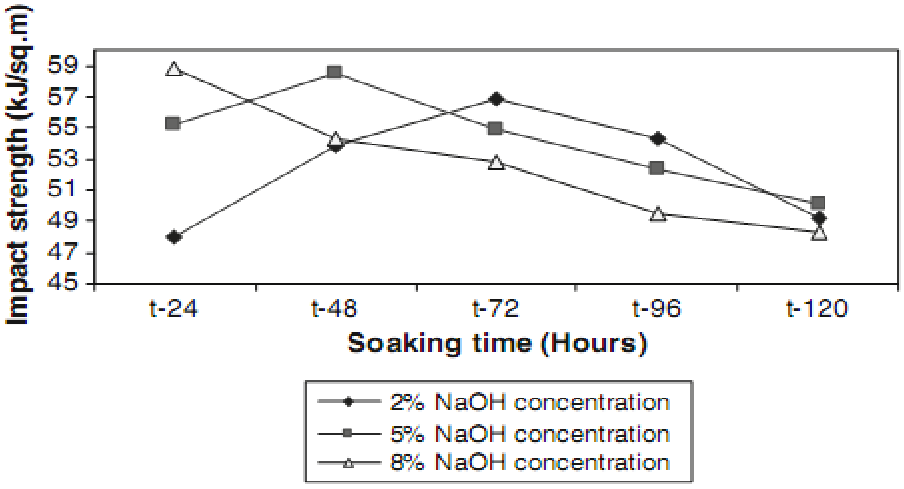


Figure 2.15: Impact Strength Value for different level of soaking (Jayabal *et al.*, 2012)

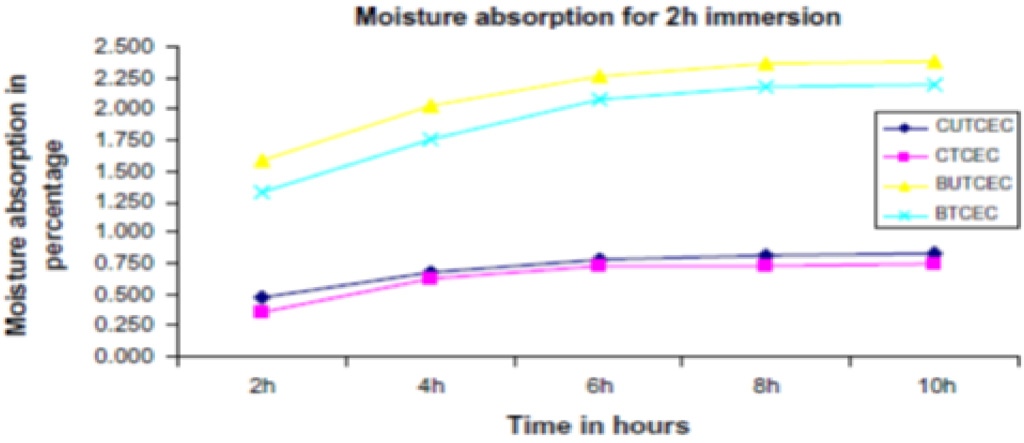


Figure 2.16: Moisture absorption of treated and untreated (CTCEC & CUTCEC) samples in cold water as well as absorption capacity of treated and untreated (BTCEC & BUTCEC) samples in boiled water (Myslamy and Rajendran, 2011)

2.6 Wood Polymer Composite Processing

Wood polymer composite processing technologies are separated into two major steps, compounding and forming. This can be carried out on a nonstop or different flow. WPCs are generally produced in a melt process such that heat has to be adequately high all through to have the thermoplastic in a melt condition. This is to ensure adequate mixing in the compounding process. However, usual challenges connected to the production of WPCs are owing to the hydrophilic and hydroscopic nature of wood filler and their poor-thermal resistance (LaMantia and Morreale, 2011). Therefore, manufacturing temperature has to be maintained as low as feasible and the option of thermoplastic is reduced to the ones with low melting point, commonly below 180°C (Caulfield *et al.*, 2005). Processing temperature generally never reaches more than 220°C (Sylvain, 2009). Compounding consists of blending the wood filler with the thermoplastic and it is generally carried out in a continuous screw extruder. The main goal is to obtain a good dispersion of the wood component in the matrix and this is made difficult by the tendency of fibres to agglomerate due to their polarity (Bledzki *et al.*, 2008). The affinity for entanglement, together with the low bulk density of wood constitutes a problem for the feeding of the fibres in the extruder

(Caulfield *et al.*, 2005). High viscosity of wood filled plastic is another issue that makes melt processing more difficult, thus, compositions generally ranges between 30% and 50% by weight of wood. Moisture presence has been reported to reduce physical properties; the wood material is thus, oven dried at 28°C for three days prior to compounding. Also, different additives can be used to improve the dispersibility (lubricant), the interfacial bonding (compatibilizers) or reduce the viscosity (plasticizers) (Bledzki *et al.* 2008). Apart from thermal degradation, the wood components are affected by compounding due to high shear rates developed, which induce a breakdown of the particles and a reduction in length of the fibres (Rowell, 2007; Segerholm, 2007). This is mainly influenced by the configuration of the screw.

Figure 2.17 shows a typical schematic diagram of an extrusion process. The type of extruder can be different for the two processing steps or depending on the product wanted. Single crew and twin counter-rotative extruders are preferably used for extrusion of profiles because of the high pressure that can be reached which enhances the flexural strength of the wood polymer composites, but, compounding is done mostly on rotating twin screw extruders for their high mixing and degassing abilities (Scwendemann, 2008). Japanese scholars have just planned an electromagnetic initiation process for jute material toughened thermoplastic moulding (Tinaka *et al.* 2008). According to the Tinaka and co-authors, the scheme allows an immediate hotness of the mould surface, thus limiting the manufacturing cycle period leading to a decrease of the fabrication costs connected to composite panels moulding. Also, previous authors have established important influence of fibre area portion on the mechanical and thermo-mechanical properties of green composites (Dauda *et al.*, 2007).

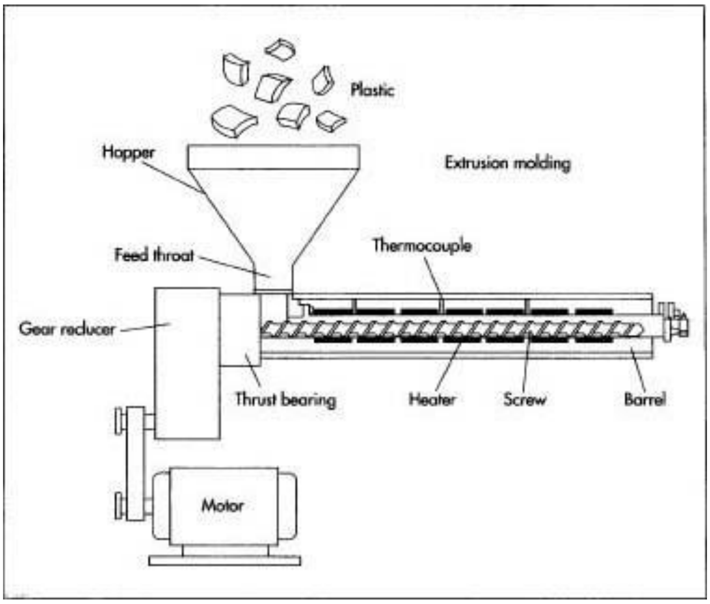


Figure 2.17: schematic diagram of a typical extrusion process (www.google.com)

A comprehensive research by Scaffaro *et al.* (2009), established by reason of numerical examination that the most influencing process changeable on the substance stiffness is the filler substance, even though initial treatments and combination speediness exert some influence as well; the impact strength was mostly subjective by mixing speed and filler aspect relation, while hotness had only a negligible consequence. In regards to the manufacturing techniques, they establish the injection moulding caused a partial deprivation of the macromolecules foremost to a decrease of the stickiness and the rigidity of the materials. Although the double-screw and single screw extrusion proceed by calendaring approved obtaining ultimate resources by advance moduli (Morreale *et al.* 2008; Scaffero *et al.* 2009). However, the particular dense morphology was established in the injection moulded samples. The relationship between extrusion and combination is not effortless, since similar results were found for the elastic modules, while the tensile force deeply relied on the filler substance (Table 2.5).

Table 2.5: Tensile characteristics of wood dust packed green composites as a function of manufacturing technique (La Mantia and Morreale, 2011)Mechanical property	Processing	Neat polymer	Composite, 15wt% WF
Elastic modulus (MPa)	Mixing	410	537
	Single – screw extrusion	378	535
	Twin – screw extrusion	373	563
	Injection molding	200	295
Tensile strength (MPa)	Mixing	17	11
	Single – screw extrusion	27	17
	Twin – screw extrusion	28	12
	Injection molding	15	18

In the work of Ferreira *et al.* (2006) partly comparable trends on PVC fibre composite were discovered. Nevertheless, from the above stated investigation, it was observed to facilitate the roles of space filler kind, filer substance, filler aspect relation and manufacturing temperature are of great significance, hence, it may be hard to search along with recommended common rules which might be relevant to the mechanical behaviour (LaMantia and Morreale, 2011). Furthermore, Takagi and Asano (2008) ready green composite based on spreading type eco-friendly resin and cellulose nano-fibres by means of stirring method at comparatively less speed and at longer times. Upon increase of the mould pressure, their observation revealed important increase in flexural modulus and force (up to 100%).

The waste wood dust of African Copaiba Balsam tree (*Daniella oliveri)* does not blend with polymer matrix because of the hydrophilic nature of wood and hydrophobic properties of polymer. In order to fix this issue and produce a better quality WPC, there is a need to carryout surface treatment of the wood dust hence, the need for this work.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials used for the research work were sourced locally. They consisted of waste product of the wood (wood dust) and virgin high density polyethylene. Saw dust is a waste-product of the saw mills and while virgin high density polyethylene is obtained from the packaging industries.

3.1.1 Wood dust

The reinforcement materials used for this study were waste wood dust made of soft wood of African Copaiba Balsam tree (*Daniella-oliveri*) commonly called Kadaura tree in Hausa language. The choice of this wood dust was as a result of the abundant availability of these particular species of wood dust at most Nigerian saw mills. It was collected from sawmill in Minna, Niger state, Nigeria.

3.1.2 virgin High Density Polyethylene (vHDPE)

The matrix material chosen for this study is virgin High Density Polyethylene (vHDPE) because of its usability in a variety of applications such as packaging and in the automobile industries. Besides, HDPE has no branching in its nomenclature thus, giving it stronger intermolecular forces and improved tensile strength over LDPE. HDPE is harder, more opaque and can withstand higher temperature (ranging between 125°C to 130°C). Its density also ranges from 0.95g/cm^3 to 0.97g/cm^3 while the melt flow index is from (0.15 to 0.4)g/10min and the molecular weight is 28g.

3.1.3 Reagents used for Treatment

Sodium hydroxide (NaOH) used as the alkali reagent for the treatment of the wood dust was obtained from Echas Scientific Company, Kaduna, Nigeria. The dilute hydrochloric (HCl) acid was obtained from the Kaduna Polytechnic Soil Science Laboratory and was used to rinse the treated wood dust in order to remove the excess alkali from the treated wood dust. Litmus paper was used to confirm the removal of excess HCl.

3.2 Methods

3.2.1 Preparation of Wood Dust

Wood dust of *Daniella-oliveri* tree (commonly called Kadaura in the Northern and Western Nigeria) was collected from a local saw mill in Minna, Niger state and dried in the sun for 48hours in order to remove the excess moisture content. The wood dust was then soaked in hot water at 100°C for 1 hour in order to open up the pore spaces of the hydrophilic wood dust. At the expiration of 1 hour, the wood dust was thoroughly washed and then air dried for 5 days at room temperature of 21°C.

With the use of hand oscillating multi-deck screen classifier, the wood dust was separated into various particle sizes and distribution +75-150µm, +150-210 µm, +210 – 300 µm, +300 - 425µm, and +425 - 600µm. Apart from the fact that majority of previous researches in the field of WPC made use of wood dust in the range of 425µm to 600 µm (Wolcott and Englund, 2000); wood dust having particle size and distribution +425 - 600µm was selected based on the largest quantity of this portion obtained after sieving. They were then treated with varying concentration of NaOH (2%, 4%, 6%, 8% & 10%).

3.2.2 Preparation of NaOH Concentrations

With the use of Digital Weighing Balance, LP202A model, product of B Bran Scientific & Instrument Company, England 20g of crystal NaOH was introduced into 1000 cm^3 distilled water to give the required 2wt% of NaOH solution while 40g, 60g, 80g and 100g were introduced into $1000cm^3$ of distilled water to produce 4wt %, 6wt %, 8wt % and 10wt % respectively. These varying concentrations were kept in air tight bottles and stored in the laboratory.

3.2.3 Preparation of Dilute HCl

A volume of $85cm^3$ of concentrated HCl was measured with a laboratory measuring cylinder and poured into $1000cm^3$ of distilled water to produce the diluted HCl. The dilute HCl was also poured in glass bottles and stored in the laboratory.

3.2.4 Alkalisation of Wood Dust

A mass of 6.72g of the selected wood dust was measured into each of five different beakers designated A, B, C, D and E containing 2%, 4%, 6%, 8% and 10% concentration of NaOH respectively. The wood dust was left immersed in the alkali solution for 30mins after which it was cleansed with adulterate hydrochloric acid (HCl) in order to take away surplus alkali. However, it was ensured that the dilute HCl acid did not neutralize the effect of the alkaline treatment. This was achieved via a quick wash in aqueous HCl. Then, the wood dust was thoroughly rinsed with distilled water. The rinsed wood dusts were dried at room temperature of 21°C for 5 days. Upon drying, the wood dust was mixed with the vHDPE and the specimen samples were produced. The properties of samples were investigated via mechanical, physical and microstructural test and the effect of alkali concentration on the properties of the resulting WPCs was used to conclude on the optimum concentration for the WPC treatment. This procedure was then repeated for wood dust soaked in the optimum concentration of NaOH obtained, for 30, 60, 90, 120 and 150 minutes. All the treated wood dusts were then taken for compression moulding and the properties were compared to that of the sample produced from the untreated wood dust.

3.3 Compounding and Preparation of Wood Polymer Composite Samples for Testing

The mould used for preparing the WPC samples was made from a gentle steel plate with a measurement of (240x90x15) mm arranged with a crest plate and a bottom plate. In order to enhance total and easy elimination of composite from the mould, a nylon sheet was laid on the spotless and dry mould prior to the injection of the blended wood dust and polyethylene into the mould at the ratio of 50:50 for full utilisation of the wood dust and for best mechanical properties (Leu *et al.*, 2012). The compression moulding process used to produce the specimen involved, pouring the solid polyethylene crystals into a hopper of the compression moulding machine. From the hopper, it is channelled to the pre-heated chamber in order to pre-heat (soften) the polyethylene. The wood dust was introduced into the molten polymer inside the mold at the melting temperature of 120°C. The mould was then closed with a top plug and pressure was applied to force the material to the mould so as to produce the specimen samples required. The process parameters were maintained at a uniform pressure of 2.94MPa, temperature (140 ^{o}C) and time (7minutes). The samples produced as shown in PlateII were machined to various geometries in accordance with the requirements of the various tests.

Plate II: Samples produced

3.4 Testing and Characterisation of Wood Polymer Samples

Six different tests were run on the specimens produced. These tests are mechanical (tensile and bending) test, physical (moisture adsorption and density) test, chemical composition and bonding (FTIR) test and microstructural observation (SEM).

3.4.1 Determination of Tensile Strength

The tensile strength test was carried out in National Research Institute for Chemical Technology (NARICT) Zaria according to the ASTM D638-03 measure. The tensile investigation was accepted out with the 5980 floor model Instron universal testing machine produced in the United Kingdom at a cross head speed of 5mm/min. The original length, width and thickness of the sample were 90mm, 20mm and 7mm respectively. The test was carried out under room temperature 21°C and relative humidity condition at 40 ± 2%. The sample was placed at the grasp of the testing mechanism to arrange in a line the long axis and the grips with a make-believe line combining the points of attachment of the grips to the appliance. The dimension of load, elongation and tensile strength were taken on a computer screen and value recorded accordingly and the tensile modulus of elasticity (MOE) was calculated using equations 3.1 – 3.3.

$\sigma = \mathrm{F/A}\ \left(\frac{N}{mm^2}\right)$ 3.1

$\varepsilon = \frac{\Delta L}{L}\ (unitless)$ 3.2

$MOE = \frac{Stress}{Strain} = \frac{\sigma}{\varepsilon}\ (N/mm^2)$ 3.3

σ=tensile stress

ε = strain

F = force applied

A = cross sectional area

$\Delta L = change\ in\ length$

L = original length

3.4.2 Determination of Bending Strength

A rectangular sample with length, width and thickness of 105mm, 20mm and 7mm respectively was prepared from produced samples of the concentration and time of treatment and used to carry out the bending test according to the ASTM D790-07 standard. It was carried out at 50 rev/min with the 5980 floor model Instron universal testing machine produced in the United Kingdom. The values for the bending tests were obtained from the digital screen of the Instron machine and recorded. The formulae involved in the calculation of bending stress and the flexural modulus are given by equations 3.4 and 3.5.

$\sigma = \mathrm{3FL/2bd^2}$ 3.4

$FM = \frac{L^3 m}{4bd^3}$ 3.5

where;

F = load at fracture point (N)

L=length of support span (mm)

b=width of material (mm)

d=thickness of material (mm)

m = gradient of slope

3.4.3 Determination of Moisture Absorption Capacity of Wood Polymer Composite

The samples were prepared according to the ASTM D570 – 98 standards for dampness inclusion test. Before immersion, the sample were made dirt free and weighed on a digital weighing balance, model LP202Awith 0.01g accuracy. The initial weight of the sample w_o was then recorded in grams. Moisture inclusion tests were carried out by immersing sample in a distilled water tub at room temperature of 21°C and hot water at 60°C. At each of these temperatures, the absorption of the samples was observed for 24 hours. During the soaking process, the samples were made to rest on at the base and completely soaked in the water bath. For each observation at the end of the 24 hours, each specimen was wiped clean with dry fabric to remove surplus moisture on it without delay and then weighed (w_t). In the hot water immersion procedure, after each immersion, the sample were brought to ambient temperature by dropping in cold water for 10minutes and then weighed and average value of three samples were recorded. The moisture content $M_{(t)}$, absorbed by each sample was obtained using equation 3.6.

$$Mt = [(wt - wo) \div wo] \times 100\% \qquad 3.6$$

$where;$

$wo = initial\ weight\ of\ sample\ in\ grams$

$wt = wet\ weight\ of\ immersed\ samples\ in\ grams$

$Mt = moisture\ content$

3.4.4 Determination of Density of Wood Polymer Composite

The density of the wood polymer composite sample was determined by the water displacement method. The mass of each specimen sample was measured using the digital weighing balance, model LP202A, with 0.01g accuracy and values were recorded. Similarly, the volume of water displaced while the weighed specimen was completely immersed in a beaker of distilled water was also recorded. The density of the specimen was calculated using equation 3.7.

$$\rho = m \div v \quad \left(\frac{g}{cm^3}\right) \tag{3.7}$$

$where;$

$\rho = density;$

$m = mass; v = volume$

3.4.5 Determination of Chemical Composition by Fourier Transform Infrared Spectroscopy

The Fourier Transform infrared (FTIR) Spectroscopy with a light microscope was used to investigate the pressure transmission at the molecular level. The FTIR equipment; SHIMADZU FTIR – 8400_s model at the National Research and Chemical Technology (NARICT) Zaria was used to get the structural investigation of the specimens. The FTIR was used in the transfer mode with a dimension of 4 cm^{-1} in the array of 4000 – 400 cm^{-1}. FTIR was used to detect the molecular shaking in order to examine the spreading of efficient groups of the specimen.

3.4.6 Observation of the Wood Polymer Composite by Scanning Electron Microscopy (SEM)

Scanning Electron Microscopic (SEM) observation of the samples was done out in order to study the consequence of alkali treatment on the wood dust - vHDPE interfacial chemistry. The fractured surfaces of the specimen obtained from tensile test were analysed using SEM equipment model JEOL 7500 SEM from Japan. In SEM, a fine query of electron scans the exterior of the specimen and the signals coming from the incident location are examined and quantified. All samples were sputtered with 10nm layer of gold before SEM investigations. Each sample was installed on the aluminium handle of the microscope by means of double sided electrical conduction carbon bonding agent tabs. The accelerating voltage of 5 – 15kV was engaged. The SEM examinations of the different specimen were observed at 100 and 500 magnification and conclusion drawn from them. The SEM fractographs of both treated (at various concentration and time of treatment of NaOH solution) and untreated wood polymer composite samples were compared.

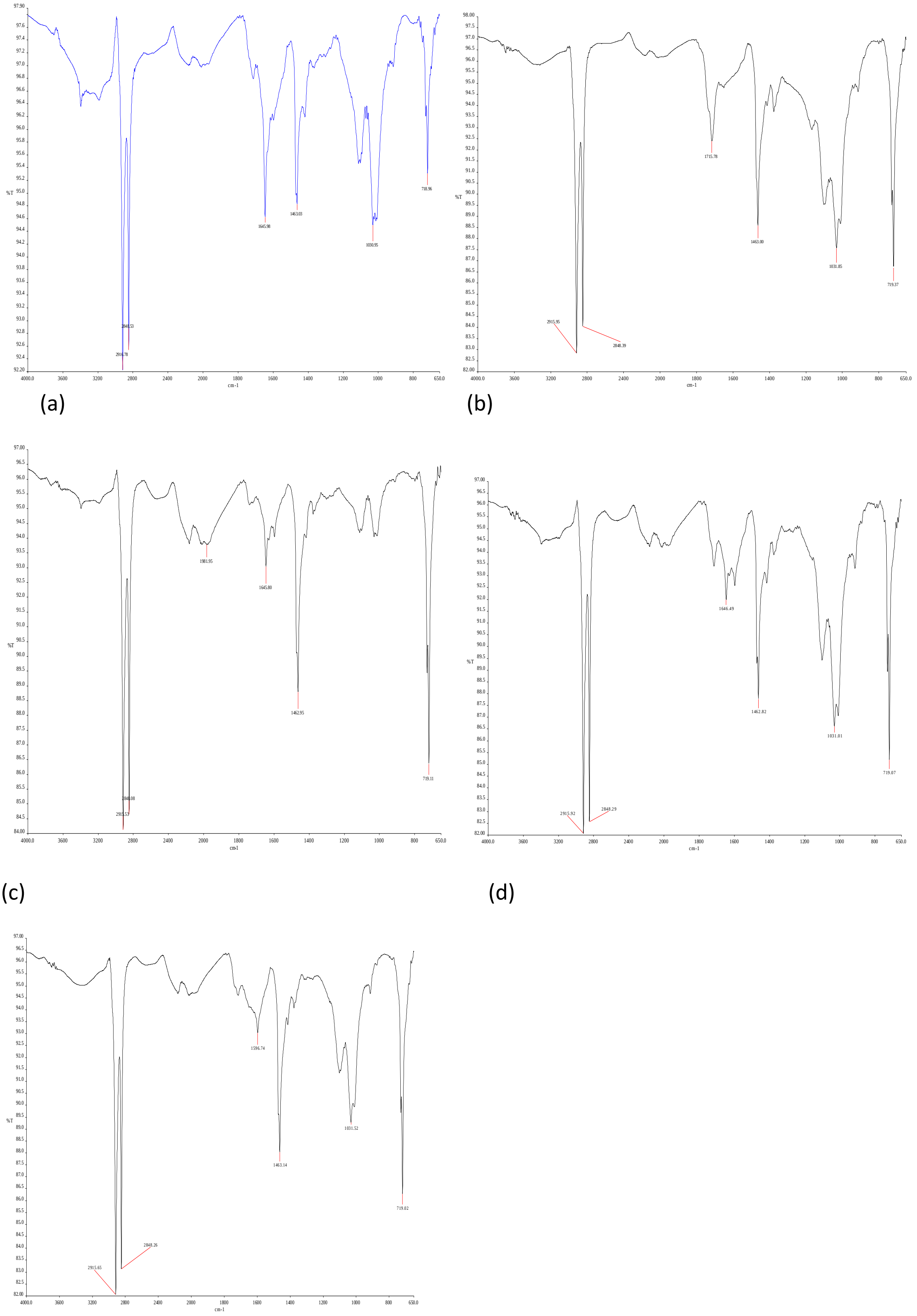
CHAPTER FOUR

4.0 RESULTS AND DISCUSSION

4.1 Fourier Transformation Infrared Analysis of Wood Polymer Composite Samples

Figures 4.1 and 4.2 shows the FTIR spectra of untreated and alkaline treated *Daniella oliveri* wood dust at different concentrations of NaOH solutions and different periods of treatment respectively. The absorbance intensity of 3400 and 1030 cm^{-1} peaks assigned to the stretching vibrations which are attributable to the hydrogen bonded hydroxyl (-OH) groups of cellulose and C–O groups of hemicelluloses (Barreto *et al.*, 2011) respectively, decrease with the alkalisation treatment of the wood dust at various concentration of NaOH and time of treatment of the wood dust as shown in Figures 4.1–a-e and 4.2–a-d. The reduction of the absorbance concentration upon alkali modification suggests a decrease in intermolecular/intramolecular hydrogen pairing flanked by the hydroxyl cluster of cellulose and hemicellulose of *Daniella Oliveri* wood dust (Saha *et al.,* 2010; Farsi, 2012). The permeability 1463 cm^{-1}, and 1031 cm^{-1} are attributed to –CH symmetric stretch and aromatic –CH in plane distortion in lignin, correspondingly (Ray *et al.*, 2002) (Figures 4.1a-e and 4.2a-d). The band at 1715 cm^{-1} is for C–O stretch of the carboxyl and ester units in hemicelluloses of the wood dust, which is well-known in untreated wood fibre (Haque *et al.,* 2009). This peak steadily decline as the concentration of NaOH and modification time improved and it disappeared beyond 150 minutes of treatment.

The peak at 1645 cm^{-1} in the untreated wood dust is associated with the adsorbed water. The decrease in this peak intensity in the alkaline treated wood dust and its disappearance after 150 minutes is due to the partial removal of hemicelluloses. The weak peak noticed at 1463 cm^{-1} is assigned to the in plane bending deformation of $–CH_2$ of lignin. The $–CH_2$ bonding of cellulose component of the wood dust which remained unchanged after alkali modification (Sui *et al.,* 2009) may increase the wood dust tensile strength and consequently the mechanical properties of WPCs (Alix *et al.,* 2009). The sharp peaks observed between 2848-2915 cm^{-1} indicate C–H asymmetric deformation of lignin. In summary, the alkaline treatment of wood dust at varying concentration and time of treatment changes the super molecular structure of wood dust while the chemical structure is not significantly affected. Due to the intra- and inter-fibrillar puffiness, the convenience of wood dust changes with the removal of hemicelluloses and more alkali dissolving materials from the exterior of the wood dust thus, increasing the softness of the fibre in order to allow better adhesion with the polymer matrix.

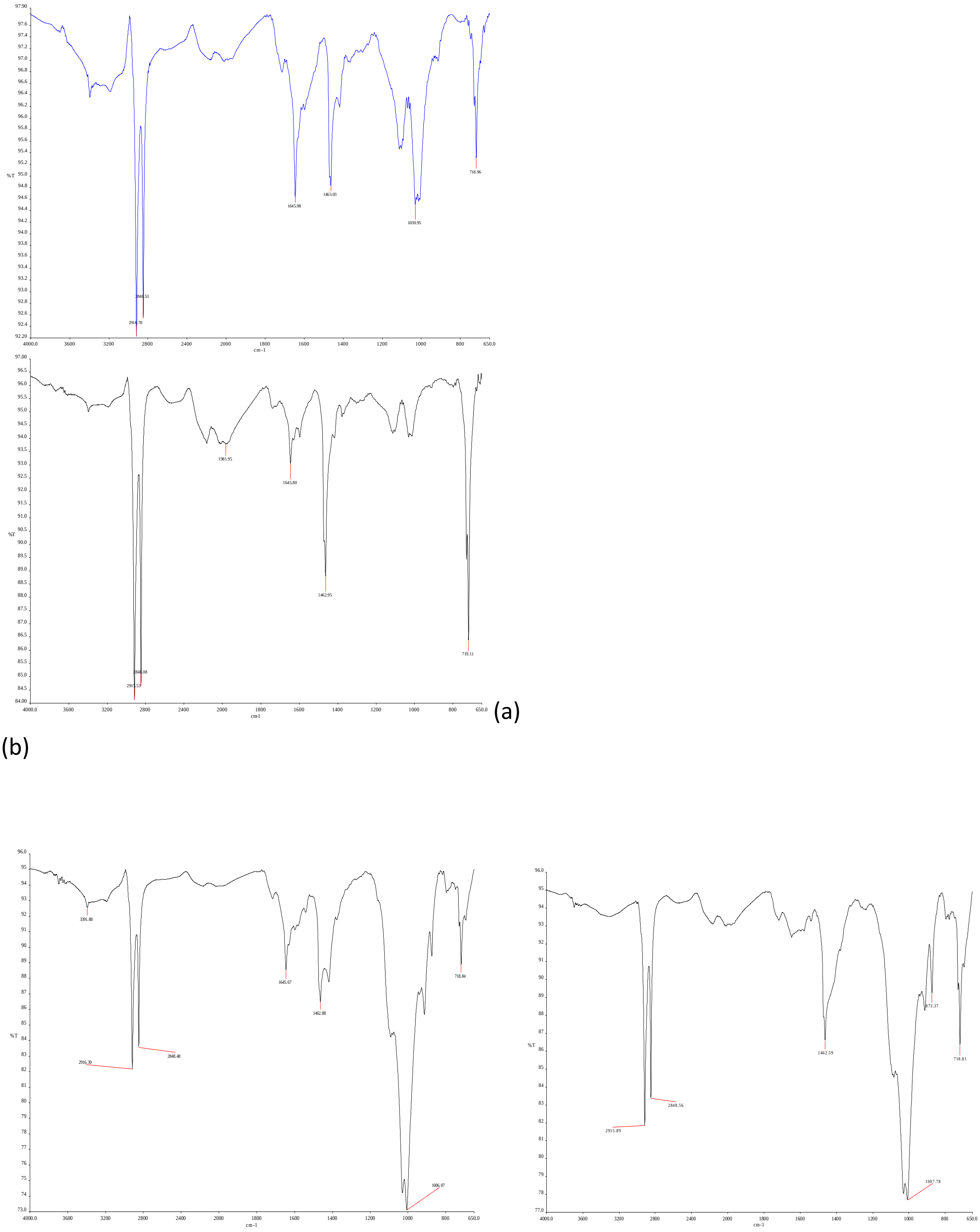


(a) (b)

(c) (d)

(e)

Figure 4.1: FTIR spectra of *Daniella oliveri wood dust* (a) untreated and treated with (b) 2wt%, (c) 4wt%, (d) 6wt%, (e) 10wt% NaOH concentration at 30minutes



(a)

(b)

(c)

(d)

Figure 4.2: FTIR spectra of *Daniella Oliveri wood dust*(a) untreated and treated for(b) 30, (c) 90, (d) 150 minutes in 4wt%concentration of NaOH.

4.2 Effect of Concentration and Time of Treatment of NaOH Solution on the Density of Wood Polymer Composite

Figures 4.3 shows that the density of the wood polymer composite samples increased to the optimum (1.095 g/cm^3) as the concentration of NaOH solution increased to 4wt% while subsequent increment in the concentration of NaOH resulted in reduction in the density of the samples for the same time of treatment (30 minutes). Figure 4.4 also depicts that the density of the wood polymer composite samples increased to 1.4 g/cm^3 as the time of treatment of the wood dust in the 4wt% concentrated NaOH solution increases to 150 minutes. Meanwhile, it is evident from both Figures 4.3 and 4.4 that density values obtained as a result of the alkalisation treatment of wood dust incorporated into the HDPE matrix are higher than that obtained when a wood polymer composite is produced with untreated wood dust that yields the density of 0.401g/cm^3.

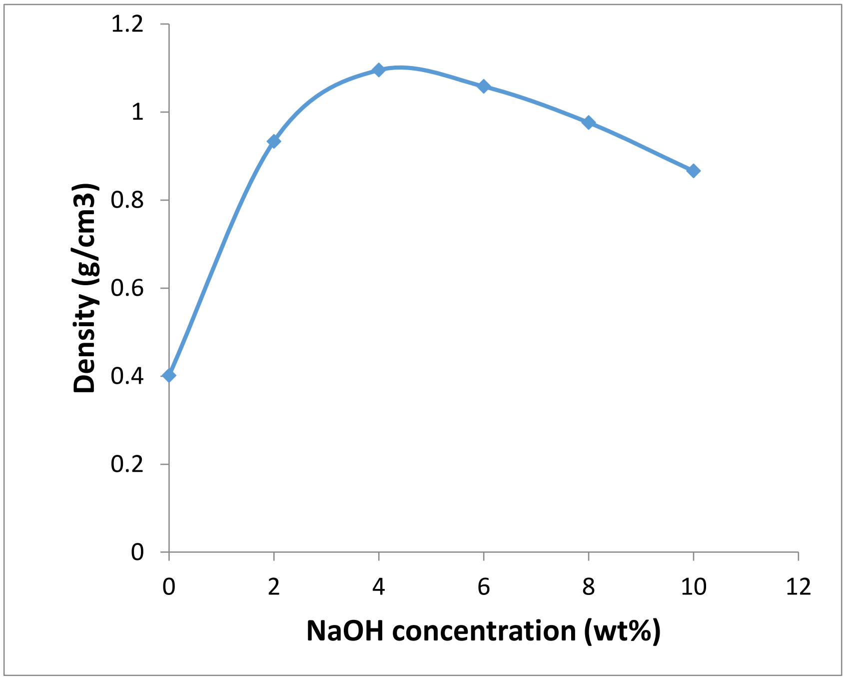


Figure 4.3: Relationship between the density of the wood polymer composites and varying concentration of NaOH solution when the wood dust was soaked for 30 minutes.

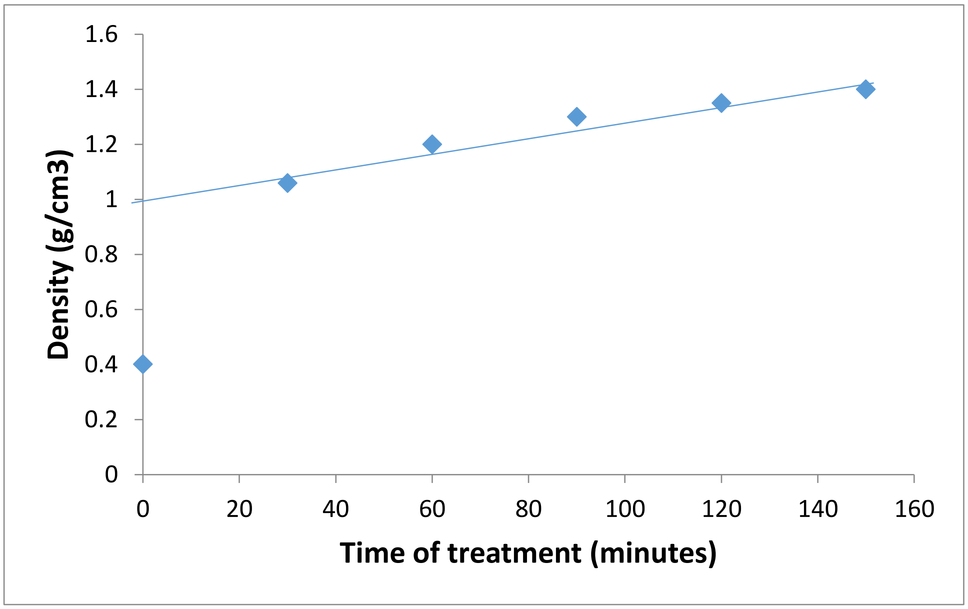


Figure 4.4: Relationship between the density of the wood polymer composites and varying time of treatment when the wood dust was treated with 4wt% concentrated NaOH solution.

The alkalisation treatment process removes hemicelluloses and additional alkali dissolvable materials from the exterior of the wood dust thus, increasing the softness of the fibre in order to allow better adhesion with the polymer matrix (Jayabal *et al.*, 2012). This eventually makes the inter fibril portion to be lower in density and less stiffened. Consequently, soaking natural fibres in a NaOH solution of appropriate concentration for an appropriate time of treatment removes components that hinder adhesion of wood dust with the polymer matrix thereby improving the densification of WPC. However, treating the wood dust with a NaOH solution of too low concentration or for insufficient time will cause the wood dust to produce very large pore spaces, hence, reducing the density of WPCs. Reduced density obtained at NaOH concentration higher than 4wt% could be attributed to the damage and peeling of the fiber surface which induces poor interface adhesion and wettability. This will eventually reduce the strength of the wood dust in the composite hence, a negative effect on the mechanical properties of the WPC. Finally, treating *Daniella oliveri* with 4wt% concentration of alkali solution for longer time (150 minutes) optimised the density of WPCs. This is expected to result in optimisation of other physical and mechanical characteristics of WPCs.

4.3 Effect of Concentration and Time of Treatment of NaOH Solution on the Water Absorption Capacity of Wood Polymer Composite

Figures 4.5 and 4.6 reveal the outcomes of water absorption test of the investigated wood polymer composites as the concentration and time of treatment of NaOH solution are varied respectively. The water absorption capacity of the wood dust plastic composites is seen to decrease as the concentration of NaOH solution increased up to 4wt% (0.70% absorption) at a water temperature of 28^{o}C as evident in Figure 4.5. Thereafter, the water absorption capacity of the composites is seen to begin to increase as the concentration of NaOH solution is increased above 4wt% at a fixed time of 30 minutes. Figure 4.6 shows that the water absorption decreased from 0.70% at 30 minutes to 0.35% at 150 minutes which establishes the fact that the moisture content reduces as the time of treatment in 4wt% NaOH concentration increases. The lowest value of 0.35% of water absorption capacity at 28^{o}C obtained in Figure 4.6 when the polymer matrix was reinforced with wood dust treated with 4wt% concentrated NaOH solution for 150 minutes corroborate the fact that the density of the wood polymer composite was optimised at the choice of the same values of parameter as observed in Figure 4.4. It is quite obvious from Figures 4.5 and 4.6 that wood polymer composites possess greater water absorption capacity at higher water temperature (60^{o}C) than at lower temperature (28^{o}C) irrespective of the concentration and time of treatment of NaOH solution employed in treating the wood dust.

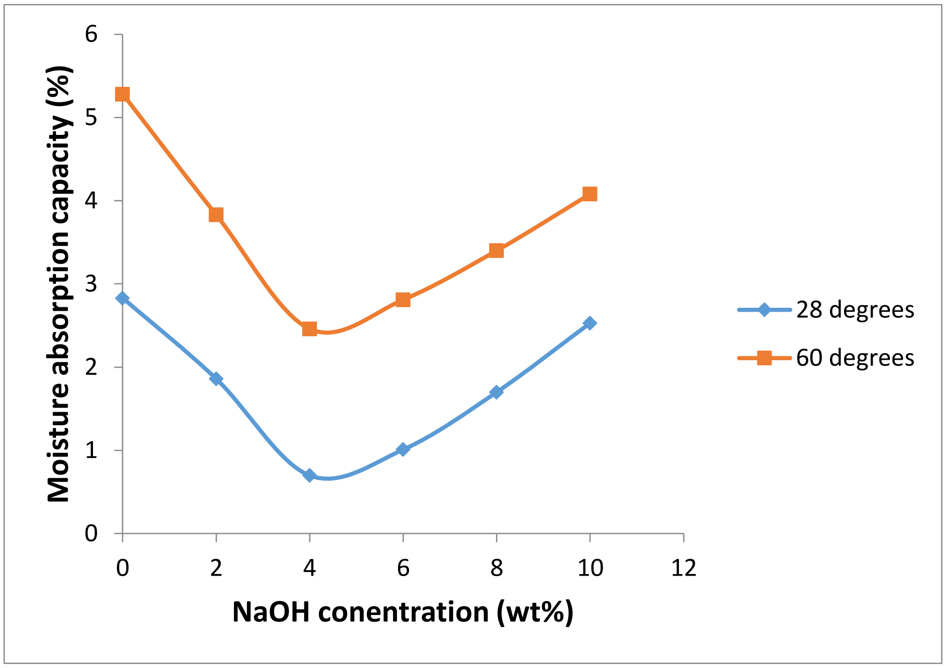


Figure 4.5: Relationship between the moistur absorption capacity of the wood polymer composites and varying concentration of NaOH solution when the wood dust was soaked for 30 minutes.

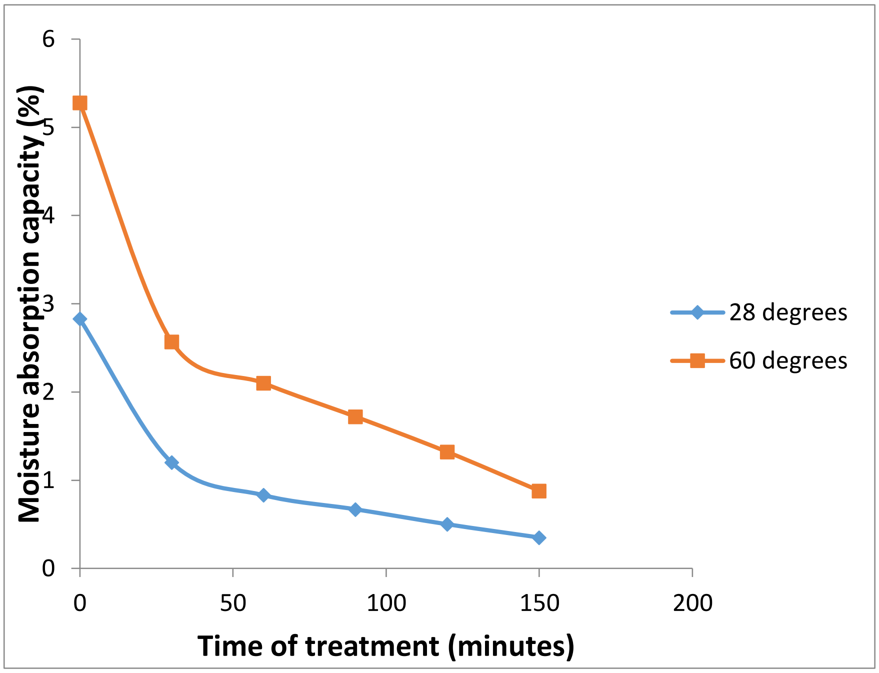


Figure 4.6: Relationship between the moisture absorption capacity of the wood polymer composites and varying time of treatment when the wood dust was treated with 4wt% concentrated NaOH solution.

Findings reported for water absorption capacity of WPCs as both concentration and time of treatment in NaOH are varied are quite in consonance with the outcome of the study carried out by Mylsamy and Rajendran, (2011). They also discovered that at the time the temperature of soaking is increased, the wetness absorption capacity of WPCs is significantly increased as seen in Figures 4.5 and 4.6. Moreso, this process can be described in terms of thermodynamics, with increased temperature favouring moisture infiltration in the WPCs. This could be attributed to the fact that at higher temperature, the stiffness of the fiber configuration is easily damaged by the percolating moisture molecules. Consequently, the absorbed water acts as plasticizer thereby permitting cellulose molecules to move freely. As water absorbing capacity of WPCs increases at higher temperature, the interlocked water molecules attains greater propensity to keenly confront the boundary, therefore, resulting in disjoining of the wood dust and the matrix inside the composite.

4.4 Effect of Concentration and Time of Treatment of NaOH Solution on the Tensile Strength of Wood Polymer Composite

Figure 4.7 confirms that the tensile strength of the wood polymer composites produced via reinforcement with wood dust treated in varying concentration of NaOH solution for 30 minutes is optimised (11 MPa) at 4wt%. Moreover, the tensile strength of wood polymer composite increased to 16 MPa when the reinforcing wood dust was treated with 4wt% concentrated NaOH solution for 150 minutes (Figure 4.8) in comparison to the optimal value obtained in Figure 4.7.

This outcome suggests that increasing the time of treatment of *Daniella Oliveri* wood dust in a constant concentration of NaOH solution increased the tensile strength of the composite samples. Further increment above this optimal time of treatment resulted in increase in tensile strength. Finally, it is clear from Figures 4.7 and 4.8 that the tensile strength of the treated wood polymer composite are higher than that of wood polymer composite reinforced with untreated wood dust.

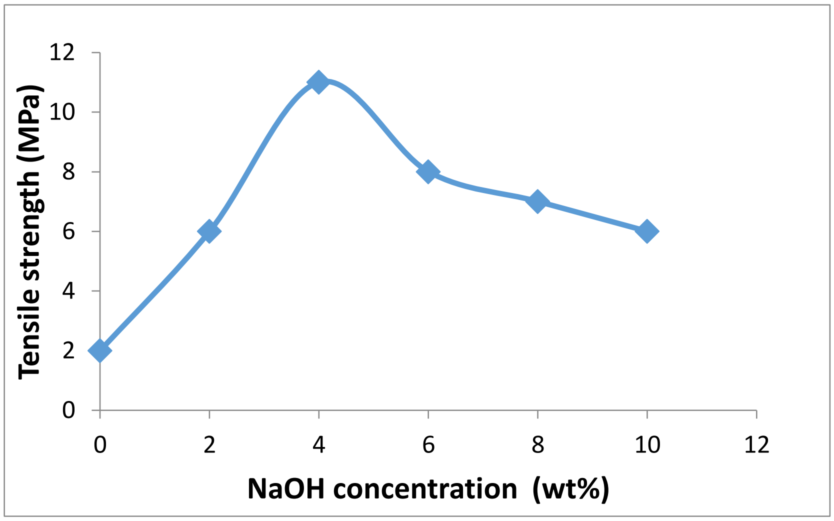


Figure 4.7: Relationship between the tensile strength of the wood polymer composites and varying concentration of NaOH solution when the wood dust was soaked for 30 minutes.

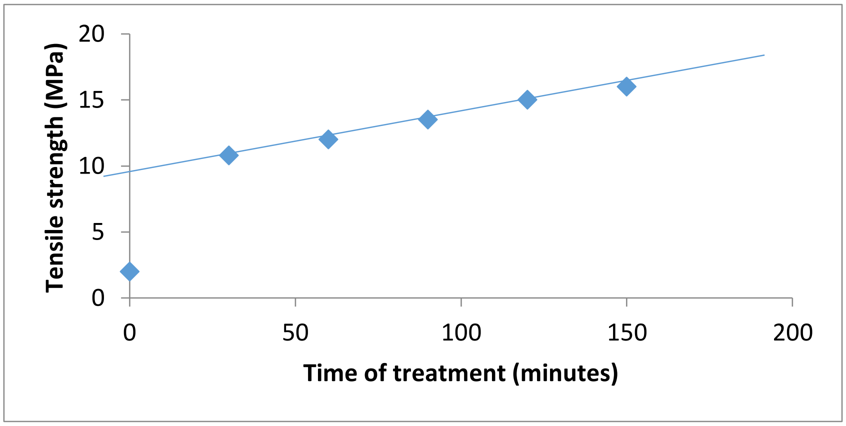


Figure 4.8: Relationship between the tensile strength of the wood polymer composites and varying time of treatment when the wood dust was treated with 4wt% concentrated NaOH solution.

4.5 Effect of Concentration and Time of Treatment of NaOH Solution on the Bending Strength of Wood Polymer Composite.

Figures 4.9 and 4.10 show the bending strength of composites made from wood flour treated at 30 minutes with varying concentration of NaOH solution and 4wt% of NaOH solution at varying time of treatment respectively. Figure 4.9 confirmed that the bending strength is optimised at 32MPa at 4wt% when treated for 30 minutes and it increased to 103MPa when the time of treatment in 4wt% NaOH increased to 150 minutes (Figure 4.10). The bending strength of the composite increased by 223% when the reinforcing wood dust was treated with 4wt% concentration of NaOH solution for 150 minutes, in comparison to the optimal value obtained in Figure 4.9. Furthermore, Figure 4.10 reveals that the longer the time of treatment with NaOH solution, the better the bending resistance of the wood polymer composite. This result agrees with that reported by Jayabal *et.al.* (2011) when considering the soaking time and flexural strength of coir polyester composites.

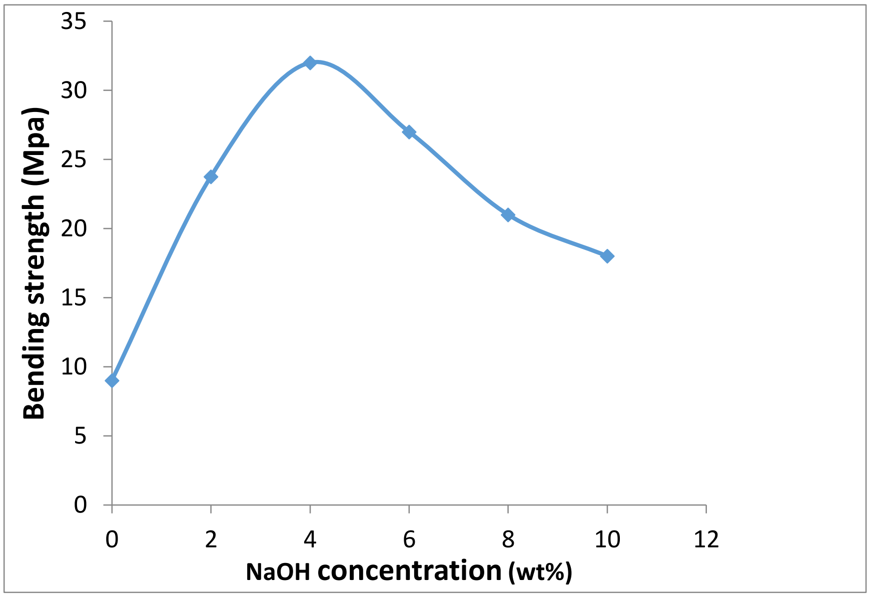


Figure 4.9: Relationship between the bending strength of the wood polymer composites and varying concentration of NaOH solution when the wood dust was soaked for 30 minutes.

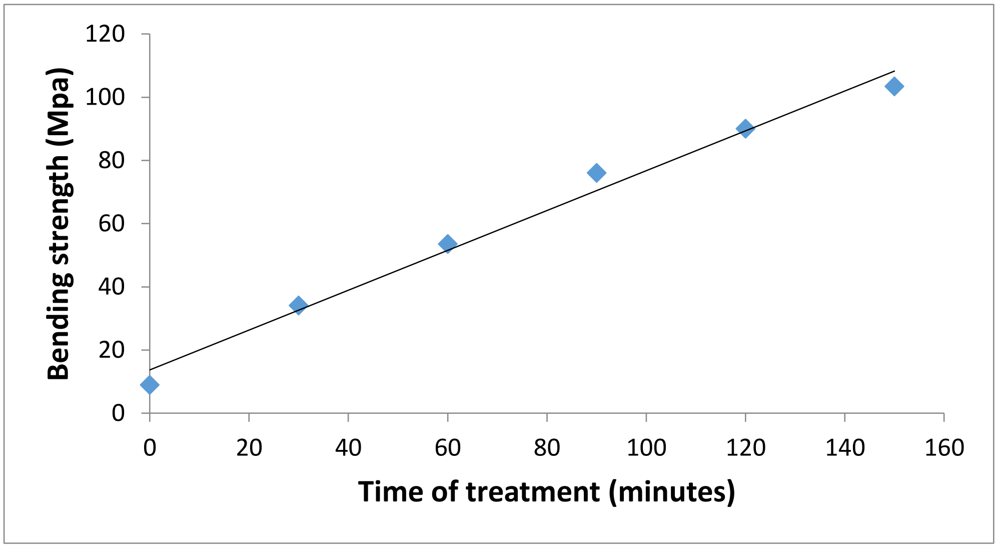
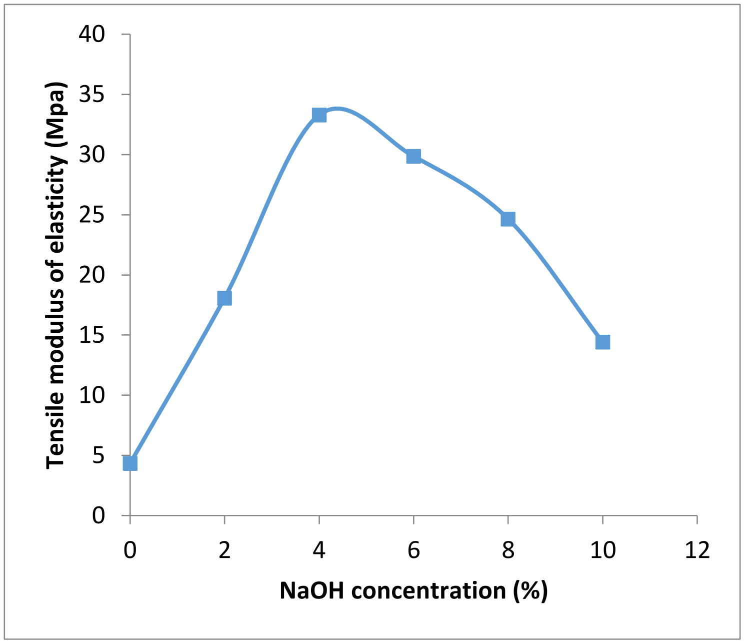


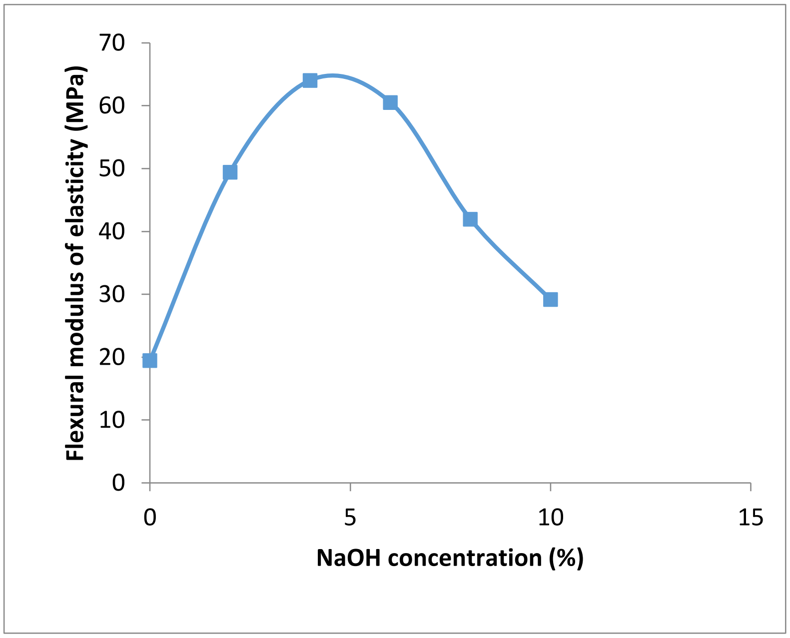
Figure 4.10: Relationship between the bending strength of the wood polymer composites and varying time of treatment when the wood dust was treated with 4wt% concentrated NaOH solution.

4.6 Effect of Concentration and Time of Treatment of NaOH Solution on the Tensile and Flexural Modulus of Wood Polymer Composite.

The effect of NaOH concentration treatment on the tensile and flexural moduli of the wood polymer samples is represented in Figures 4.11a and b respectively. Highest tensile (33.28 MPa) and flexural (64.03 MPa) moduli of the wood polymer composite samples were obtained when wood dusts were treated with 4% NaOH concentration for 30 minutes as shown in Figures 4.11a and b respectively. Moreover, Figures 4.12a and b show that both tensile and flexural moduli of wood polymer composites increase with time of treatment. Optimum values of tensile and flexural moduli of wood polymer composite were 96.10MPa and 165.10MPa respectively when wood dusts were treated with 4wt% NaOH for 150 minutes is (Figures 4.12a and b). This outcome implies that the value obtained from the analysis of the tensile and flexural modulus confirms the previous results that the best interfacial adhesion of the vHDPE polymer matrix and the reinforcing wood filler material can be obtained when the wood dust is treated in 4wt% NaOH concentration for 150 minute.

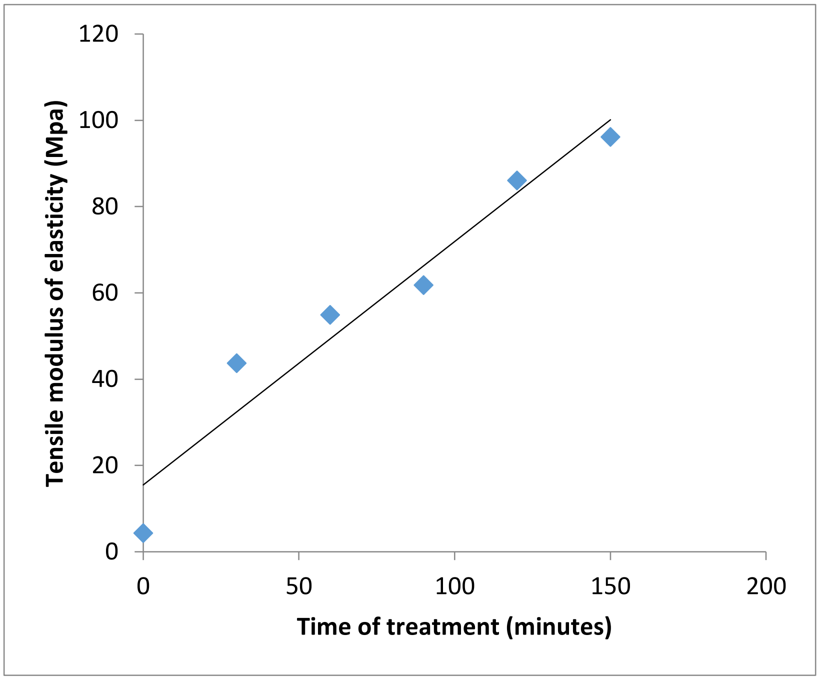
(a)



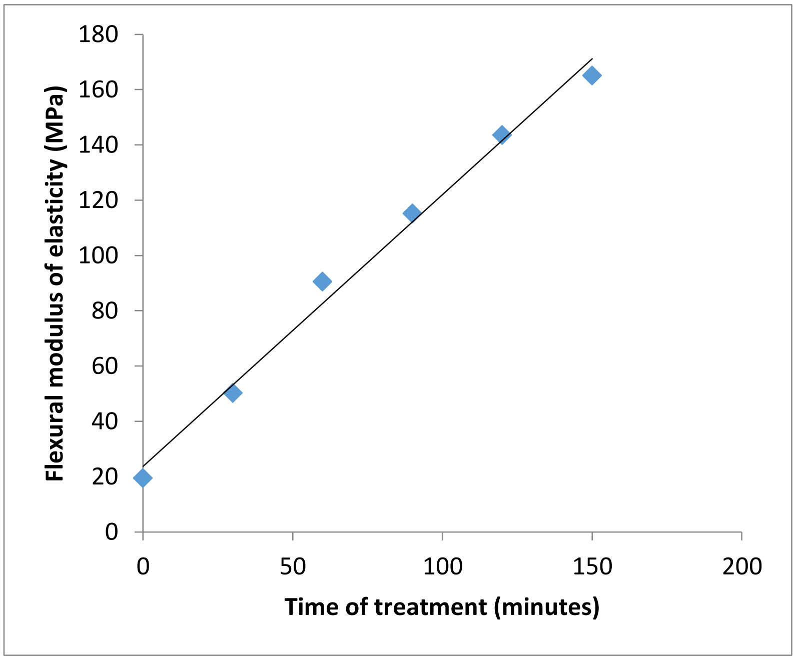


(b)

Figure 4.11: Results of modulus of elasticity (a) tensile and (b) flexural at varying NaOH concentration and fixed time of 30 minutes.



(a)



(b)

Figure 4.12: Results of modulus of elasticity (a) tensile and (b) flexural at varying time of treatment and fixed concentration of 4wt% NaOH.

4.7 Effect of Concentration and Time of Treatment of NaOH Solution on the Microstructure of Wood Polymer Composite

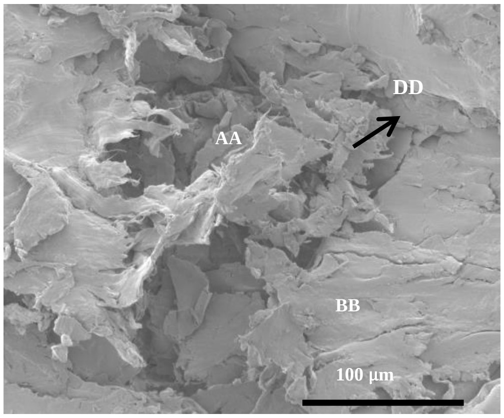
SEM fractographs obtained for wood polymer composite samples produced from wood dust treated in NaOH solution of varying concentration for different time of treatment are presented in Plates 4.13 and 4.14 respectively. Observation of the fractographs of the tensile test samples of the wood polymer composite was undertaken to further clarify the significance of fibre sticking together the test results of the mechanical properties. Plates 4.13(a) and 4.14(a) reveal that most regions on the fractographs of wood polymer composite reinforced with untreated wood dust come into view to be free of any matrix resources sticking to the wood dust (see the region labelled AA), thus signifying poor fibre matrix union. There are also few regions where the untreated wood dust partly adhered to the HDPE (see the region labelled BB in Figures 4.13a and 4.14a), thus, suggesting the occurrence of weak fibre matrix adhesion. Moreover, the untreated wood polymer composite is seen to have been futile in a breakable manner and displayed what comes up to be a substantial amount of impurities on the wood dust exterior (see the portion arrowed DD in Figure 4.13a). According to Myslamy and Rajendran (2011) and Venkateshwaran *et al.* (2013), the presence of impurities/debris on the wood dust surface is responsible for poor wood dust-matrix interface adhesion. Similar to the observation in this study, the compatibility between the untreated wood dust and the matrix is reduced and hence lower the mechanical properties when compared with the treated wood dust composites. Observation of the SEM images of fracture surfaces of the

NaOH treated wood polymer composites suggests reduction in the degree of occurrence of uneven matrix cracking and void, an indication of the good laminar bond between the treated wood dust and the HDPE (Figures 4.13b-f).The regions labelled CC and XX on the fractographs indicate occurrence of uneven matrix cracking and void in the composites' microstructure and clean surfaces of wood dust particles (pull-out) respectively. It is pertinent to note that the preponderance of regions CC and XX in the composite's microstructure suggests the occurrence of poor interfacial adhesion between the wood dust particles and the vHDPE matrix. Moreover, regions labelled YY on the fractographs indicate rough portions of the matrix found on the surface of the wood dust particles or ductile tearing of wood dust particles encapsulated with the vHDPE matrix thereby indicating better adhesion.

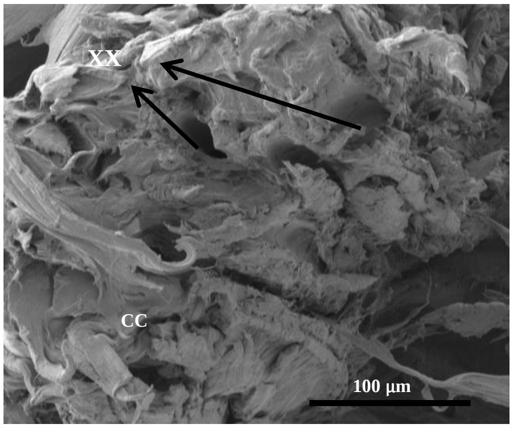
Meanwhile, comparative analysis of wood polymer composites treated with varying concentration of NaOH solution reveals greater degree of occurrence of regions CC and XX in composites reinforced with wood dusts treated with 2wt%, 6wt%, 8wt%, and 10wt% concentrated NaOH solution (Figures 4.13b, d, e, and f) relative to the composite sample reinforced with wood dust treated with 4wt% concentrated NaOH solution (Figure 4.13c). Furthermore, a study of the SEM fractographs of wood polymer composites treated at varying time at 4wt% NaOH concentration also shows increased occurrence of regions CC and XX in composites reinforced with wood dusts treated for 30, 60, 90, and 120 minutes (Figures 4.14b, c, d, and e) in comparison to the composite sample reinforced with wood dust treated for 150 minutes in 4wt% concentrated NaOH solution (Figure 4.14f). The finding from the fractographs compare favourably with the results obtained for the density and mechanical characteristics of the wood plastic composites treated at varying concentration of NaOH solution and time in which the sample treated with 4wt% NaOH concentration for 150 minutes had the highest density and mechanical properties (Figures 4.4, 4.7

to 4.12). The presence of voids in the fractographs of the sample (Plate III and IV) indicate less degree of densification and the development of less desirable mechanical properties during processing due to inappropriate choice of concentration of NaOH solution or time of treatment of the wood dust.

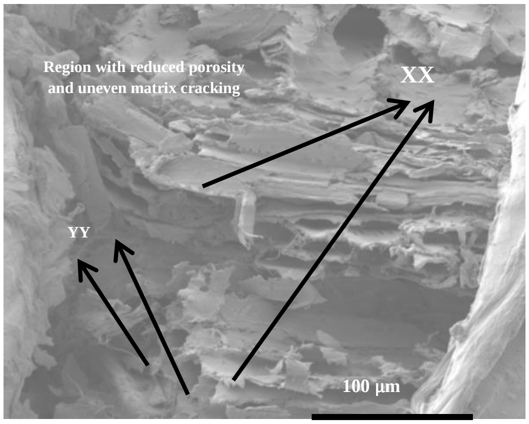
(a)



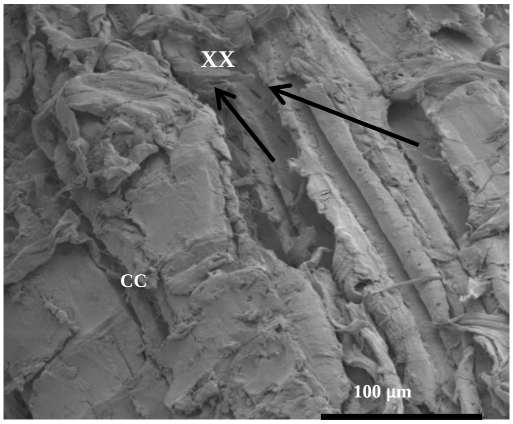
(b)

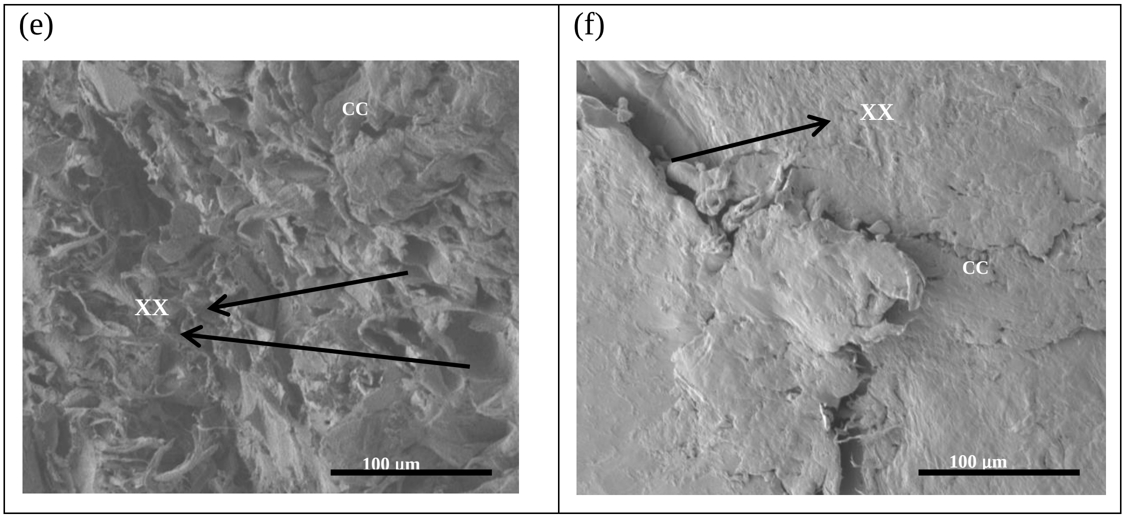


(c)

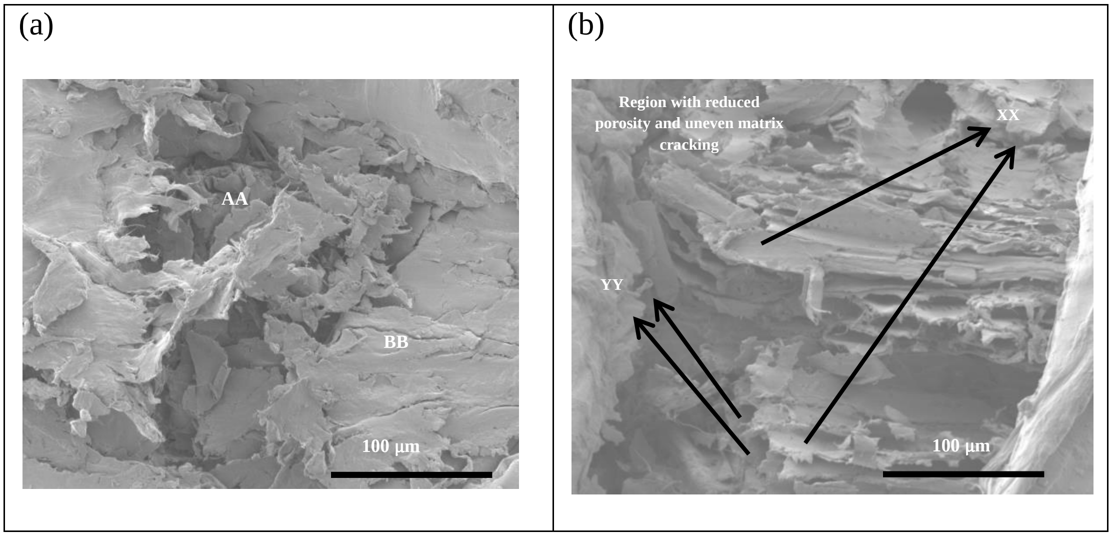


(d)





PlateIII: SEM fractographs of wood polymer composites reinforced with wood dust treated with varying concentration of NaOH solution for 30 minutes (a) Untreated (b) 2wt% (c) 4wt% (d) 6wt% (e) 8wt% (f) 10wt%



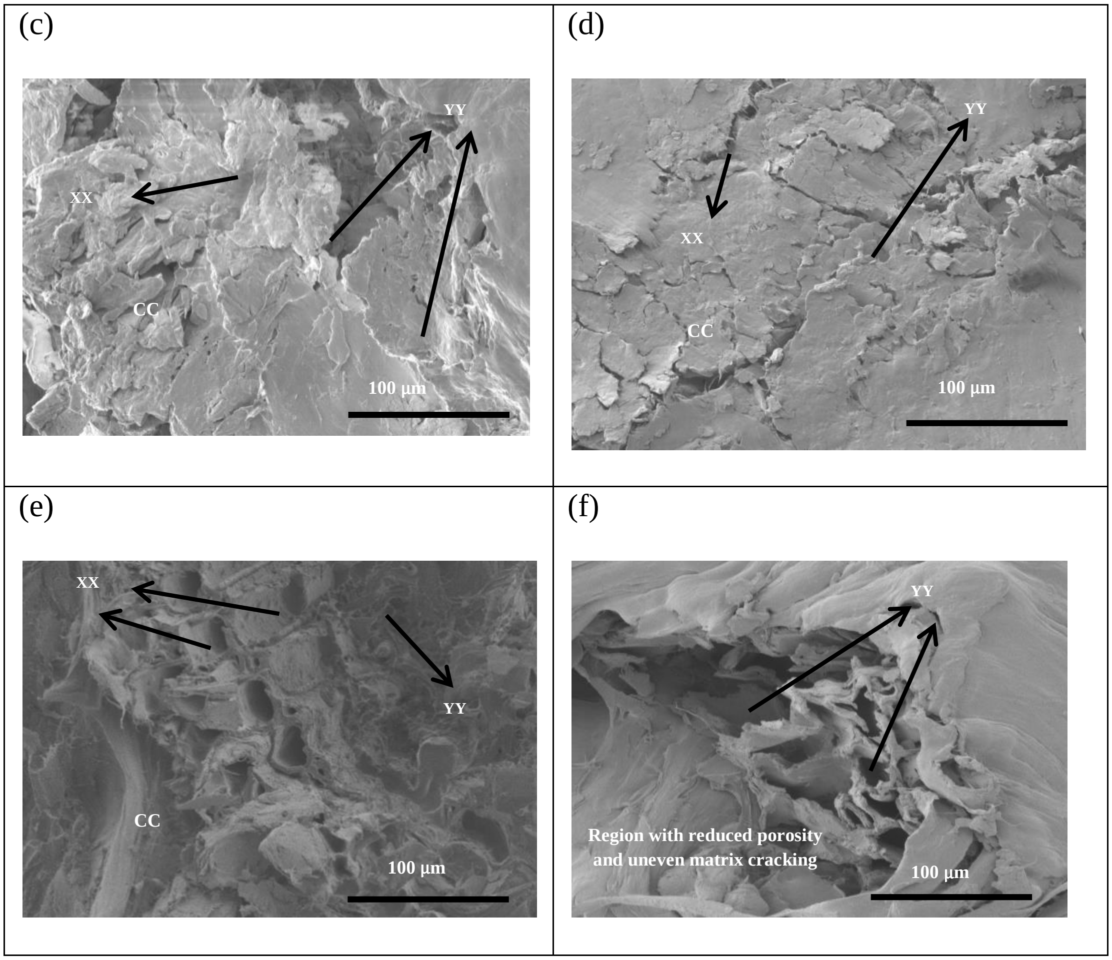


Plate IV: SEM fractographs of wood polymer composites reinforced with wood dust treated with varying time of treatment at 4wt% NaOH concentration (a) Untreated (b) 30 (c) 60 minutes (d) 90 minutes(e) 120 minutes (f) 150 minutes

The mechanical properties of all the WPCs increased after alkali treatment of wood dust at varying NaOH concentrations and time of treatment. Meanwhile, the optimisation of the mechanical properties of WPCs samples when the polymer matrix was reinforced with wood dust treated with 4wt% NaOH concentration for 150 minutes could be attributed to the elimination of non-cellulosic substance and debris, which remained scattered in inter-fibrillar section of wood dust (Gu, 2009). In this instance, treatment of wood dust with 4wt% NaOH concentration for 150 minutes makes the fibrils to become more competent for reorganizing themselves alongside the bearing of tensile deformation since the inter-fibrillar region happens to be of lesser density and lesser rigidity due to the exclusion of hemicellulose. Consequently, while fibres are stretched, changes amongst the fibrils ends up in larger stress occurrence owing to improved load input among them (Bledzki and Gassan, 2001). Therefore, as the hemicellulose drains out because of the alkali modification, close stuffing of cellulose sequence in fibre takes place due to the release of inner strain resulting to improved mechanical properties (Ray and Sarkar, 2001). When wood dusts were modified with NaOH concentration lower than 4wt% or for treatment times less than 150 minutes, the mechanical properties of WPCs deteriorate (Figures 4.7 to 4.12) because of unproductive removal of non-cellulosic substances. However, when wood dust were modified with NaOH concentration above 4wt%, the mechanical properties of WPCs gradually decreased, possibly, as a result of the increasing brittleness of the wood dust (Ray *et al.* 2002). Above 4wt% NaOH concentration, the major structural constituent of the wood dusts were affected, ensuing in the depolymerisation of native cellulose and also excessive delignification of the wood dust which affects its strength. The increasing rough surface or grooves on the surface of the wood dust particles is supposed to enhance better fibre-matrix adhesion as it provides additional sites for mechanical interlocking, thereby enhancing more matrix penetration into the wood dust surface, leading to better mechanical properties, and consequently, better mechanical characteristics of the WPCs (Alawar *et al.* 2009).

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

The following conclusions can be drawn from this study:

(1) Alkali treatment of the *Daniella-oliveri* wood dusts induced the removal of hemicellulose and lignin from its fibre.

(2) Alkali modification of wood dust improved the interfacial bond and the wet-ability of the wood dust by vHDPE resulting to the development in densification of the treated composites in comparison to the untreated samples.

(3) The density, water absorption capacity and mechanical properties of the WPCs containing treated wood dust increased with increasing NaOH concentration up to 4wt%, but over 4wt% NaOH concentration, the properties of the biodegradable composite decreased.

(4) The density, water absorption capacity and mechanical properties of the WPCs containing treated wood dust increased with increasing time of treatment in 4wt% concentration NaOH up to 150 minutes.

(5) Optimum density (1.095g/cm^3), water absorption capacity (0.35%) and mechanical properties (tensile and bending, 16MPa and 103MPa respectively) of the WPCs containing treated wood dust were obtained with treatment parameters of 4wt% concentration NaOH and 150 minutes time of treatment.

(6) The investigational outcome in the current work recommend that valuable composite with good strength could be productively developed using NaOH treated *Daniella-oliveri* wood dust as a strengthening agent for the vHDPE matrix for tape rack applications.

5.2 Recommendations

(1) This work was carried out purely using wood dust from *Daniella oliveri* and vHDPE without using any coupling agents or additives and positive results were obtained. It is thus clear that when the accurate quantity of additives and coupling agents are introduced, a better improved results will be generated hence, it is recommended that a further research be carried out using the same wood source (*Daniella oliveri*) as filler material with the addition of coupling agents and additives in order to obtain an optimum formula for mass production of wood polymer composites.

(2) In order to reduce the pollution of the Nigerian environment with used polymeric materials, it is recommended that further studies be carried out on the development of WPCs using recycled polymers and this same wood source.

(3) It is recommended that future studies should consider the production of these engineering components of various sizes and test their durability as well.

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