

Production of Wood Polymer Composite from African Copaiba Balsam

(Daniella oliveri) Wood Dust : Effects of Wood Dust Particle Size

Distribution and Contents

BY

THOMPSON, Olatunji Mohammed

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DEPARTMENT OF MECHANICAL ENGINEERING

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,

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PRODUCTION OF WOOD POLYMER COMPOSITE FROM ALKALINE MODIFIED
AFRICAN COPAIBA BALSAM (*Daniella oliveri*) WOOD DUST: EFFECTS OF WOOD
DUST PARTICLE SIZE DISTRIBUTION AND CONTENTS

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ABSTRACT

This thesis explore the effects of wood dust particle size distribution and contents on the functional properties of wood plastic composites WPCs prepared from virgin high density poly-ethylene (vHDPE) and dilute sodium hydroxide (NaOH) treated African Copaiba Balsam (*Daniella oliveri*) wood dusts via compression moulding technique. Changes in the structure and the properties of WPCs resulting from variation in *daniella oliveri* wood dusts particle size distribution and contents were studied by scanning electron microscopy (SEM) as well as physical and mechanical tests. Findings from this study suggested that appropriate choice of the wood particle size distribution and contents improved interactions between the dust and the vHDPE matrix. It could be inferred from this study that the mechanical, physical and micro-structural properties of the WPCs were optimised when dilute NaOH treated *daniella oliveri* wood dusts of particle size distribution and contents of +300-425 μ m and 35wt% respectively were blended with vHDPE matrix at 160⁰C when the pressure was 3.05 MPa within 5 seconds.. Mechanical properties of WPCs obtained from this study indicated that engineering items such as television frames for household applications could be produced with the optimised *daniella oliveri* wood dusts characteristics.

TABLE OF CONTENTS

Content	Page
Cover Page	i
Title Page	ii
Declaration	iii
Certification	iv
Dedication	v
Acknowledgments	vi
Abstract	vii
Table of Contents	viii
List of Tables	xiv
List of Figures xviii	
List of Plates	xix
Abbreviations and symbols	xx
CHAPTER ONE	1
1.0 INTRODUCTION	1
1.1 Background to the study	1
1.2 Statement of Problem	3
1.3 Justification for the study	4
1.4 Aim and Objectives of the study	5
1.5 Scope and limitation of the study	5

CHAPTER TWO	6
2.0 LITERATURE REVIEW	6
2.1 The Definition of composites and its Historical Background	6
2.2 Types of composites	8
2.2.1 Metal matrix composites	8
2.2.2 Ceramic matrix Composites	8
2.2.3 Polymer matrix Composites	9
2.2.4 Particulate Reinforced Composites	9
2.2.5 Fibrous Reinforced Composites	10
2.3 The Reinforcing Wood Fillers	10
2.3.1 The Chemical Nature of Wood	10
2.3.2 Cellulose	12
2.3.3 Hemicellulose	13
2.3.4 Lignin	14
2.3.5 Extractives	15
2.3.6 Advantages of using natural fibre (wood dust) reinforcement over inorganic fibres in composites	16
2.4 Chemical Structure of Commonly Used Polymers for Wood Polymer Composites	17
2.4.1 High Density Polyethylene (HDPE)	17
2.4.2 Polypropylene (PP)	18
2.4.3 Polyvinyl-chloride (PVC)	20

2.4.4	Polymer Selection	20
2.5	Effects of surface Treatments on the Functional Properties of WPCs	22
2.6	Surface Modification Mechanism of wood fibres with sodium hydroxide (NaOH) solution	24
2.7	Effects of Process Parameters on the Functional Properties of WPCs	27
2.8	Effects of Material Parameters on the Functional Properties of WPCs	34
	CHAPTER THREE	43
	3.0 MATERIALS AND METHODS	43
3.1	Materials	43
3.2	Methods	43
3.2.1	Wood dust Preparation	43
3.2.2	Chemical surface modification of wood fibre with alkaline solution	45
3.2.3	Mould Preparation	45
3.2.4	Mixing of vHDPE polymer and wood dust	46
3.3	Characterisation of wood polymer composites samples	48
3.3.1	Determination of Tensile Strength	48
3.3.2	Determination of Flexural Strength	50
3.3.3	Determination of Moisture Absorption Capacity	51
3.3.4	Determination of the Density of the wood polymer composite samples	52
3.3.5	Observation of the structure of wood polymer composite by	

Scanning Electron Microscopy (SEM)	53
CHAPTER FOUR	54
4.0 RESULTS AND DISCUSSION	54
4.1 Effect of wood particle size distribution and contents on the density of wood polymer composite	54
4.2 Effect of wood dust particle size distribution and contents on the water absorption capacity of wood polymer composite	57
4.3 Effect of wood dust particle size distribution and contents on the tensile Strength of wood polymer composites	60
4.4 Effect of wood particle size distribution and contents on the bending strength of wood polymer composites	61
4.5 Effect of wood dust particle size distribution and contents on the tensile and flexural modulus of wood polymer composites	64
4.6 Effect of wood dust particle size and contents on the microstructure of wood polymer composites	67
CHAPTER FIVE	72
5.0 CONCLUSION AND RECOMMENDATIONS	72
5.1 Conclusions	72
5.2 Recommendations	73

LIST OF TABLES

Table		Page
2.1	Chemical Composition of softwood and hard wood	12
2.2	Effect of various chemical treatment on the mechanical properties of polypropylene and banana fibre (BF) composites	22
2.3	Effect of melt flow index and compounding techniques on the mechanical properties of composites	30

LIST OF FIGURES

Figure	Page
2.1 Schematic picture of a softwood (gymnosperm)	11
2.2 Schematic picture of a hardwood (angiosperm)	11
2.3 Chemical structure of cellulose	13
2.4 Simple chemical structure of Hemicelluloses of a softwood	14
2.5 Simple chemical structure of lignin in angiosperm	15
2.6 Simple representation of amorphous and crystalline region of HDPE	18
2.7 Chemical structure of commonly used polymers	19
2.8 Lattice structure of cellulose I and cellulose II	25
2.9 Effect of processing time on Young's modulus of composites	29
2.10 Effect of wood fibre content on maximum tensile strength of composites	34
2.11 Effect of (a) tensile modulus of elasticity (b) maximum tensile strength (c) flexural modulus of elasticity (d) maximum flexural strength	35
2.12 Effect of particle size on (a) energy to break and (b) elongation at break	36
2.13 Effect of immersion time of composites on water uptake	38

2.14 Effect of jute fibre content on tensile and blending (flexural) strength of composites	39
2.15 Effect of jute fibre content on the tensile and flexural modulus of composites	40
2.16 Effect of wood fibre content on elongation at break of composites	41
4.1 Relationship between the density of the wood polymer composites and varying wood particle size	54
4.2 Relationship between the density of the wood polymer composites and varying wood content	55
4.3 Relationship between the moisture absorption capacity of composites of varying wood dust particle size	58
4.4 Relationship between the moisture absorption profile of composites of varying wood dust contents in vHDPE	58
4.5 Relationship between the adhesive bond between the polymer and varying wood dust particle size distribution	61
4.6 Relationship between the adhesive bond between the polymer and varying wood dust content	62
4.7 Relationship between the bending strength of the wood polymer composites and varying wood dust particle size	63
4.8 Relationship between the bending adhesion of the wood dust	

and the polymer when varying wood dust content	63
4.9 Results of modulus of elasticity (a) tensile and (b) flexural at varying wood dust particle size	65
4.10 Results of modulus of elasticity (a) tensile and (b) flexural at varying wood dust content	66

LIST OF PLATES

Plate	Page
I SEM micro-graph of polished cross-section of (a) compounded and blended 50wt% wood fibres	31
II SEM micro-graph showing PE penetrating into wood lumens in Compounded MMFI composite containing 40wt% wood fibres	33
III The mild steel mould used for preparing composites of daniella-oliveri wooddust in vHDPE	46
IV A typical wood polymer composite sample produced for this study	47
V SEM fractographs of wood polymer composites reinforced with wood dust of varying particle size distribution	68
VI SEM fractograph of wood polymer composites reinforced with modified daniella oliveri wood dust of varying contents	69

ABBREVIATIONS AND SYMBOLS

A – Cross sectional area in millimetres

ACQ – Alkaline Copper Quaternary

Al₂O₃ – Aluminium Oxide

ASTM – America standard of Testing Machines

b – Width of specimen

CCA – Chromate copper arsenate

WPC – Wood Plastic Composite

NaOH – Sodium hydroxide

vHDPE – Virgin High Density Polyethylene

SiC – Silicon carbide

PE – Polyethylene

PP – Polypropylene

PVC – Polyvinyl chloride

LDPE – Low Density Polyethylene

MMFI – Medium Melt Flow Index

LMFI – Low Melt Flow Index

SEM – Scanning Election Microscopy

MPa – Mega Pascal

GPa – Giga Pascal

T – Temperature

TP – Tensile Properties

FP – Flexural Properties

Eb – Elongation at break

σ - Stress

ε - Strain

F – Force in Newton

Δl – Difference in length

l – Original length

F_s – Flexural strength

F_m – Flexural modulus

h – thickness of specimen

W₁ – weight of sample before soaking in water

W₂ – weight of sample with soaking in water

ρ - Density

m – mass in grams

v – volume of water displaced in cm³

MTS – Maximum Tensile Strength

M_t – moisture content

F_l – Applied load

m – slope of the tangent

L – length of the support span

μm – micro-meter

CHAPTER ONE

1.0

INTRODUCTION

1.1 Background to the Study

Generally, woods are mainly grown for the production of furniture, roof frames and related constructions. Therefore, most of the woods from tree plantations end up in wood factories or saw-mills. As a result, large quantities of saw-dust or wood dust particles are generated as waste which needs to be disposed of because of their degrading effect on the environment and health hazards. The high availability of low value wood dust as a result of milling and their negative environmental impact is a concern to researchers and industrialists. This has brought about the long search to convert low value wood dust into other high value products. The focus then was to combine wood dust with thermoplastic resin which resulted in wood polymer composites (WPCs). This development attracted huge interest of the manufacturers and the consumers due to their added value aesthetics. As a result, average yearly increase in demand for WPCs products between 2003 and 2007 improved. This has grown more than ten times the polymer market (Wood-plastic, 2008). Wood dusts have high specific strength and modulus, they can be bought at reduced prices with assistance of workers in the industry and above all, it is natural (Shinoj, P., Raju, S.S, and Joshi, P.K, 2011). Furthermore, at the expiration of their useful period, they can be disposed off either to be used to produce more composites or by recovery of their calorific values in a furnace which is not possible with conventional inorganic fillers like talc, calcium carbonate, clay, carbon, non-ferrous metals and glass fibre composites (Kuo, P.Y., Wang, S.Y., Chen, J.H., Hsueh, H.C. & Ysai, M.J, 2009).

However, wood dusts show some un-required peculiarities like having affinity for water absorption and easily degraded by heat (Tajvidi, M., Najafi, S.K. and Moteei, N, 20006). Thermoplastic resins such as polypropylene (PP), polyethylene (PE), polystyrene (PS) and polyvinylchloride (PVC) soften (lowering viscosity) when heated and harden (increasing viscosity) when cooled. These properties allow materials such as wood dust particles to be mixed with the plastic to form composites of many products and shapes which could also be recycled. However, the polar wood dusts (hydrophilic) naturally do not adhere with non-polar polymers (hydrophobic) as a result of incompatibility between wood dusts and the polymer matrix. As a result of this poor interfacial adhesion between wood dust and thermoplastic composites, problems such as debonding of dusts within the interface of matrix may occur (Kuo *et al*, 2009). For economic reason, wood dusts are often subjected to various surface treatments and modification. The degree of molecular interaction within polar dust and non-polar polymer is the essential characteristic of WPCs. To optimise WPCs engineering performance, research work is currently exploring the effects of various surface treatments and methods, as well as materials with a view to improving matrix-reinforcement interfacial bonding, hence, the functional properties.

This research work makes use of soft wood particles of African Copaiba Balsam Tree (*Daniella-oliveri*) waste materials from a local saw-mill in Maitumbi area of Minna in Niger State causing environmental and health hazards to the community. *Daniella-oliveri* is abundantly available in Nigeria. It is called *Kadaura (Maje)* in Hausa language, *Danchi* in Nupe language, *Iyaa* in Yoruba language, *Oziya-ato* in Bini language and *Ozabua* in Igbo language. The waste wood particles were modified with dilute sodium hydroxide (NaOH) solution in order to improve their interfacial adhesion before blending with virgin high density polyethylene (vHDPE). The mechanical, physical and micro-structural characteristics

of the composites were investigated by varying wood particle size distribution and their contents with a view to determining the optimal particle size and content for optimising the functional properties of WPCs.

1.2 Statement of Problem

Solid wood has been traditionally utilised for different applications in many parts of the world. It has been argued that the effects of pollution and inhalation of chemicals used in treating solid wood were unhealthy and the resulting degradation of the wood materials on usage in those applications have brought about shift to replace solid wood with WPCs (Smith, P.M. and Wolcot, M.P, 2005). The shift is largely due to the improved properties and environmentally friendly attributes of WPCs one of which is colour. Indeed, colour is an attribute that adds value to wood-polymer based products. The demand for value-added wood based composite products were higher than the conventional wood products in the market. These could be attributed to quality and aesthetic appeal shown by wood based composites (Bennington, 1996). Also in the past, problems associated with compatibilisation of polymer with inorganic materials (fillers) like carbonates, talc, carbon and glass fibre have made composites of these reinforcements for both domestic and industrial applications undesirable. The undesirability of these reinforcements are due to the following reasons:

- (i) The cost of compatibilising the fillers with the host polymer is quite high.
- (ii) The weight of their composites after compounding was undesirable.
- (iii) Related production and transportation cost was also quite irrational.

(iv) The environmental effect (pollution) was unhealthy.

(v) Dermatological effect on employees were devastating.

Therefore in this study, it is believed that material composition contribute significantly to mechanical and physical properties of WPCs. Hence, it is imperative to first understand the chemical constituents and nature of woods and plastics in order to produce quality WPC products.

1.3 Justification for the Study

Nigeria, being an oil producing country and having the plant; *Daniella-oliveri* dotting her landscape can annex the two resources for production of non-structural components. The economic advantages of the two materials could be utilised to solve the problem of job creation; employment for the teeming population in the country and reduction of inclusive capital flight. Furthermore, the multiplying economic effects will be a plus in re-generating employment, conserving our resources and generating foreign exchange for the country, diversification of the economy, reservation of petroleum based products, exportation and reduction of capital flight from the country.

1.4 Aim and Objectives of the Study

The primary focus of this research is to find the optimal influence of particle size distribution and contents of wood dust of *Daniella-oliveri* modified with dilute sodium hydroxide

(NaOH) solution on the properties of WPCs when blended with virgin high density polyethylene matrix. The objectives of this study are as follows:

1. To investigate the effects of particle size distribution of modified *Daniella-oliveri* wood dust blended with virgin high density polyethylene (vHDPE) matrix on the functional characteristics of the resultant composition.
2. To investigate the effects of wood dust contents of modified *Daniella-oliveri* blended with virgin HDPE matrix on the physical and other functional characteristics of the produced composites.
3. To investigate the effects of particle size distribution and contents of modified *Daniella-oliveri* wood dust on the adhesion bond between the dust and the polymer matrix as well as its inter-relationship with the mechanical properties.
4. To identify the optimal wood dust particle size distribution and content for optimising the functional properties of WPCs.

1.5 Scope and Limitation of the Study

The scope of this research is using derived optimal 4% dilute concentration of sodium hydroxide (NaOH) solution and time of 150 minutes to modify wood dust of *daniella-oliveri* in order to determine the optimal wood dust particle size distribution and contents on composites made from virgin high density polyethylene (vHDPE) in relation to their mechanical and physical properties for non-structural applications. The study is limited by the available particle size distribution supplied by the saw-mill.

CHAPTER TWO

2.0

LITERATURE REVIEW

2.1 The Definition of Composites and its Historical Background

Composites are produced by combining the properties of two or more materials to achieve desired objectives which could not be obtained with the separate materials. Hence, WPCs are the combination of wood dust or flour or fibre and polymers to attain a synergy of the materials which could not be achieved with the separate resources. For example, combining properties of wood dusts with thermoplastic materials to achieve composites with higher mechanical properties. Therefore, composite materials are in proportional percentages blended together. The materials are of two types; discontinuous and continuous phases. The discontinuous phases are usually harder and stronger than the continuous phase and are called reinforcement or reinforcing materials whereas, the continuous phase is termed the matrix which is more ductile and less hard. The reinforcement serve to strengthen composites and improve the overall mechanical properties of matrices.

Historically, wood plastic composites (WPCs) have been utilized for over two twenty years but demand for them have greatly increased. The United States of America was the first set of consumers to use WPCs in 1983. There production started with flat sheets of equal wood/resin volume ratio; equal volume of wood dust and polypropylene (PP) in the manufacture of three dimensional (3D) products being use in automotive industry for non-structural applications (Clemons, 2002). An affiliate of Mobil Oil, Trex Company was one of the pioneers in the production of the WPCs as a way of saving cost by compositing recycled plastics. In 1990s, Trex Company and Advanced Environmental Recycling Technologies started production of

composites of equal volume ratio of wood dust and polyethylene (PE) for landscaping and decking needs (Clemons, C, 2002). As a result of the new innovation, increase in consumer consumption for decking increased as a result of marketing innovations and aesthetics of the products. In view of these facts, demand for composite decking had been increased at yearly rate of 17.3% (Clemons, C, 2000). Meanwhile, as a result of high demand for WPCs product, wood market share in the decking market caused reduction in wood market from 95% in 1977 to about 83% in 2007 (Lauzon, 2004). Increase in for WPCs was attributed to low cost, low weight, comparative advantages over inorganic composites and aesthetics. However, the demand for wood polymer composites for decking materials declined because of the health hazard caused for the use of chromate copper arsenate (CCA) in the treatment of compressed wood.

Decking materials treated with CCA were unavailable as at January, 2003 to consumers (Taylor, 2004). CCA had been replaced by another chemical (alkaline copper quaternary, ACQ) that had performed equally in the treatment of lumber with CCA. Therefore, treatment of pressured wood was economically not rational. The duration of exploitation of ACQ was quite short due to its high cost. The gap created between high cost of ACQ and low cost CCA spurred the market growth. Due to market growth, new technologies were introduced to produce goods at lower cost and better output design. As a result of new technologies, second generation WPC materials were produced that out-perform their predecessors. This development was accomplished with introduction of new processing machines, better researched improved additives, wood dust and plastic type (Markarian, 2002). For WPCs to be used where less pressure is required, a new approach must be evolving.

2.2 Types of Composite

Composites can be characterized on the bases of the type of matrix and reinforcement used (Amar, K.M., Manjusri, M, and Lawrence, T.D, 2005). For example, metal matrix composites, ceramic matrix composites and polymer matrix composites. Composites may also be grouped on the basis of the type of reinforcement employed such as particulate reinforced composites and dust or flour reinforced composites.

2.2.1 Metal Matrix Composites

A composite material consisting of metal as matrix is called metal matrix composite (Amar *et al*; 2005). It has many uses over non-alloyed metals like higher specific strength and modulus, lower coefficient of thermal expansion and better properties at elevated temperatures. Due to the above benefits, different applications of composite of metals are being considered for their uses. For example, nozzle of engines, electrical conductors and structural members of housing are produced from composites of metal.

2.2.2 Ceramic Matrix Composites

If the matrix material is ceramic, then the composite is called ceramic matrix composite (Amar *et al*; 2005). The primary objective in ceramic matrix composite production is to improve rigidity. Generally, whenever composites of ceramic matrix are produced, there is always correlation of improvement in strength and stiffness. The most well known materials for making ceramic composites are SiC (Silicon Carbide) and Al₂O₃ (Aluminium Oxide).

2.2.3 Polymer Matrix Composites

The matrix materials often used in composites are polymeric. There are two reasons for these; polymeric material strength are not appropriate for many structural applications, their stiffness and rigidity are lower in comparisons to metals and ceramics. As a result of these challenges, polymers are reinforced with polymers (Drzal, L., Mohanty, D., Burgueno, R and Misra, M, 2004). Secondly, composites production processing of polymer matrix did not accommodate high temperature and pressure. Also, machines being use in the production of composites of polymer matrices are lighter in weight and easier to operate. As a result of these reasons, composites of polymer matrices became known and soon being used for structural applications.

2.2.4 Particulate Reinforced Composites

If the reinforcement in composite material is in the form of particle, then the composite material is called particulate reinforced composites. They may have different shapes while their sizes are equal. It is known that particles of wood do not contribute to adhesion development in fracture resistance of composites. However, they improve stiffness of composites to some extent (Belmares, H., Barrera, A, and Monjaras, M, 1983). Wood particle reinforcement are generally explored in improvement of composites by encapsulating in matrices to improve thermal and electrical performances, better resistance at higher temperatures, lower friction and improve wear. They are also use in contact resistance, better machinability, surface hardness and lower shrinkage. Furthermore, dispersion of particulate reinforcement in composites could be randomly oriented or preferred.

2.2.5 Fibrous Reinforced Composite

If the reinforcement is in the form of dust or flour, the composite material is called fibrous reinforced composite. Wood dust is classified by its length being much higher in relation to its cross-sectional dimensions. The capacity of reinforcements in contributing to the strength of composites is a function of the dimensions. Generally, dusts are quite effective in making better fracture resistance of polymeric matrix. Moreover, reinforcements with long dimension wood fibre did not allow development of initial breakages incidental to the fillers that could lead to fracture, especially when the matrices are brittle (Razi, P. S., Portier, R, and Raman, A, 1999).

2.3 The Reinforcing Wood Fillers

2.3.1 The Chemical Nature of Wood

Woods can either be gymnosperms (soft woods) as shown in Figure 2.1 or angiosperms (hard woods) as shown in Figure 2.2. Soft wood is made up of a single cell, longitudinal tracheid. The make up of the tracheid is not complex in relation to that of hardwood. The length of wood dust is highly variable but averagely about 1mm for hardwood and 3-8 mm for softwoods (Miller, 1999).

Both softwoods and hardwoods are lignocellulosic materials; made up of complex chemical compositions that include macro-molecular cell wall components; cellulose, hemicellulose, and lignin and an array of low molecular mass compounds known as extractives (Miller,

1999). Table 2.1 shows the percentage by weight of both softwood and hardwood (Sjostrom, 1993). Wood dusts are a production of both chemical and mechanical forms for composites. Wood dusts are mainly produced by mechanical means.

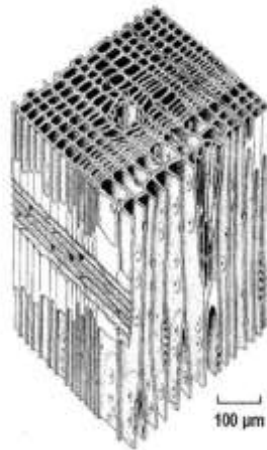


Figure 2.1 Schematic picture of a softwood (gymnosperm) (Source: Marino, 2010).

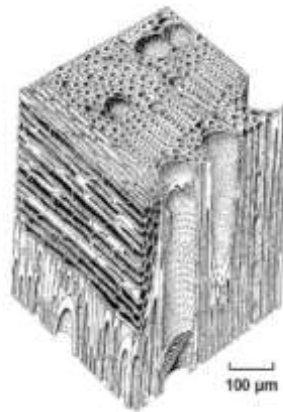


Figure 2.2: Schematic picture of hardwood (angiosperm) (Source: Marino, 2010).

Table 2.1: Chemical composition of softwood and hardwood by weight percent (Sjostrom; 1993).

Wood type	Cellulose%	Hemicellulose%	Lignin%
Hardwood	40-44	15-35	18-25
Softwood	40-44	20-32	25-35

2.3.2 Cellulose

Cellulose, the major component of the wood dust, consists of inter-connected chains. It is located in the secondary wall and constitute about 40-44% of total dry weight of the wood cell wall (Sjostrom, 1993, Miller, 1999). Most part of cellulose is believed to be crystalline and bonded together by inter-molecular hydrogen bonding (Hill, J., Nelson, E., Tilman, D., Polasky, S, and Tiffany, D, 2006). As a result of its high degree of polymerisation and crystallinity, cellulose is responsible for strength in wood (Pandey, 1999). It is generally believed that the presence of polar crystals in cellulose is responsible for the hydroscopic nature of wood. Therefore, cellulose is hydroscopic due to its polar molecule and as a result undergoes hydrogen bonding (Hill *et al*; 2006). However, the hydroxyl groups in the crystallites are linked with each other; they are not free, therefore, they are not susceptible to reaction with water. More importantly, only hydroxyl groups that are present in the amorphous region of cellulose are available for chemical reactions (Miller, 1999). Figure 2.3 shows a simple chemical structure of cellulose wood dust.

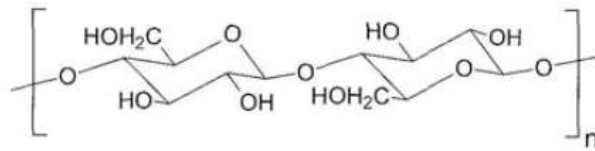


Figure 2.3: Chemical Structure of Cellulose. (Source: Marino, 2010).

2.3.3 Hemicellulose

Hemicelluloses are alkaline extractable hetero-polysaccharides that are branched and of lower molecular weight than cellulose (Miller, 1999). Hemicelluloses represent about 20 - 32% of wood and are predominantly found in the primary and secondary cell walls (Sjostrom, 1993, Baeza and Freer, 2001). The main constituents of hemicelluloses in soft woods are glucose, mannose, galactose, xylose, arabinose and multiple-methyl soluble glucuronic acid whereas hemicelluloses in hardwoods consist of simple glucose and multiple-methyl soluble glucuronic acid (Rowell, R. M., Petterson, R., Han, J.S., Rowell, J. S, and Tshabalala, M.A, 2005). Simple chemical structure of hemicellulose of a soft wood is shown in Figure 2.4. Hemicelluloses differ from cellulose because they contain various sugar units with much shorter chains and are branched chain molecules (Baeza and Freer, 2001). Hemicelluloses are more reactive than cellulose mainly due to their amorphous nature. The combination of cellulose and hemicellulose is known as holocellulose.

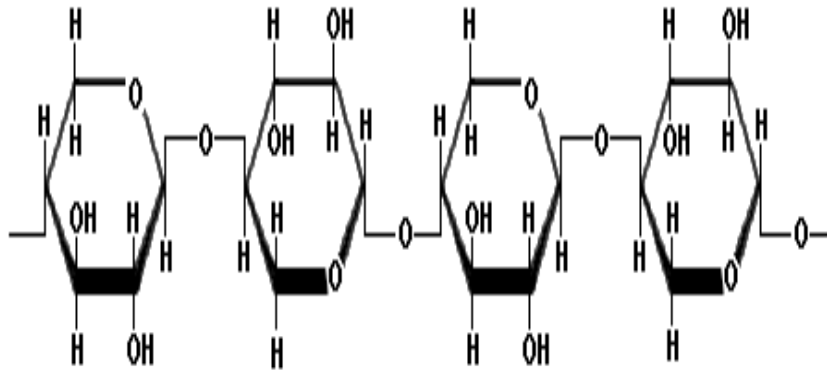


Figure 2.4: Simple chemical structure of Hemicellulose of a soft wood. (Source: www.google.com.ng).

2.3.4 Lignin

Lignin is widely spread round the cell wall but mostly found in the middle lamella region and accounts for about 18-35% of wood (Sjostrom,1993).It is characterised by the presence of different functional groups such as phenolics, methoxyls, aliphatic alcohols, aldehydes, ketones, and ethers (Sjostrom,1993). It is the matrix associating cellulose fibrils and hemicelluloses, thereby forming a supra-molecular structure known as the wood cell wall. It is believed that lignin content of softwood is higher than that of hardwood with 23-33% and 16-25% respectively (Fengel and Wegener, 1984; Sjostrom, 1993; Miller, 1999). Lignin has low occurrence of hydroxyl groups compared to polysaccharide components (Fengel and Wegener, 1989; Winandy and Rowell, 2005). It has been suggested that lignin is responsible for the stiffness of wood and non-accommodation of water by encapsulating crystal structure of carbohydrates (Fengel and Wegener, 1989; Winandy and Rowell, 2005). This is due to lignin's hydrophobic nature and its capability of encapsulating cellulose and hemicellulose

carbohydrates. Figure 2.5 shows the lignin precursor (synapyl alcohol) in angiosperms (Sundar, S. T., Mc Donald, A. G and Wolcott, M, 2004).

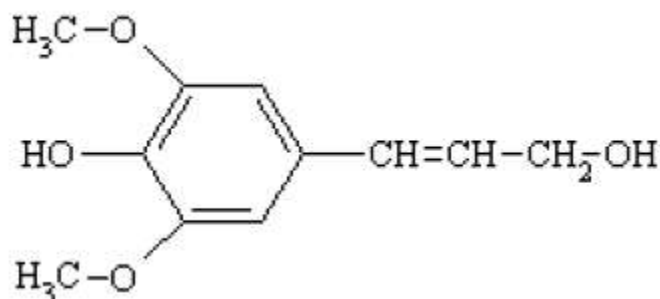


Figure 2.5: Simple chemical structure of lignin in angiosperms. (Source: Sojstrom, 1993).

2.3.5 Extractives

Extractives are generally low molecular weight compounds that are readily obtained by solvent extraction of wood (Baeza and Freer, 2001). Extractive constituents and contents can vary from 1 to 30% of wood, depending on species, growth conditions, soil and time of the year the wood is harvested (Miller, 1999; Hill *et al*; 2006). Wood extractives contain both organic and inorganic components which influence wood properties such as colour, odour, taste, decay resistance, density, hygroscopicity, and flammability. These organic components of extractive content of wood include tannins and other poly-phenolics and simple metabolic intermediates which have various uses while inorganic components are sodium, calcium, iron, chloride and silicates.

2.3.6 Advantages of using Natural Fibre (Wood Dusts) Reinforcement over Inorganic Fibres in Composites

Preference for natural fibres (e.g. wood dusts) over inorganic materials as reinforcements in polymer matrix with a view to producing wood polymer composites (WPCs) is gaining interests among industrialists and academic researchers due to the following reasons:

1. Wood dust is environmentally friendly as it is fully bio-degradable.
2. Wood dust is cheap and abundantly available;
3. Wood dust has lower density, therefore, composites produced from it are lighter compared to glass, carbon and aramid fibre;
4. They are cheap and are used in the production of high quality goods which fulfil the economic interest of industries;
5. The release of carbon-dioxide after their useful life is neutral;
6. The advantage of non-abrasiveness of dust on polymer processing equipment during compounding to composites;
7. Natural wood dust composites are used in non-structural applications in place of glass for aesthetics e.g. automotive components.

Although, the reinforcement of polymer matrix with wood dust promotes environmental friendliness and renewability of dust however, they have the following drawbacks:

1. They have poor wettability, therefore, they have poor adhesion with polymeric matrices.
2. They are hygroscopic in nature.
3. Composites with unmodified dusts exhibit unsatisfactory physical and mechanical properties.

4. Mechanical properties of wood dust are much less compared to glass dusts while their precise characteristics e.g. rigidity and toughness could be compared to glass dusts.

To overcome these challenges, surface treatment or compatibilisation techniques employing sodium hydroxide solution, permanganate of maleic anhydrides, peroxide, benzoyl chloride, acrylic acid and silane have been used.

2.4 Chemical Structure of Commonly Used Polymers for Wood Polymer Composites

In this section, attention is paid to polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC) with a view to gaining an understanding of how each of the polymer matrix differ in properties and influence the functional properties of WPCs. Comparative analyses of the properties of these polymers are also undertaken with a view to providing justification for the use of the choice polymer in this study.

2.4.1 High Density Polyethylene (HDPE)

This is made from ethylene ($\text{CH}_2=\text{CH}_2$) by an ionic polymerization process that results in higher relative molar mass. HDPE is a linear polymer; it does not branch. Its molecular structure is made up of hydro-carbon without other elements. It is crystalline in nature which makes it stronger than low density polyethylene (LDPE) or poly-vinyl chloride (PVC) that are amorphous in nature. HDPE density ranges between 0.950 and 0.965 g/cm^3 (Chanda, M., and Roy, S. K, 1998). However, the reduction in energy absorbing amorphous phase of HDPE also reduces its toughness. HDPE have the following characteristics; excellent chemical resistance, lightweight, resilience, abrasion resistance, high impact, high tensile

strength, stress and crack resistance and an economical thermoplastic. Therefore, HDPE is often used in a variety of products such as composites, pipes and bottles. Figure 2.6 shows a representation of amorphous (jelly) and crystalline (cellulose) regions of HDPE.

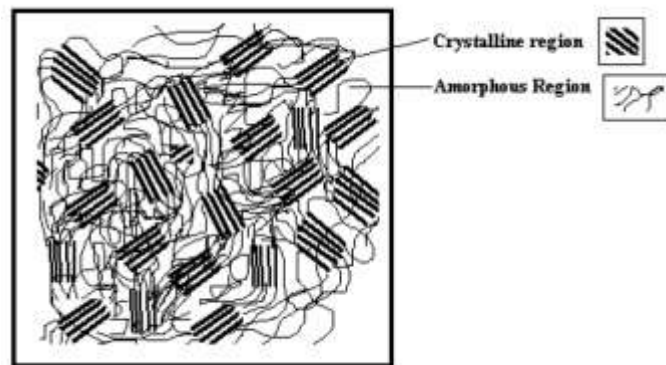


Figure 2.6 : Simple representation of jelly and cellulose regions of HDPE (Source: Sperling, 1992).

2.4.2 Polypropylene (PP)

This is polymerised from propylene ($\text{CH}_3(\text{CH})=\text{CH}_2$) and has a methyl(CH_3) side group at every other carbon along the molecular backbone (Chanda and Roy, 1998). PP molecules have a methyl(CH_3) side group at every other carbon along the molecular backbone that makes it highly aligned and compact resulting in high durability and resistant to chemicals. PP can be either isotactic or atactic based on the arrangement of the methyl side groups. PP that has all the one atom of carbon and three atoms of hydrogen set on one part of the carbon asymmetry is called isotactic while PP with random arrangements of methyl side groups is

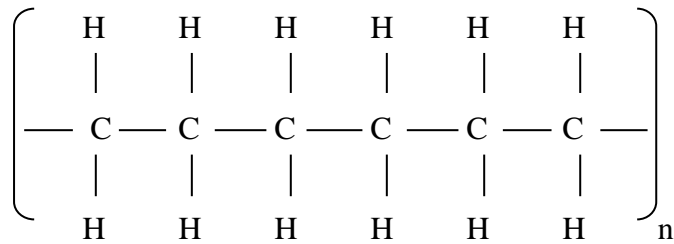
called atactic. Commercial homo-polymers of PP are usually about 90-95% isotactic while the other structures being atactic and syndiotactic (alternating arrangement of methyl groups) (Chanda and Roy, 1998). PP is very similar to HDPE but exhibits a lower density (0.90g/cm^3) and a higher softening point which makes it to withstand boiling water and many sterilizing operations (Chanda and Roy, 1998). PP exhibit high tensile strength and stiffness at high temperatures better than HDPE. It is an economical, lightweight thermoplastic and render high corrosion, impact and abrasion resistances.

Figure 2.7 shows chemical structures of commonly used polymers for composites.

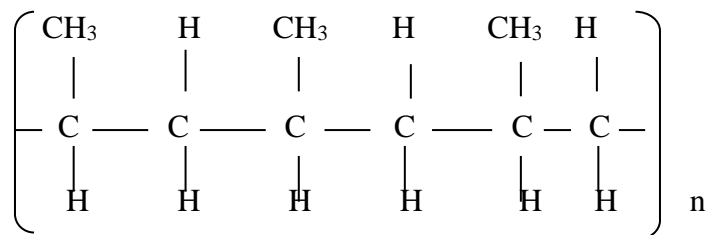
Polymer Type

Structure

PE C_6H_{12}



Isotactic PP



Isotactic PVC

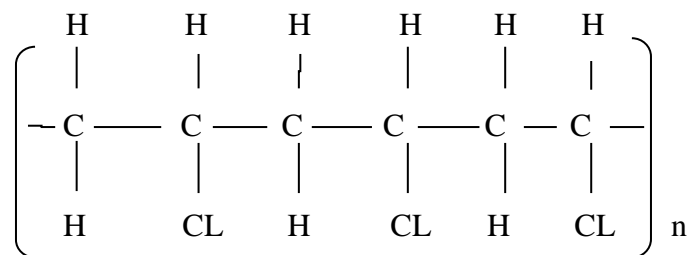


Figure 2.7: Chemical structures of commonly used polymers (Chanda and Roy,1998).

2.4.3 Polyvinyl-chloride (PVC)

The molecular structure of polymer molecules are often bond with chains of other elements such as chlorine, fluorine, nitrogen and silicon. The bonding of these elements with the carbon backbone of polymers give them distinct characteristics. For example, the substitution of hydrogen atoms with chlorine atoms along the backbone gives polyvinyl chloride (PVC). PVC is amorphous plastic synthesized from the organic compound vinyl chloride ($\text{CH}_2=\text{CHCl}$). Chlorine atoms bonded to the carbon asymmetry of it structural chains give polyvinyl chloride its strong and flame retardant characteristics. It is the most widely used of the amorphous plastics. It is durable, lightweight and waterproof. The commercially available PVC contain stabilizers. PVC is weather-resistant and in its rigid form, it is extruded into house siding, pipe and gutters (Chanda and Roy, 1998).

2.4.4 Polymer Selection

The two major classes of polymers, thermosets and thermoplastics may be classified in terms of their responses to changes in temperature. Thermosetting polymers change structure and harden in stiffness when temperature first changes. On the other hand, thermoplastic

continuously soften or melt as temperature increases. The molecular anatomy of the polymer chains can also be used to characterize thermoplastics. Polyolephines show both amorphous (Jelly) and semi-crystalline (crystal) chemical structures. Jelly thermoplastics comprise of chemically unstructured chemical interaction such as amorphous polymers. The other polymers such as semi- crystalline polymers show different forms of crystallinity that distinguishes their mechanical and physical characteristics. A chemically structured order of molecular chains with appropriate inter-molecular cohesion to guide against dismemberment from heat energy is required to form a three-dimensional crystalline lattice (Rosen, 1993). However, response to temperatures by crystalline polymers differ to amorphous polymers. The crystal structure of the polymers metamorphous into an initial stage melt phase transition when temperature increases (Sperling, 1986). Again, jelly polymers do not show true melt stage though, undergo disengagement of their molecular chains to form a phase of medium melt flow index like melting.

There are many types of thermoplastic that can be selected for production of wood polymer composites. Though, only few of them can be used in this case. The main reason for using thermoplastics in WPCs is the melting or softening temperature that is lower than heat disintegration temperature of wood which is about 210^{0c}. Also, HDPE polymer matrix possesses one of the lowest coefficient of linear thermal expansion and low thermal processing temperature relative to PP and PVC, thus minimising wood flour degradation (Fabiya, J.S., Armando, G. M., Michael, P.W, and Peter, R.G, 2008). These thermal criterion, coefficient of expansion and processing temperature restrict the choice to the group of polymers known as polyolephines. Thermoplastics that are appropriate for WPCs production temperature changes are polystyrene (PS), low and high density polyethylene (LDPE and HDPE), polypropylene (PP) and polyvinyl chloride (PVC). Of all the mentioned

thermoplastic, polyethylene is most commonly used and takes about 75 percent share of the total plastic utilized in the production of WPCs (Fabiya *et al*, 2008).

2.5 Effects of Surface Treatments on the Functional Properties of WPCs

The influence of modification is to enhance adhesion bond between the polar natural fibres and the non-polar matrix. Chemical treatment of reinforcing fillers resulted in better wetting, dispersion of wood fibres in the matrix and the consequential good interaction between the components as well as improved mechanical properties of WPCs as shown in Table 2.2 when banana fibres (BF) were modified with different types of chemical modifications such as sodium hydroxide (NaOH) solution, stearic acid, benzoyl chloride, vinyl trimethoxy silane, and potassium permanganate (KMnO₄).

Table 2.2: Effect of various chemical treatments on the mechanical properties of polypropylene and banana fibre (PP/BF) composites (Source: Paul *et al.*, 2010).

Composites	Tensile strength (MPa)	Young's modulus (MPa)	Elongation at break (%)
Untreated fiber composite	31 ± 1.3	1521 ± 35	6.0 ± 0.8
NaOH (10%) treated fiber composite	40 ± 1.7	1660 ± 26	3.4 ± 0.2
Stearic acid treated fiber composite	37 ± 1.7	1650 ± 26	4.5 ± 0.5
Benzoylated fiber composite	35 ± 0.9	1595 ± 36	4.0 ± 0.9
Vinyl trimethoxy silane	34 ± 1.0	1615 ± 24	4.0 ± 0.5
KMnO ₄ treated fiber composite	33 ± 1.0	1635 ± 34	4.0 ± 0.5

According to Paul, S. A., Sinturel, C., Joseph, K., Mathew, G., Pothan, L. A, and Thomas, S, (2010), composites of wood fibre modified with 10% NaOH solution were found to have appropriate mechanical properties while the tensile strength of all treated composite samples increases in comparison to the composite containing un-modified wood dust. However,

elongation of the NaOH treated sample was discovered to be less in properties than modified composites. The reason for this could be due to strong inter-molecular interaction within fibres and the matrix because of alkaline surface modification of the fibres. Also, according to Paul *et al*, (2010), the positive influence of the chemical treatments of the BF on the composites could be attributed to the fact that the elastic moduli of BF were discovered to increase linearly as molecular dispersion increases consequence upon elimination of complex molecular compounds like hemicelluloses, lignin and pectin by caustic soda which led to improve dispersion of fibres and homogeneous alignment of it molecules within the polymer matrix.

In the light of findings from the work of Paul *et al*, (2010), alkaline treatment was employed in the present study for the surface modification of *Daniella oliveri* wood dust due to the following reasons.

1. Alkaline modification exposes the crystallite structure of cellulose for adhesion with polymers..
2. Alkaline modification depolymerises the natural fibre bundles (breaking down hydrogen bonding) and fibrillating it to increase area of contact with thermoplastics when compounding to form composites.
- 3 In view of the above statements, alkaline treatment of wood dust can improve distribution, wetting and penetration of polymer matrix into fibre lumens. These actions promote mechanical interlocking between the two components for good adhesion. The treatment preclude the use of coupling agents in the composite formation.

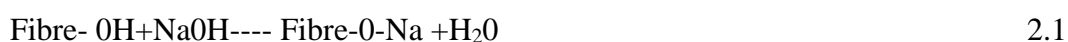
- 4 In general alkaline treatment can improve wettability of wood dust and reduce the need for coupling agent in wood plastic composite processing.

2.6 Surface Modification Mechanism of Wood Fibres with Sodium Hydroxide (NaOH)

Wood and plastics are non-related materials in view of hydroxyl components of wood dust (moisture absorption) while polymer non-hydroxyl (moisture repellent). These apparent difference between wood and plastic results in poor adhesion when the two components are in composites. Due to poor interaction between the components, stress transfer is reduced at both low and high wood loadings. To improve interfacial interaction between the components, surface improvement of wood dusts are usually take place. By improving interfacial adhesion between the components, stress transfer is made better; mechanical properties maximized (Keener, T. J., Stuart, R. K., and Brown, T. K, 2004). The present industrial method has been the use of coupling agents such as, maleic anhydride polyethylene (MAPE). Apparently, coupling agents play large roles in improving interfacial adhesion between wood fibre and polymeric materials. However, based on findings from Paul *et al*, (2010), this research is utilising an effective and rational treatment using alkaline; sodium hydroxide (NaOH) solution as a modifying agent which also improves the interaction interface between wood fibre and the matrix.

The influence of alkaline modification on cellulose in the wood dust is a bulging and depolymerizing reaction which makes the wood dust crystalline structure of the cellulose to relax. In fact, concentration of the alkaline solution treatment on wood fibre will affect the form of bulging of complex molecular mass substances of wood fibre. This reaction resulted to the transformation into polymorphous lattice form (cellulose II) as shown in figure 2.8. Investigations have indicated that Na^+ has a favourable diameter; has the ability to penetrate

and open smallest pores within lattice planes; increasing the aspect ratio of fibres. In view of this, caustic soda treatment makes better swelling of crystallites of cellulose. This chemical reaction resulted in the transformation of a new Na^- cellulose -1 lattice. These new lattices promote appropriate gaps within cellulose molecules being filled with water molecules. The hydroxyl groups of cellulose are transformed into $\text{O}^- \text{Na}^+$ groups enlarging the dimensions of molecules. The main reaction is between hydroxyl (OH^-) groups of cellulose and hydrogen of alkaline is as shown in equation 2.1



Immediately after the reaction, dilute hydrochloric acid and distilled water were used to wash away the linked Na^- ions and change cellulose I to a new crystalline structure (cellulose II) and dried at room temperature for three days. Caustic soda (NaOH) causes total transformation of cellulose I to cellulose II while other alkalis produce only partial transformations as shown in Figure 2.8.

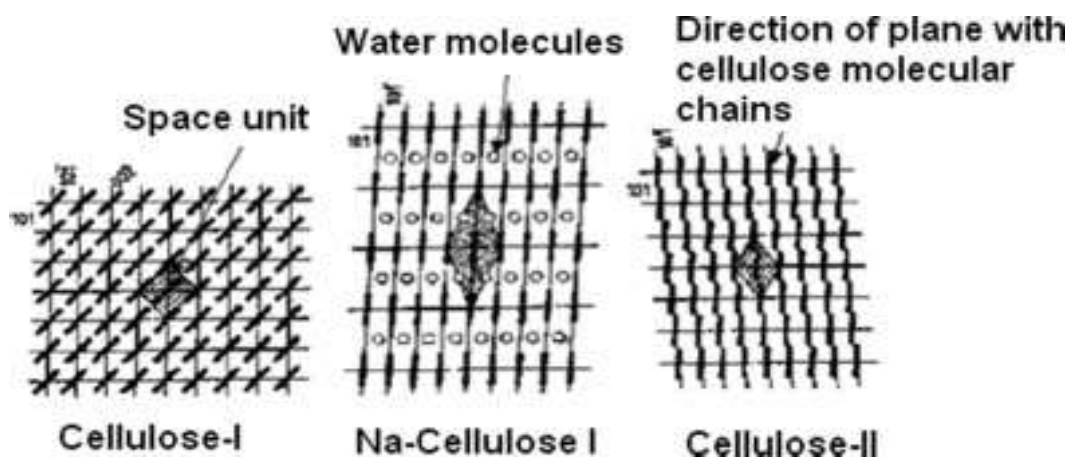


Figure 2.8: Crystal transformation of cellulose I to cellulose II. (Source: Weyenberg *et al*;2006).

The caustic soda solution affect both the cellulosic and non-cellulosic components of the plant fibres (hemi-cellulose, lignin, pectin and extractives). As a result of caustic soda modification, the change in value due to the treatment is in the breaking of hydrogen bonding in the lignocellulosic bundles, therefore exposing more fibre surfaces for contact with polymers. These modification remove some quantity of non-cellulosic materials like lignin, pectin, wax, oils and extractives encapsulating the exposed surfaces of the fibres cell walls, dismembering cellulose and exposing the fibrillated crystallites (Li, M. F., Fan, Y. M., Sun, R. C, and Xu, F, 2010). This shows that mixing of caustic soda with wood fibre results in ionization of hydrophilic wood to alkoxide (Agrawal, Y. C., McCave, I. N, and Riley, J.B, 2000). Therefore, caustic soda treatment affect cellulosic fibrillation. John *et al*, (2008) reported that alkaline treatment led to fibre fibrillation i.e. breaking down of fibre bundles into smaller fibres or the level of breaking down of fibre bundles and the removal of lignin and hemicellulose substances, reducing fibre diameter (Cordeiro, N., Gouveia, C, and Jacob, J. M, 2011) and improving aspect ratio of the fibres for effective adhesion with the matrix. The treatment result in larger surface areas to react with polymers. These also improve the dispersion, wetting and mechanical interlocking of the components in composite. This results in aspect ratio (length/width) increasing and effective area of contact with the matrix. As a consequence of this, the facial quality of the wood fibre is improved hence the mechanical interlocking of the components are also improved. From several scientific (modified) analyses of wood fibre/polymer matrix reinforcement investigated, the conclusion was that alkaline modification of fibres give appropriate adhesion than their untreated fibres. The aforementioned method has its merits.

2.7 Effects of Process Parameters on the Functional Properties of WPCs

It is pertinent that WPCs be reviewed from the perspective of process parameters using the available literature in order to understand how they influence both processing and properties of WPCs. Moreover, it is expected that this will assist in making appropriate choice of processing parameters and techniques during experimental design which had enabled optimisation of the functional properties of WPCs from previous studies since this study focuses on the exploration of the influence of wood dust particle size distribution and contents.

The influence of processing temperature on functional characteristics of WPCs was studied by (Siaotong, B.A.C., Tabil, L. G., Panigrahi, S.A, and Crerar, W.J, 2006) by determining the maximum values for fibre volume percentage by mass (0%, 12.5% and 25%) having extrusion barrel temperatures measured as (75-110-120-130-140 and 75-120-130-140-150°C) with extrusion screw speed (110 and 150 rpm) during compounding of flax wood fibre with thermoplastic resin of polyethylene (HDPE and LDPE) composites. Statistically, the maximum values that produced the highest tensile test (17.09 MPa for LDPE while 21.70 MPa for HDPE composites) when wood fibre volume ratio was 6.25%. The barrel temperatures were 75-100-120-136-146^{0c} and screw speed of 118rpm for LDPE composites. Furthermore, at fibre volume ratio of 5% reinforced HDPE with barrel temperatures; 75-120-128-140-148^{0c} and screw speed of 128rpm for the composites were attained. The temperature optimized was near to the appropriate level (75-120-130-140-150^{0c}). This is in view of the fact that lower temperatures resulted in improper flow behavior of resins which led to poor distribution of fibres in composites concluding in lower mechanical properties. The maximum value of screw speed was near the lower level (110rpm). The reason for this was at

high screw speed, fibre resistance occurred leading to poor fibre distribution in the matrix and high porosity resulting in lower tensile strength (Siaotong *et al*, 2006).

Studies carried out by (Khondker, O.A., Ishiaku, U. S., Nakai, A and Hamada, H, 2005) on unidirectional jute yarn reinforced polypropylene composites processing conditions, optical analysis of the micrographs suggested that temperature must be optimized to produce composites with high tensile properties. The results of optical microscope reveal that composites compounded when the temperature was 160^{0c} while moulding pressure was under 2.0MPa had pp matrix penetrated into the fibre lumens. These parameters were considered appropriate for processing composites using lignocellulosic fibres as reinforcement. It is pertinent to note that most lignocellulosic fibres cannot withstand processing temperatures above 175^{0c} for longer time thereby limiting their ability for processing with some thermoplastic resins (Khondker *et al*, 2005).

The appropriate injection and pressure for flax reinforced HDPE bio-composites were also determined by (Li, X., Tabil, L. G., Panigrahi, S, and Crerar, W. J, 2009). According to the results, increase fibre content in composites promote better mechanical strength. Temperature for injection process less than 192^{0c} was recommended to give high quality composites .This is because higher temperatures (200 °C – 210°C) leads to integrity of fibres being compromised; leading to inferior functional properties. As a result of this, injection temperature should not be less than 160°C to ensure adequate melting of the polymer matrix. Comparatively, to have composites with appropriate mechanical properties, higher injection pressure is required (Li *et al*, 2009). The compression for optimum pressure for natural fire reinforced acrylic resin was at 60 bars. Any pressure higher than this will damage fibre

structure and reduce tensile properties of composites (Medina, L., Schledjewski, R and Schlard, A. K, 2009).

Also, the mechanical properties of fibre reinforced thermoplastic composites could be effected when melting-mixing time was reduced; below 10 minutes, resulting in inadequate mixture of fibre and PP matrix resulting in poor distribution of fibres in the matrix (Joseph, P.V., Joseph, K, and Thomas, S, 1999). Again, at 10 minutes, the melt flow index of the matrix attained its highest viscous flow and penetrate into the fibres (Joseph *et al*, 1999).

It could be seen in figure 2.9 that the optimum melting-mixing time was obtained at 10 minutes when mechanical properties were optimised. As mixing time was higher than 10 minutes, fibre integrity was compromised resulting in poor mechanical properties (Joseph *et al*, 1999).

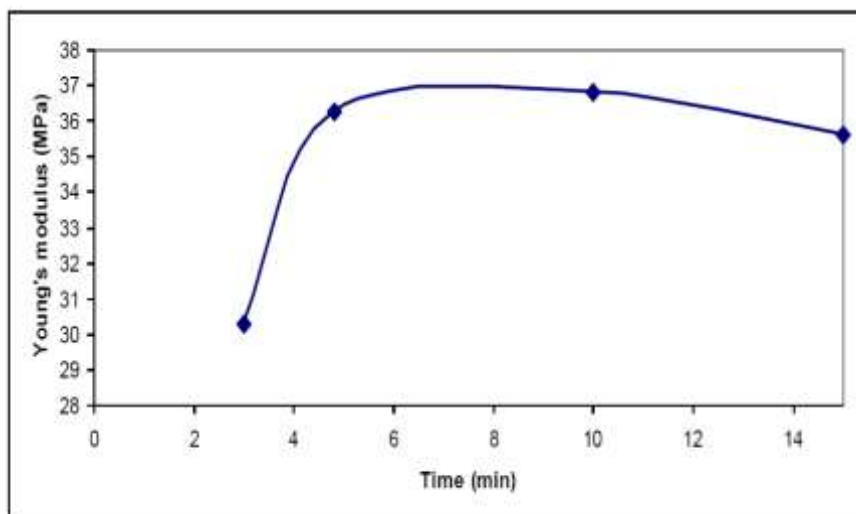


Figure 2.9: Effect of processing time on Young's modulus of composites. Source (Joseph *et al*, 1999).

Effects of various processing techniques on the functional properties of WPCs could also be viewed from the perspective of processing techniques: twin-screw compounding and mechanical or compression blending and their relationship with different melt flow indexes of HDPE. According to (Balasuriya, P. W., Ye, L, and Mai, Y, 2001), inter-penetration of melt flow of the matrixes: medium melt flow index (MMFI); mild viscous flow index and low melt flow index (LMFI); low viscous flow index could influence both mechanical and physical properties of WPCs on the basis that the strength and modulus of compression moulding of low melt flow index of HDPE are somewhat lower than those reinforced with medium melt flow index (MMFI) HDPE. It is a known fact that high viscosity liquids (LMFI) LDPE have low strength and modulus due to low molecular mass. On the other hand, medium melt flow index (MMFI) HDPE having mild viscosity have high strength and modulus due to mild molecular mass. However, mechanical properties of (LMFI) HDPE (low viscosity) slightly deviated from this trend probably because of incomplete fusion of LMF polymer in compression moulding. Table 2.3 shows the influence of melt flow index and compression moulding methods on functional properties of composites.

Table 2.3: Effect of melt flow index and compounding techniques on the tensile and flexural properties of composites. (Source: Balasuriya *et al*, 2001).

Property	HDPE grade			
	MMFI		LMFI	
	Injection moulded	Compression moulded	Injection moulded	Compression moulded
Flexural strength (MPa)	22.5 (0.28)	21.4 (0.37)	23.9 (0.80)	20.8 (0.19)
Flexural modulus (GPa)	0.916 (0.031)	0.903 (0.050)	1.102 (0.017)	0.856 (0.037)
Tensile strength (MPa)	20.89 (0.4)	20.86 (0.28)	22.2 (0.62)	18.85 (0.64)
Notched Izod impact strength (J/m)	163.8 (9.1)	165.75 (6.8)	224.6 (8.6)	153.56 (12.7)

Balasuriya *et al*, (2001) also explored the natural – chemical characteristics interrelationship between wood flakes and HDPE in terms of matrix melt flow behaviour and processing

techniques in forming composites. When optimizing mechanical properties of composites, flake distribution and wetting give acceptable parameters via twin-screw compounding and mechanical blending. The Scanning Electron Microscopy analyses of fractured composites show that composites compounded with medium melt flow index (MMFI) HDPE using twin-screw technique achieve better flake wetting and distribution (Plate I (a) and (b)) and as a result, mechanical properties of their composites are higher compared to the ones compressed or twin-screw compounded with low melt flow index (LMFI) HDPE; (Plate I (c) and (d)).

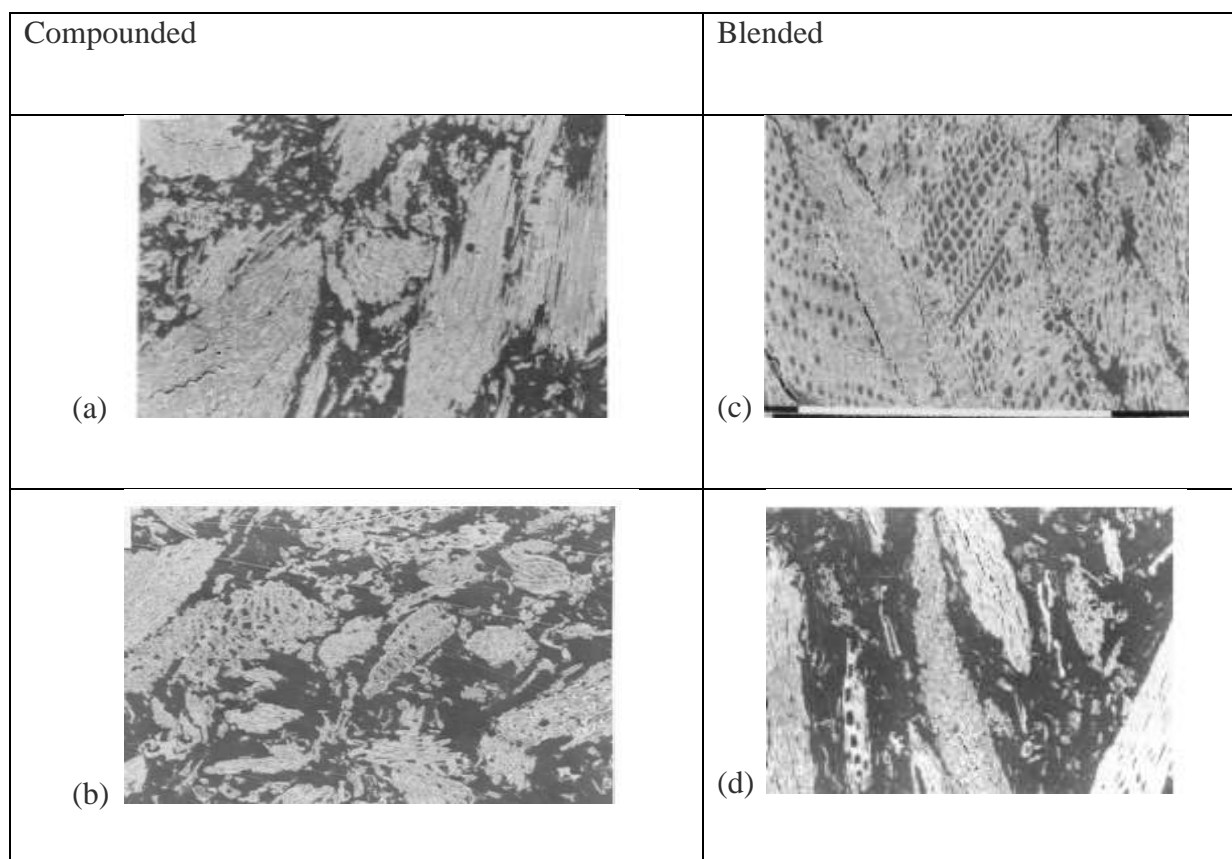


Plate I: SEM micro-graph of polished cross-section of (a) compounded MMFI (b) Compounded LMFI, (c) blended MMFI and (d) blended LMFI composite containing 50wt% wood fibres. (Source: Balasuriya *et al*, 2001).

Composites where the components (wood flakes and polymer) are 50:50 wt%, flake wetting depends on matrix flow behaviour and processing technique. It shows that medium melt flow index (MMFI) HDPE has the ability of wetting, dispersing and penetrating into lumens of wood flakes. These characteristics promote mechanical inter-locking between matrix and flakes resulting in better mechanical properties of composites especially for impact strength. These analyses show that low viscosity PE has the ability to wet wood flakes. Finally, compounded moulding technique was used in producing WPCs for this study because it is expected to impart uniformity of wood dust distribution within the polymer matrix.

Generally, the properties of fibre-reinforced polymer composites depend on the characteristics of the components and their adhesive interaction. Therefore, in flexural properties, fibre dispersion, proportional mixing and wetting of components in composite need to be considered. The reason for this is that in flexural tests, the concave side of specimen is compressed while the convex side is stretched (Doan, H. B., Brodowsky, H, and Mader, E, 2007). Therefore, the extent of flake distribution and homogeneity of the mixture, flake wetting and matrix penetration into flake lumens are important parameters when analyzing flexural properties of composites.

Obviously, increase in flake loading increases modulus which varies with mode of preparation and polymer flow pattern. As a result, composites based on (MMFI) HDPE matrix have higher moduli than those based on (LMFI) HDPE matrix. Comparing the

modulus of a composite, the tensile strength of composites are based on the degree of interfacial interaction between the matrix and the flakes (Doan *et al*, 2007).

Generally, increasing wood flake content, decrease tensile strength of composites. This is a general phenomenon because wood flakes are hydrophilic (absorb water moisture) in nature while polymers are hydrophobic (expel moisture) in nature. Due to these facts, there is poor interfacial interaction between flakes and polymers. The increase in tensile strength of composites is as a result of chemically blended interfacial adhesion between wood flakes and polymers giving rise to improved wetting and dispersion of flakes in the matrix and penetration of PE into lumens of wood fibres.

Analytical examination of fractured tensile test specimen of MMFI based WPC in Plate 2.2 reveals that the matrix (PE) penetrate into the lumens of wood flakes. It is suggested that ductile tearing of wood fibres encapsulated in the matrix is associated with PE penetration into lumens of the fibres showing mechanical interlocking between the components. This action is an indication of better tensile and flexural properties for composites compounded with (MMFI) HDPE that uniformly wet, disperse and penetrate into the lumens of fibres.

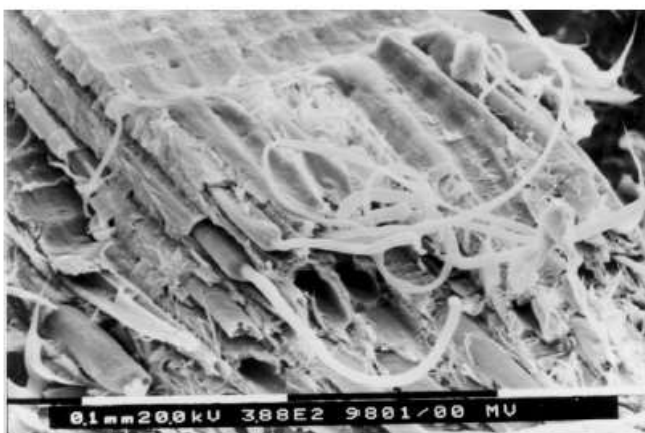


Plate II: SEM micrograph showing PE penetrating into wood lumens in compounded MMFI composite containing 40wt % wood fibres. (Source: Balasuriya *et al*, 2001).

2.8 Effects of Material Parameters on the Functional Properties of WPCs

In this section, attention is paid to the roles of materials parameters such as wood species, wood dust particle size distribution and content. This is carried out with the intention of having a wholistic knowledge in respect of wood dust particle size distribution and content on the functional properties of WPCs. (Zaini, M. J., Fuad, M. Y., Ismail, Z., Mansor, M. S, and Mustafah, j, 1995) carry out studies in the effects of wood species; soft woods (ponderosa pine), (pinus ponderosa), loblolly pine (pinustaeda) and hard woods (oak), (quercusspp) and maple (acerspp); and wood fibre contents (20, 30, 40, 50 or 60 wt%) on the functional properties (tensile and flexural) of WPCs. Figure 2.10 establishes that the maximum tensile strength (MTS) of the WPCs decreased with increasing filler content (Myers, G. E., Chahyadi, I.S., Coberly, C.A, and Ermer, D.S, 1991) irrespective of the samples of wood fibres reinforced within the polymer matrix. Moreover, oak, maple and ponderosa pine acted likely, loblolly pine acted not quite good (Figure 2.10).

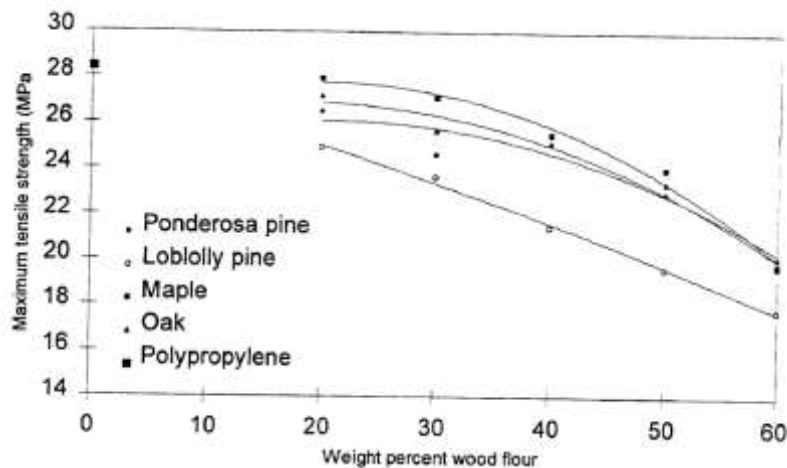


Figure 2.10: Effect of wood fibre content on maximum tensile strength of composites.

(Source: Zaini *et al*; 1995)

Meanwhile, it was noticed that the more the filler content, the stronger the adhesive modulus of elasticity (MOE) but percentage elongation decreased. Comparatively, tensile strength and modulus of elasticity properties of hardwoods are higher but show lower percentage elongation values than softwoods.

Bouafif, H., Koubaa, A., Preer, P, and Cloutier, A, 2009 studied the influence of particle size on mechanical properties of WPCs. They discovered that adding particle size to WPC production will improve modulus of elasticity and strength for both tensile and flexural tests as depicted in Figures 2.11 a-d. This report is inconformity with past results on wood-plastic composites (Dikobe, D.G, and Luyt, A.S, 2009). Moreover, steady increase with particle size increases flexural modulus of elasticity at higher filler content as shown in Figure 2.11c. For instance, it increases from 2.1GPa within particle size distribution from 100 mesh to 48 mesh (150-300 μm) to 2.7GPa when average particle size distribution of 24 mesh (710 μm) and 45 wt% fibre content. Flexural strength development as depicted in Figure 2.11d also demonstrated that as particle size increases the effect is realized when fibre content increases

from 65 mesh to 24 mesh at 45wt % when average particle size is about 24%. However, reinforcing HDPE matrix with wood particles will progressively increase tensile strength as shown in Figure 2.11b independent of filler content.

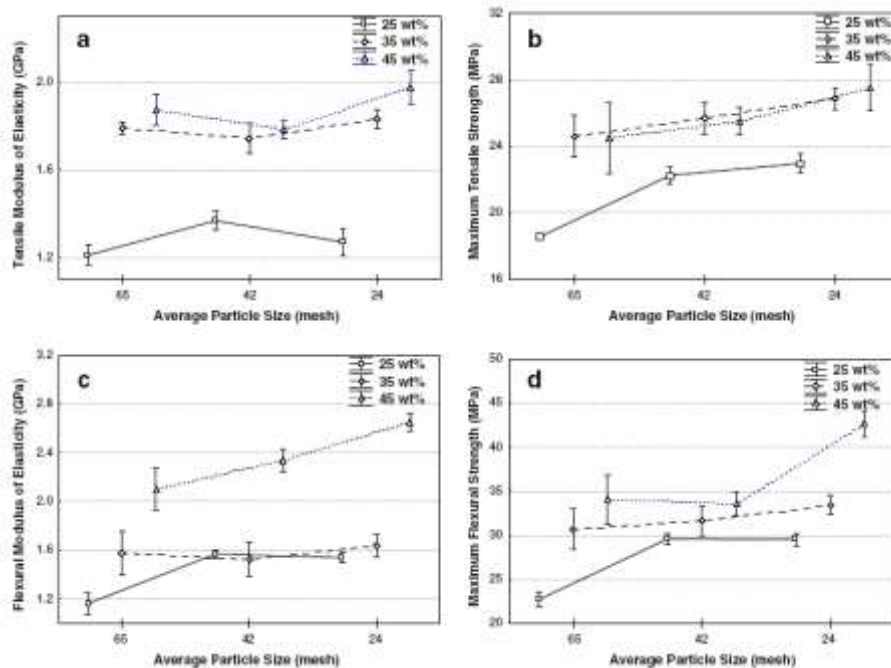


Figure 2.11: Effect of (a) tensile modulus of elasticity (b) maximum tensile strength (c) flexural modulus of elasticity (d) maximum flexural strength. (Source: Bouafif *et al*; 2009).

Increasing average particle size from 65mesh (230 μm) to 24 mesh (710 μm), improves tensile strength Zaini *et al*; (1996) recommended to increase maximum tensile strength, large particle size distribution of 250 mesh to 63 mesh oil palm should be used in reinforcing isotactic polypropylene .Also, (Stark, N. M, and Rowlands, R. E, 2003), reported the use of higher aspect ratio wood fibres instead of large size fibres to improve functional properties of WPCs. On the other hand, (Migneault, S., Koubaa, A., Erchiqui, F., Chaala, A., Englund, K, and Krause, C, 2008) indicated that as fibre length increased and the diameter unchanged, the strength and elasticity of HDPE reinforced composites are improved. The stress required to

fracture composites and the energy required to stretch composite materials to break respectively are shown in Figures 2.12 (a) and(b).

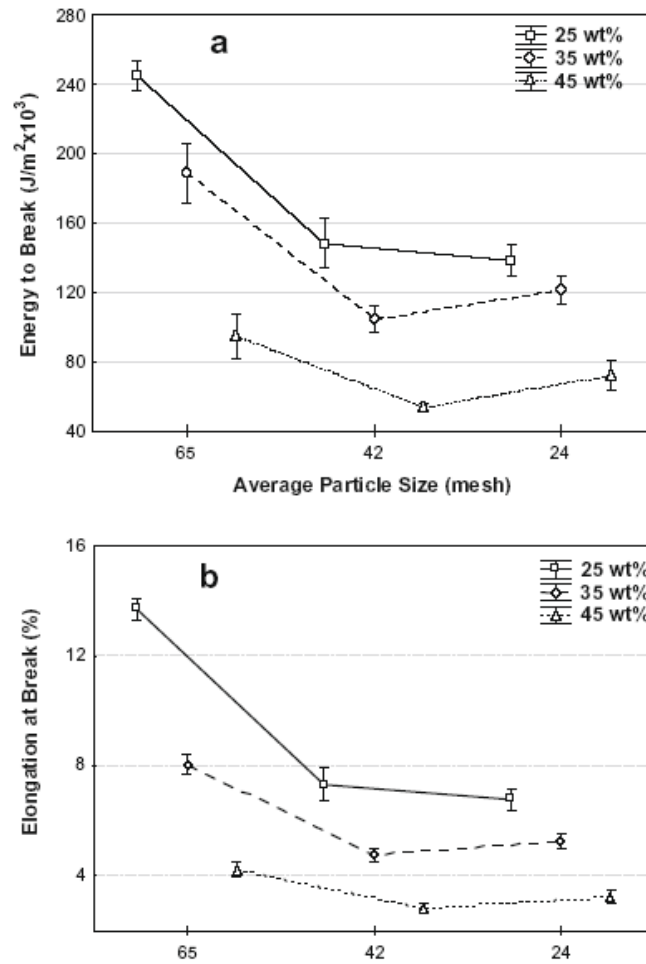


Figure 2.12: Effect of particle size on (a) energy to break and (b) elongation at break. (Source: Bouafif *et al*, 2009).

Energy to break and Elongation at break decrease as particle size increases. The effect is appreciated as particle size increases. Moreover, as tensile strength increase with increase particle size, the rupture energy decreases as shown in Figure 2.12a. This may be as a result of decrease in energy required to stretch and break. These have been general trend been reported with inorganic fillers as well (Dubnikova, I. L., Berezina, S.M, and Antonov, A. V, 2004). In

conclusion, reinforcing HDPE matrices with larger particle sizes improve tensile strength properties. However, this development comes with a consideration of energy to break. Though, depending on product end use, optimization for composites should be blended either for stiffness or stress to fracture based on filler particle size and content.

According to (Tajvidi, M., Najafi, S. K, and Moteei, N, 2006), water uptake of WPCs based particle size is not important. On the other hand, interfacial adhesion between fibre size and content is important. As a result, water uptake is a function of fibre content and not fibre length. According to (Steckel, V., Clemons, C.M, and Thoemen, H, 2007), the larger the particle size at low content, the higher the water absorbed as shown in Figure 2.13.

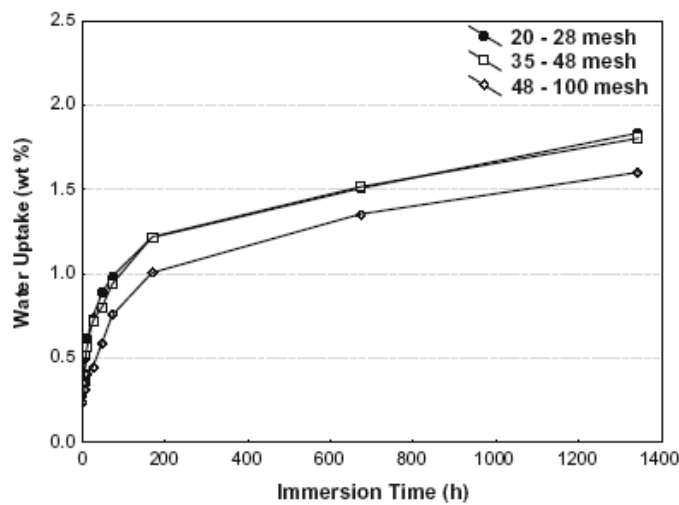


Figure 2.13: Effect of immersion time of composites on water uptake. Source: (Bouafif *et al*,2009).

Steckel *et al*; (2007) elucidated the findings as follows:

1. The larger particle size, the higher the hydroxyl groups attraction to moisture; and

2. Incompatibility shown in composites of wood particle and polymer matrix will exhibit presence of voids and porosity at interfacial contacts between the components. The presence of voids accommodate water.

Generally, at higher fibre content, effect of fibre size may not be noticeable compared to that at lower content. However, during processing, fibre size changes. It was noticed that higher aspect ratio particles were subjected to crushing effect due to shear stresses while blending the components. Obviously, particle size squeezes to smallest aspect ratio particle length.

Apparently, high wood dust content yield composites with high rigidity and strength. Variation in wood fibre content also influences mechanical properties of composites. Tensile strength (TS) and bending strength (BS) of composites were measured, by Doan *et al*; (2007) relative to jute fibre contents as shown in Figure 2.14. It was observed that adhesive bond and flexural bond of the composition improved until they attained 45% jute content. Thereafter, the strengths of the composites decreased. Also recorded were related observations for modular functional properties of composites as shown in Figure 2.15.

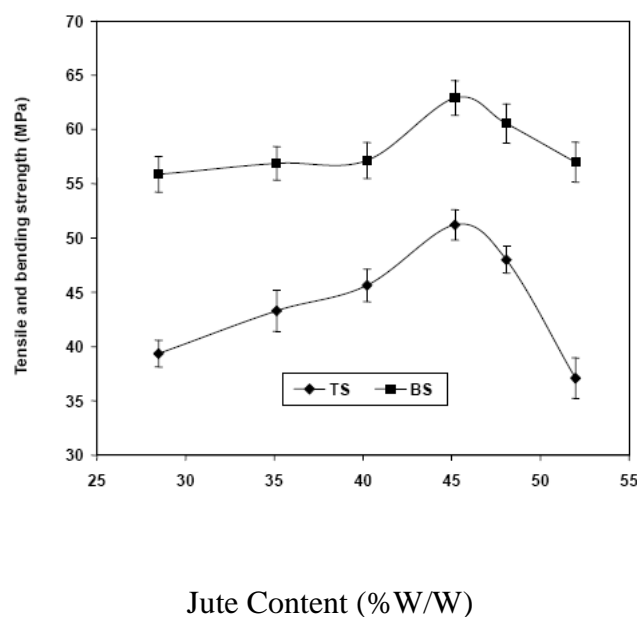


Figure 2.14: Effect of jute fibre content on tensile and bending (flexural) strength of composites. (Source: Doan *et al*; 2007).

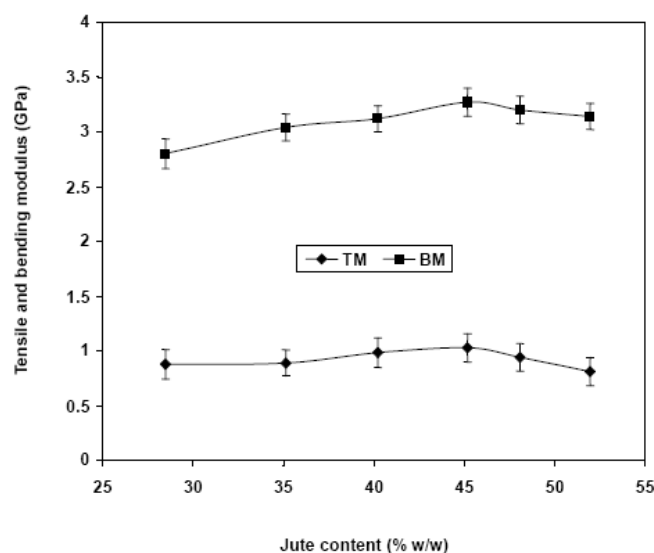


Figure 2.15: Effect of jute fibre content on the tensile and flexural modulus of composites. (Source: Doan *et al.*, 2007).

As reported by Doan *et al*; (2007), the highest worths of functional properties exhibited by 45% fibre wood particle contents may be suggested as a result of equilibrium distribution and mixed of fibres within the matrix. At 45wt% fibre content, it is envisaged that fibres attained optimum level of orientation and mixed equilibrium within the matrix. Therefore, stress will be evenly distributed when it is applied among the fibres. In view of these, tensile and flexural properties of the composites achieve maximum values. However, at low fibre content, poor fibre population causes interruption of stress movement within the fibres. Due to this defect, accumulation of strain occur at different locations in composites and strains are also found highly localised in the matrix (Doan *et al*; 2007). This contributes to poor tensile

and flexural properties of composites at low fibre contents. When jute particle contents increase, fibres get agglomerated within the matrix promoting particle-particle contacts resulting in non-uniform stress transfer capacity. Also too many fibre ends up promoting micro-crack formations at the interface. As a result, fibre pull-outs occur within the matrix again decreasing the composite properties (Doan, 2007). The influence of jute volume ratio on elongation at break (E_b) of the composites is shown in Figure 2.16.

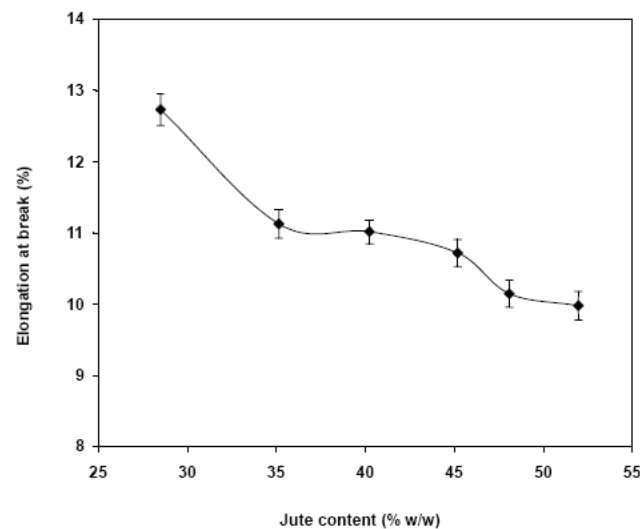


Figure 2.16: Effect of wood fibre content on elongation at break of composites.(Source: (Mohanty *et al.*; 2006).

It was found that E_b of the composites decreases as jute fibre content increasead. Generally, when modulus of composites increase, elongation at break decrease. (Mohanty, S and Nayak, S. K, 2006). It was observed that when fibre contents was more than 45%, the values of E_b further reduce as modulus of composites decrease. The reason may be that modular integrity of jute fibre is much lower than PP matrix and fibre volume fraction increases gradually after 45% jute content. Although, the formation of micro-cracks at higher fibre contents could be

as a result of degradation of Eb. Therefore, at the maximum fibre content, modulus may be responsible for the degradation of Eb. Apparently, after the optimum level, reduction of Eb is attributable to higher fibre content. The bulk density of adhesion of wood fibre interfacial interaction with polymeric materials depend on dry moisture-less wood particle sizes, particle contents and the group reinforcement is. As a reinforcement, wood fibre is made up of equivalent sizes and light in weight therefore, it is compressible and the fairly open physical structure of solid wood give rise to their densities in the range of .032- 0.72g/cm³ when dry (Simpson, W, and Ten Wolde, A, 1999). However, the high pressure found during plastic processing can collapse the hollow fibres that comprise the wood fibre or penetrate into them with high viscosity additives and matrices. In view of these, densities of polyolephines can be improved by reinforcing them with wood fibres.

In view of the fact that many reported cases have been made in the literature about mechanical characteristics of thermoplastic reinforced with wood dust, quite little research work have been carried out on particle size distribution and contents of wood dust on reinforcement of virgin high density polyethylene for mechanical, physical and micro-structural properties of composites. Against this background, the present research work is being undertaken to explore the potential of modified *Daniella oliveri* wood dust particle size distribution and contents as reinforcements in virgin high density polyethylene (vHDPE) composites. In order to develop an insightful knowledge of the functional properties of WPCS, it is important that the fundamental nature of the components of WPCs (the wood fillers and the polymer matrix) are properly understood.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The materials used for this research work were sourced locally. The wood dust of African Copaiba Balsam Tree (*Daniella-oliveri*) was collected from Maitumbi saw-mill, Minna, Niger State, Nigeria. The polymer-thermoplastic used in this study was virgin high density polyethylene (vHDPE) with melting temperature of 135°C and molecular weight of 28g produced in Nigeria by Nigeria National Petroleum Corporation, Eleme, Port-Harcourt. It has a density of 0.902 g/m³ and a melt flow index of 10g/10minute. It was bought from a retail shop in Minna, Niger State.

Sodium hydroxide (NaOH) Pellets was obtained from Echas Scientific Company, Kaduna, Nigeria. Derived optimum 4wt% sodium hydroxide solution and distilled water were prepared at the Kaduna Polytechnic Soil Science Laboratory for this research work. Dilute hydrochloric (HCL) acid and litmus paper also obtained from the Kaduna Polytechnic Soil Science Laboratory again for this research work.

3.2 Methods

3.2.1 Wood Dust Preparation

The wood dust was sun dried for one week to reduce the moisture content and measured at constant weight. It was then wrapped in a polythene bag. The wood dust was further heated in

water at about 90°C for 30 minutes to dissolve wood impurities such as extractives and other soluble substances on the surface of the wood dust. This procedure was undertaken in order to prepare clean surface area of the wood dust for effective adhesion between the polymer and dust. Water was completely drained from the wood dust upon attaining the required temperature and time. The wood dust was spread on a polythene sheet in a large room for three days to dry at ambient temperature so that the wood dust would retain the micro particles on the surface of the dusts which assists in wetting for effective adhesion. On the third day, the wood dust had dried and later shuffled. The wood particles were then screened and classified into various micro-meter size ranges using Hand Oscillating Multi-deck Screen Sieve.

As a result of sieve analysis, the following different particle size distributions were sieved from the hand-oscillating multi-deck screen: +75 -150 μm ; +150 - 210 μm ; +210 - 300 μm ; +300-425 μm ;+425-600 μm ;+600-710 μm ;+710-850 μm ;and+850-910 μm respectively. Particle size ranges of+600-710 μm ; +710-850 μm ; and +850-910 μm were disregarded because they were coarse, relatively lower in quantity and contained overlapping particle sizes at larger micro-meters which may be difficult to characterise based on specific particle sizes. Although, +75 - 150 μm particle size range was powdery and lower in quantity, it was included because of the relative size of specimens to be blended. As a result, the following particle size distributions were chosen: +75-150 μm ; +150-210 μm ; +210-300 μm ; +300-425 μm ; and+425-600 μm .

3.2.2 Chemical Surface Modification of Wood Dust with Alkaline Solution

The raw dried *Daniella-oliveri* wood dust of +75-150 μm ; +150-210 μm ; +210-300 μm ; +300-425 μm ; and +425-600 μm particle size distributions weighing 6.72g each were poured in different 100 milliliter beakers. Four percent (4%) dilute sodium hydroxide (NaOH) solution was added to the wood dust in the beakers and stirred with glass rod for homogeneity and left for 150 minutes at ambient temperature. The percentage content of sodium hydroxide solution and time of treatment being the optimal concentration and time derived from a study carried out by (Ogunesan, unpublished, 2013). The sodium hydroxide solution was completely drained using finer sieves and were thoroughly rinsed with dilute hydrochloric acid (HCL) in order to wash off alkaline remaining on the surface of wood dusts. This action was to prevent further chemical reaction between the dusts and the alkaline in excess of time for the experiment. Then the wood dusts were also rinsed with distilled water three times to remove the remaining alkaline not completely washed with dilute hydrochloric acid to prevent post-reaction of alkaline on the wood dust and to make the pH of the surface neutral which was confirmed by a litmus paper. The rinsed wood dusts were dried at room temperature for another three days ready for blending into composites of vHDPE.

3.2.3 Mould Preparation

The mould used for preparing *Daniella-oliveri* wood dust reinforced in vHDPE matrix is a locally fabricated rectangular mild steel plate with external dimension of 120mm by 90mm by 15mm (Plate III). It is made up of two parts; top and bottom plates, strewed on the four

edges to bolt the two halves together. On the bottom side of the internal surface of the plate is an engraved shape of the specimen. The dimension of the engraved shape is 57.5 mm by 15.5mm by 5 mm. A hole is drilled at the centre on the upper half of the mould to allow molten composite to be compressed at varying pressures depending on the gradient of wood dust size distribution and contents. The mould is shown in plate III.



Plate III: The mild steel mould used for preparing composites of *Daniella-oliveri* wood dust in vHDPE.

3.2.4 Mixing of vHDPE Polymer and Wood Dust

For easy evacuation of compressed composites specimen from the mould, a thin transparent nylon polythene sheet was laid on the engraved surface of the mould before compressing the mixture (molten polymer and wood dust). The mould was clamped in a preheat stand at 70°C to prevent the molten mixture from sudden solidification that could create different cooling zones (core and the surface) in the composites which could deform the product after cooling. Compression moulding process machine, Carver Hydraulic Press Machine Manufactured by Loctite Corporation, Mexico and installed at Tommy Polymer Company, Kaduna Road,

Suleija, Niger State was used in producing the WPCs. The mixture of crystals of virgin high density polyethylene and modified (with 4% NaOH solution for 150 minutes) wood dust at 60/40 (60% vHDPE 40% wood dust) ratio were poured into the hopper of the compression moulding machine after thorough mixing to give a homogenous mixture. The ratio 60/40 was chosen to explore the wetting effectiveness of various particle size distribution on the functional properties of WPCs. This also enabled the functional properties of WPCs to be optimised when the wood dust particle size distribution was varied.

The mixture was channeled through heated barrel at different temperatures (100°C- 120°C- 160°C) to soften and melt the matrix. The temperature was optimised at 160°C to prevent the degradation of wood dust. Generally, wood dusts start degrading at 190-210°C. At 160°C, the mould was connected to the compressing machine through a nozzle. The molten mixture was compressed into the mould through the nozzle at 160°C when the pressure was at 3.05MPa within 5 seconds. After each compression, the mould was cooled by air blower for 5 minutes, unclamped and then removed from the clamp by loosening the bolted edges and scrapping the extruded parts of it to give the required shape as shown in Plate IV.



Plate IV: A typical wood polymer composite sample produced in this study.

Moreover, different percentages (15, 25, 35, 45, and 55wt%) of wood dust contents at the optimised particle size distribution of +300-425 μ m were also blended with vHDPE with a view to optimising the wood dust content for imparting desirable functional properties into the WPCs. The choice of compression moulding technique is premised on the fact that it is expected to impart more desirable mechanical properties into WPCs in comparison to extrusion and injection moulding processes (Tungjitpornkull, S, and Sombatsompop, N, 2009). Moreover, the compression moulding pressure is lower than shear stress in twin screw extrusion (Tungjitpornkull *et al*; 2009). As a consequence, thermal degradation of HDPE molecules was reduced and damage of wood dusts by using compression moulding. Therefore, WPCs produced through compression moulding in this study are expected to be reinforced with higher specific density resulting in less void, porosity and air traps within its structure (Tungjitpornkull *et al*; 2009).

3.3 Characterisation of Wood Polymer Composite Samples

Five different tests were carried out on the composites produced. The tests were mechanical (tensile and flexural), physical (moisture and density) and micro-structural (Scanning Electron Microscopy; SEM) analysis.

3.3.1 Determination of Tensile Strength

The adhesive bond of the composition of blending modified *Daniella oliveri* wood dusts and virgin HDPE were determined according to ASTM D638-03. The test was conducted using Instron Universal Testing Machine, model Batten-Feld Plus 350, Austria. The machine is

provided with measuring devices; 20 KN load cell, PC 2000 equipped with software using digital loading system, vertical movement micro-processor with cross head speed of 5mm/minute. The specimen measured as length 57.5 mm, width 15.5mm and thickness 5mm. The test was carried out at ambient temperature. Five composites of different particle size distribution and contents of *Daniella-oliveri* wood dust modified with 4% sodium hydroxide solution for a period of 150 minutes and encapsulated in vHDPE were tested for tensile properties. The specimens were placed simultaneously within the grip distance of 55mm of the testing machine having gauge length of 50 mm to be in line with the grids forming invisible straight line joining the two ends on the grid to the machine. Synchronized computerized measurement of load applied (F), extension (ΔL), stresses (σ) and cross-sectional area (A) were used in computing the tensile modulus of elasticity (MOE) of the composites. The mathematical relationships of the parameters are as follows:

$$\sigma = \frac{F}{A} \quad N/mm^2 \quad 3.1$$

$$\varepsilon = \frac{\Delta L}{L} \quad 3.2$$

$$MOE = \frac{\sigma}{\varepsilon} = stress/strain(N/mm^2) \quad 3.3$$

Where,

σ = tensile stress

ε = tensile strain (dimensionless)

F= force applied (load in N)

A= cross-sectional area (mm²)

ΔL = difference in length (mm)

L = original length (mm)

3.3.2 Determination of Flexural Strength

Flexural or bending test was also carried out for chemically modified *Daniella-oliveri* wood dust blended with vHDPE. The tests were performed to investigate the effects of flexural load carrying capacities of alkaline treated wood dusts in the composites of vHDPE when varying both wood dust particle sizes and contents. It was carried out at a standard laboratory condition according to ASTM-D790. The composite specimens were prepared for three-point bending test. Each test specimen of length 57.5 mm, width 15.5mm and thickness 5.0 mm were prepared. The span of the supports was 50 mm. The specimens were loaded at the centre of the span through a loading cell and the loading rate was 2mm/min and degree of deflection of 30mm using Instron Universal Testing Machine model Batten-Feld Plus 350, Austria. The relationships describing the flexural strength (FS) and the modulus of flexure are as follows:

$$\text{Flexural Strength}(FS), \sigma_s = \frac{3FL}{2bh^2} \quad 3.4$$

$$\text{Flexural Modulus}(FM), E = \frac{L^3m}{4bh^3} \quad 3.5$$

Where F = maximum applied load (N)

m = slope of the tangent

L = length of the support span

b = width of specimen (mm)

h = thickness of specimen (mm)

3.3.3 Determination of Moisture Absorption Capacity

The composite samples were prepared according to the ASTM D570-98 required test for moisture absorption. The samples were cleaned to remove dirt and weighed to know their actual weight prior to immersion in a static distilled water. Digital weighing balance of model HP 1108 A with 0.001 g accuracy was used to measure the weights. The dimension of the specimens (57.5x15.5x5) mm dried to constant weight w_1 before immersion was recorded in grams. Three samples each of the five different samples were completely embedded in water at temperature of the surrounding. The absorption of the specimens were observed for 24 hours. The specimens were completely immersed in water at the bottom side of a beaker. For each observation, samples were taken out of the water at an interval of six hours, wrapped in tissue paper to remove surface water, reweighed, w_2 , incremental dimension recorded and again re-immersed in the water. As a result, four observations for each sample was tested within 24 hours at six hours intervals and average values arrived at. Therefore, percentage water absorption was calculated using the formula:

$$\text{Water Absorption \%} = \frac{W_2 - W_1}{W_1} \times 100 \quad 3.6$$

Where

w_2 = specimen weight after soaking in water

w_1 = weight of sample before soaking in water

3.3.4 Determination of the Density of the Wood Polymer Composite Samples

The density of wood polymer composite samples were determined using Archimedes Principle; water displacement method. The mass of each specimen was measured using the digital weighing balance model HP 1108 A with 0.001 g accuracy before gently and separately placing in static distilled water. The specimens were immersed in the static distilled water contained in a calibrated glass beaker and the volume of water displaced was recorded for each sample independently. This process was carried out three times for each specimen and the average volume of water displaced recorded. The density of the specimen were calculated using the formula:

$$\rho = m/v \text{ (g/cm}^3\text{)} \quad 3.7$$

where

ρ = density

m = mass in gram

v = volume of water displaced in cm^3 .

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

Scanning Electron Microscopic (SEM) observation of the samples were carried out in order to investigate the influence of wood dust particle size distribution ranges and contents on the interfacial chemistry of WPCs. The test was carried out at Sheda Science and Technology Complex (Shestco), Abuja. It was done using a JSM-6301F Scanning Electron Microscope (JEOL, Tokyo, Japan) operating at 15KV accelerating voltage. Before SEM observation, specimens were sputtered with 10nm layer of gold. The specimens were placed on aluminium holder of the microscope equipped with double sided carbon adhesive electrical conduction tabs. During SEM operation, a spectral of electron scanned the fractured surface of samples obtained from tensile test and signals from the incident areas were processed and valued. The operating electrical power of 5-15KV was used. SEM analyses of the different specimen were observed at 100 and 500 magnification and conclusion drawn as such.

CHAPTER FOUR

4.0

RESULTS AND DISCUSSION

4.1 Effects of Wood Particle Size Distribution and Contents on the Density of Wood Polymer Composite

Figure 4.1 shows that the density of the wood polymer composite samples increased to the optimum (0.97g/cm^3) as the wood particle size distribution increased from $+75\text{-}150\mu\text{m}$ to $+300\text{-}425\mu\text{m}$ while subsequent increment in the wood particle size distribution resulted in reduction in the density of the samples. Figure 4.2 also shows that the density of the wood polymer composite samples increased to the optimum of 1.04 g/cm^3 as the wood contents increase to 35%. Thereafter, a reduction in density of WPCs was observed.

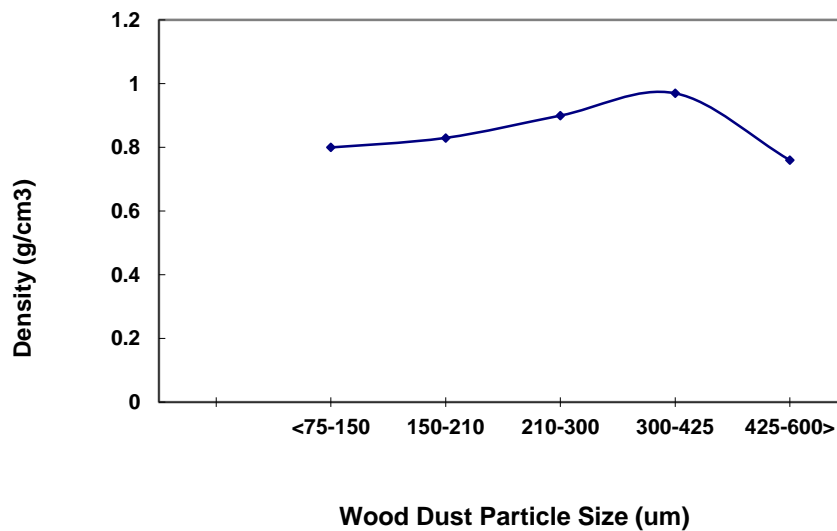


Figure 4.1: Relationship between the density of the wood polymer composites and varying wood particle size.

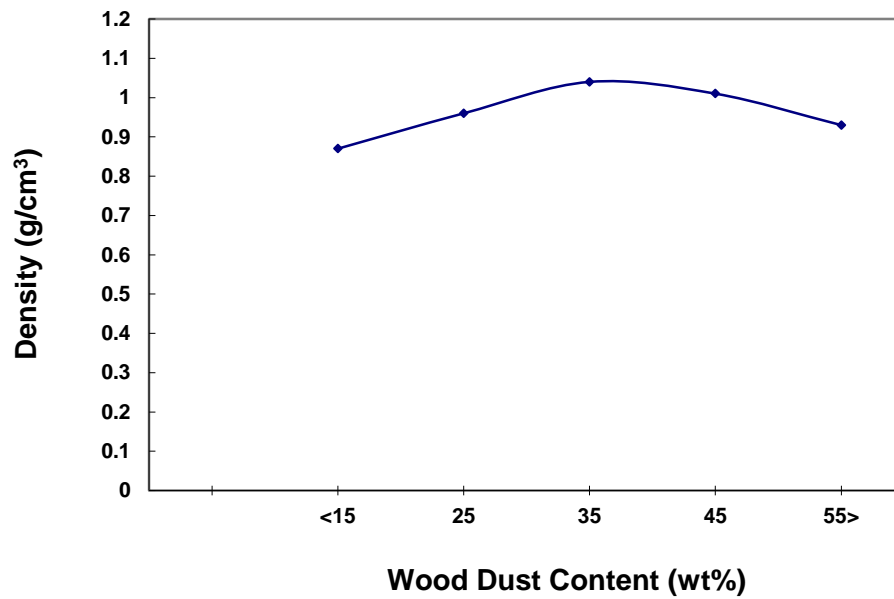


Figure 4.2: Relationship between the density of the wood polymer composites and varying wood content.

In considering the influence of particle size distribution on the density of WPCs, it is pertinent to note that the percentage of polymeric matrix to wood dust was put at 60/40% for all WPCs. As a result, the actual percentage of wood dust blended with the polymer matrix remains the same independent of quantity of particle size of wood dust. Therefore, wood flour surface area per volume encapsulated in the matrix decreases with increasing wood dust (particle size and content) (Leu, S. Y., Yang, T. H, and Lo, S. F, 2012). It is seen that the density of WPCs first increased due to the reinforcement with wood dust of particle sizes varying between +75 -150 μ m to + 300-425 μ m whereas wood dust of larger particle sizes imparted reduction in density to the WPCs. The reduction in density of WPCs when produced with wood dust of particle sizes larger than +300-425 μ m could possibly be attributed to incomplete wetting of the wood dust particles during compounding. Therefore, the wood dust

particles might have agglomerated such that they were not properly blended with the polymer matrix. This is presumed to have resulted in occurrence of porosity within the WPC microstructure, thereby leading to reduced density. The reported findings on the influence of wood particle size distribution on the density of WPC is similar to the outcome of the study carried out by Migneault *et al.*; (2008) in which it was established that the microstructure of WPCs reinforced with large size wood dust particles were bedevilled with pores due to uneven distribution of wood dust in polymer matrix.

In order to gain insight into how modified *Daniella oliveri* wood dust content influences the densification mechanism of WPCs as shown in figure 4.2, according to Doan *et al.*, (2007) in which functional characteristics of composites were studied with varying jute content was related to this study. Optimisation of density exhibited by 35wt% wood dust content may be explained in terms of fibre matrix dispersive orientation, homogeneity and penetration of the vHDPE in *Daniella oliveri* wood dust .At 35wt% wood dust content, it is envisaged that wood dust attained maximum level of distribution orientation, mixed homogeneously and wetting within the matrix. However, at low wood dust content, poor wood dust population causes non-homogeneous mixing with the vHDPE matrix. Hence, it is possible that wood dust contents are segregated at certain regions of the composite and densification become localised within the matrix (Doan *et al.*; 2007). This contributes to occurrence of porosity within the composites at low wood dust content. At high level of wood dust contents, it is envisaged that wood dust contents get agglomerated within the vHDPE matrix. Therefore, too many wood dust contents end up promoting porosity formation at the interface. As a result, density of the composite again decreases (Doan *et al.*; 2007).

4.2 Effects of Wood Dust Particle Size Distribution and Contents on the Water

Absorption Capacity of Wood Polymer Composite

Figures 4.3 and 4.4 show the outcomes of water absorption test of the investigated wood polymer composites as the wood dust particles and contents were varied respectively. The water absorption profile of the polymer reinforced with wood dust particle size distribution is seen to decrease as the wood particle size distribution increased up to +300-425 μm (1.5 % absorption) at the laboratory temperature as evident in Figure 4.3. Thereafter, the water absorption capacity of the composites is seen to begin to increase as the wood dust particle size distribution is increased above +300-425 μm . Figure 4.4 shows that the water absorption decreased from 0.6% to 0.3% when wood dust content varied from 15% to 35%. Above 35% wood dust content, the water absorption of the WPCs is seen to be increasing. These findings establish the fact that the water absorption capacity of the WPCs was minimised at the same parameters for which the density of the wood polymer composite was optimised as seen in Figure 4.2.

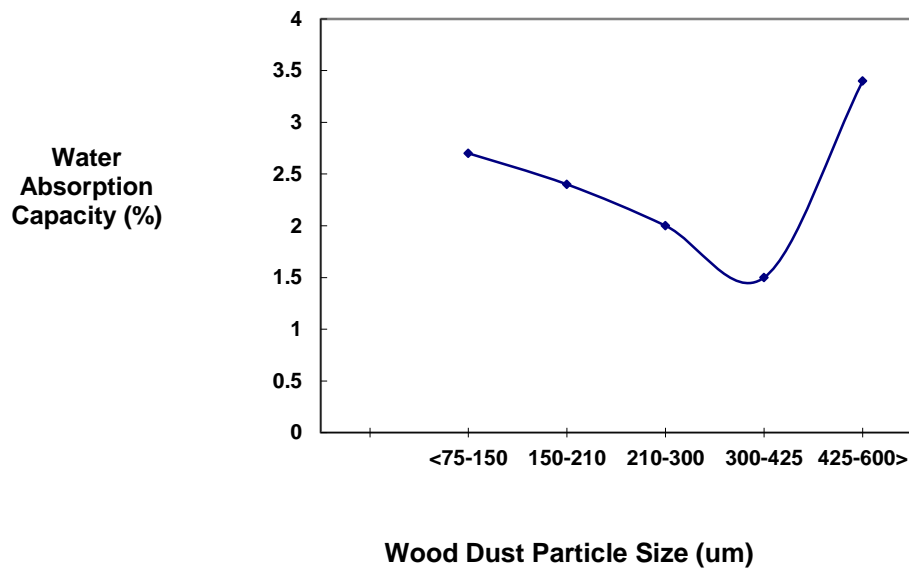


Figure 4.3: Relationship between the moisture absorption capacity of composites of varying wood particle size distribution in vHDPE.

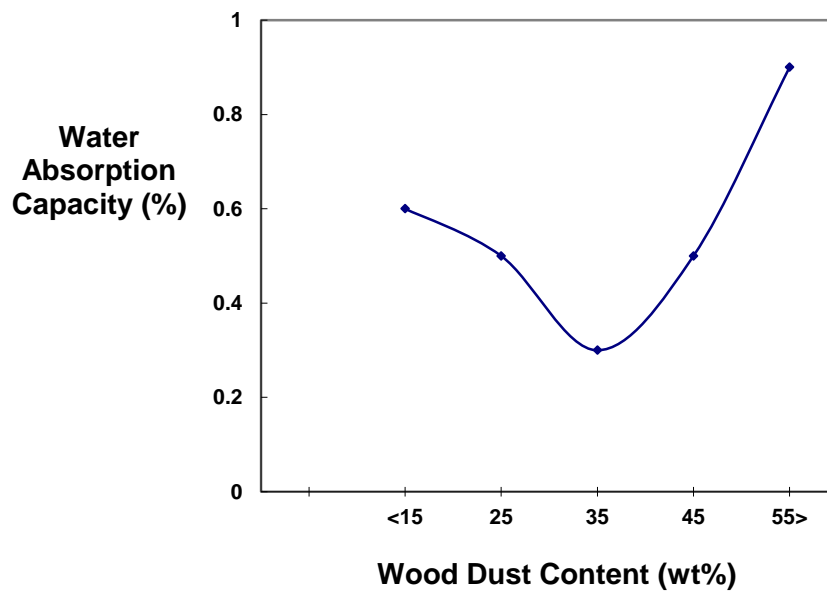


Figure 4.4: Relationship between the moisture absorption profile of composites of varying wood dust contents in vHDPE.

Findings reported for water absorption capacity of WPCs as wood dust particle size distribution are varied are quite in consonance with the outcome of the study carried out by Berger and Stark (1997) in which mechanical properties were found to increase for particle sizes ranging from +50-210 μm to +300-425 μm while the mechanical properties deteriorate for composites produced with fibres having average particle sizes greater than +300-425 μm . Also for particle sizes less than +300-425 μm , it was suggested that maximum level of orientation and mixed homogeneity were not attained hence, segregation of the particles was presumed. These resulted in voids and porosity in the composites leading to poor functional properties. Increment in mechanical properties for particle sizes ranging from +150-210 μm to +300-425 μm indicate an improvement in the wood dust-polymer matrix adhesion which lends credence to the reduced water absorption capacity reported for the same range of particle size distribution in this study. However, the deterioration of mechanical properties for WPCs containing wood dust of particle sizes larger than +300-425 μm suggests the degradation of the adhesion mechanism between the wood dust and the polymer matrix. Findings on the effect of particle size on the water absorption could be attributed to the fact that large sized particles lead to greater hydroxyl groups absorbing moisture. Since wood fibres and polymers are incompatible, poor adhesion between wood particles and polymers will result. Apparently, this non-interactive combination will voids around wood particles that are filled with water (Bouafif *et al*, 2009).

Similar to the explanation given for the influence of variation of wood fibre volume fraction on the densification of WPCs minimisation of the water absorption capacity of WPCs at

35wt% wood dust content may be explained by minimal occurrence of porosity within the composite structure due to homogeneous mixing of modified *Daniella oliveri* wood dust with the vHDPE matrix. Meanwhile, at lower wood dust content, it is envisaged that low wood dust population causes non-homogeneous mixing with the vHDPE matrix. Therefore, the segregated wood dust particles within certain localised regions of the composite are deemed to have introduced porosity into the microstructure of the WPCs which increased its water absorption capacity (Doan *et al*, 2007). At higher level of wood dust contents, it is envisaged that wood dust particles get agglomerated within the vHDPE matrix and consequently enhanced weak adhesion, leading to pull outs of wood dusts and porosity formations at the interface which resulted into undesirable increase in the water absorption capacity of WPCs (Doan *et al*, 2007).

4.3 Effects of Wood Dust Particle Size Distribution and Contents on the Tensile Strength of Wood Polymer composites

Figure 4.5 ascertained that the adhesive bond of the wood polymer composites produced by varying the particle size distribution of the wood dust is optimized at tensile strength of (15.4 MPa) within particle size +300-425 μ m. Moreover, the adhesive bond increased to 20.0 MPa when the polymer was reinforced with 35wt% wood dust content (Figure 4.6) in comparison to the optimal value obtained in Figure 4.5. This outcome suggests that there exist threshold values for both wood dust particle size distribution and contents at which the tensile strength is optimised. Further increment in both wood dust particle size distribution and contents is seen to have resulted in reduction of the tensile strength of the WPCs. Finally, optimal values of tensile strength of the WPCs obtained in this study are quite comparable to similar studies

in which NaOH treated wood dusts were reinforced with high density polyethylene polymer matrix using sisal wood dust (Kuruvila, J, and Sabu, T, 1993).

4.4 Effects of Wood Particle Size Distribution and Content on the Bending Strength of Wood Polymer Composite

Figures 4.7 and 4.8 show the bending strength of the wood polymer composite reinforced with varying wood dust particle sizes and contents respectively. Figure 4.7 confirms that the bending strength is optimised at 22.1 MPa when the WPCs were reinforced with +300-425 μm particle size distribution while it increased to 24.9 MPa when the WPC was further blended with 35wt% wood dust content (Figure 4.8).

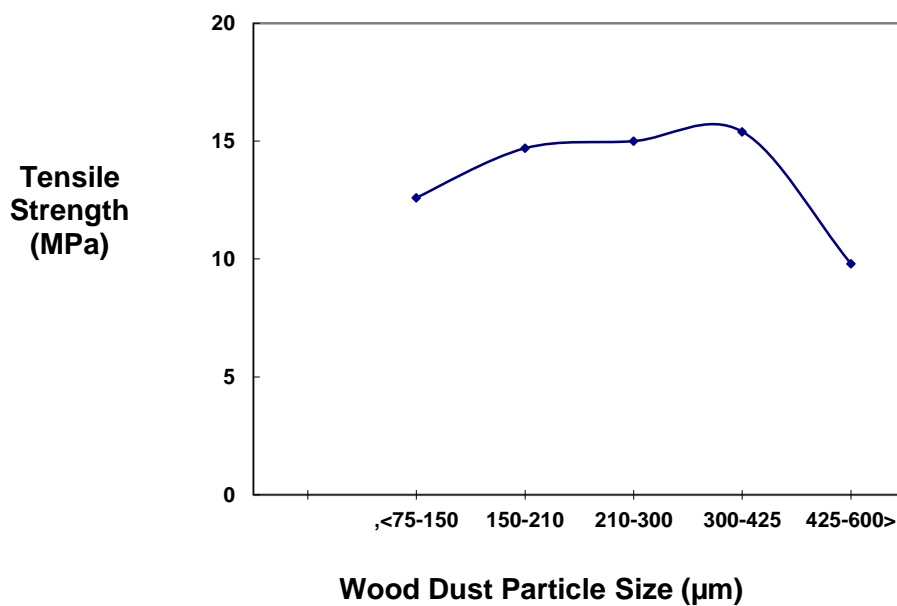


Figure 4.5: Relationship between the adhesive bond between the polymer and varying wood dust particle size distribution.

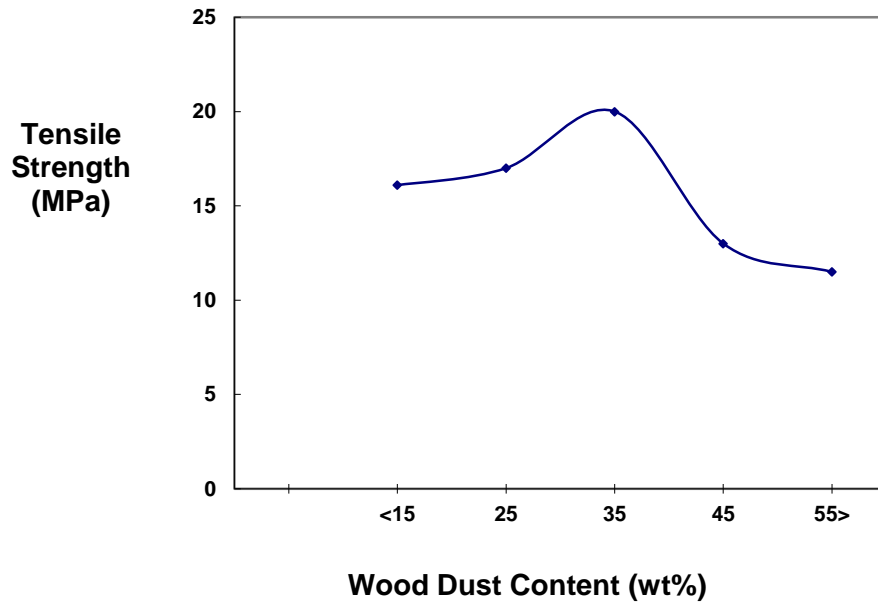


Figure 4.6: Relationship between the adhesive bond between the polymer and varying wood dust content.

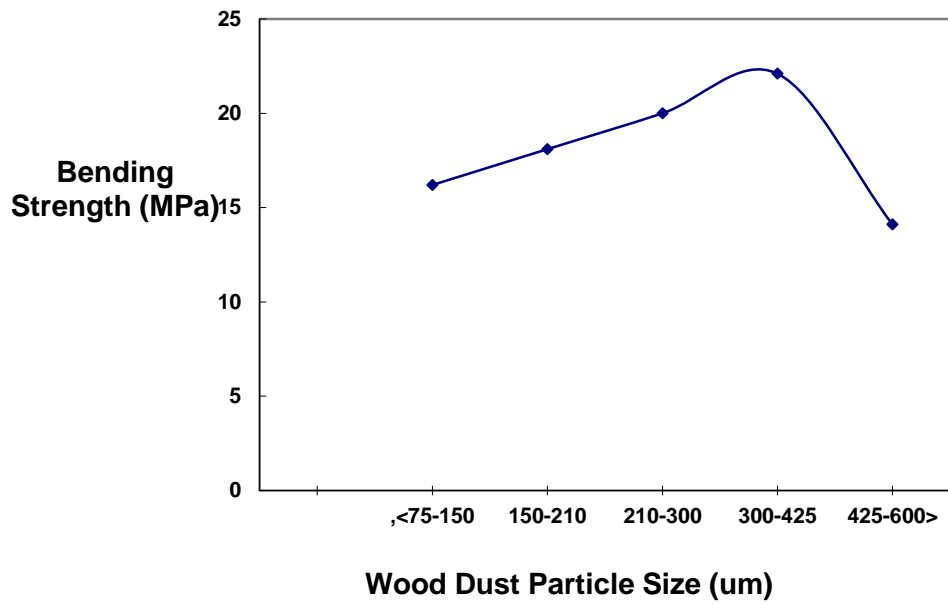


Figure 4.7: Relationship between the bending strength of the wood polymer composites and varying wood dust particle size.

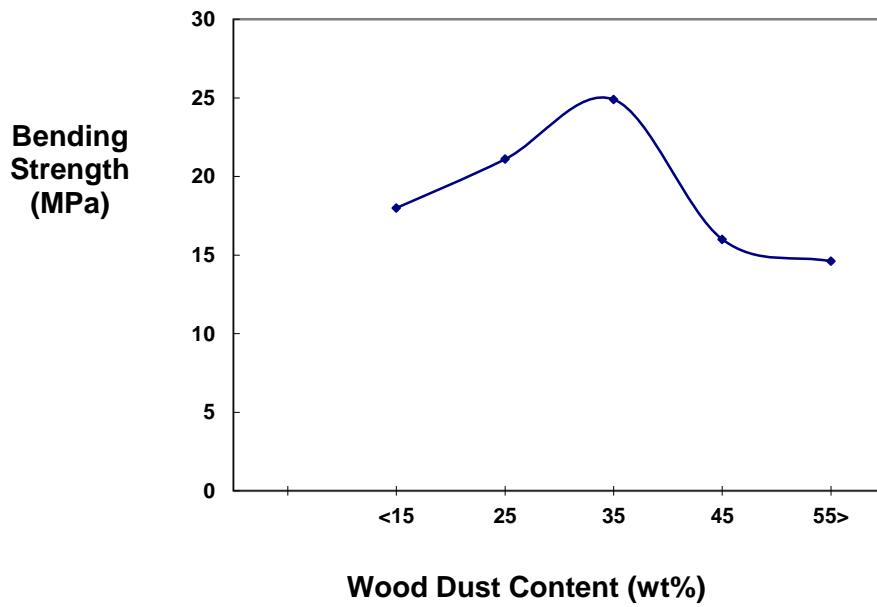
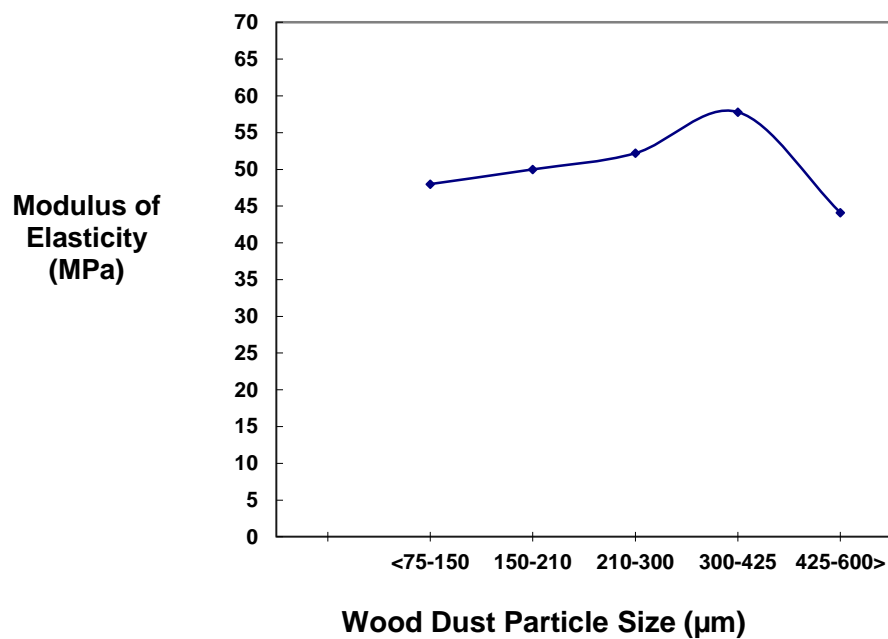


Figure 4.8: Relationship between the bending adhesion of the wood dust and the polymer when varying wood dust content.

4.5 Effects of Wood Dust Particle Size Distribution and Contents on the Tensile and Flexural moduli of Wood Polymer Composites

The effect of wood dust particle size on the tensile and flexural moduli of the wood polymer samples is represented in Figures 4.9a and b respectively. Highest tensile (55.8MPa) and flexural (230 MPa) moduli of the wood polymer composite samples were obtained when the polymer matrix was reinforced with wood dusts having +300-425 μm particle size distribution as shown in Figures 4.9a and b respectively.



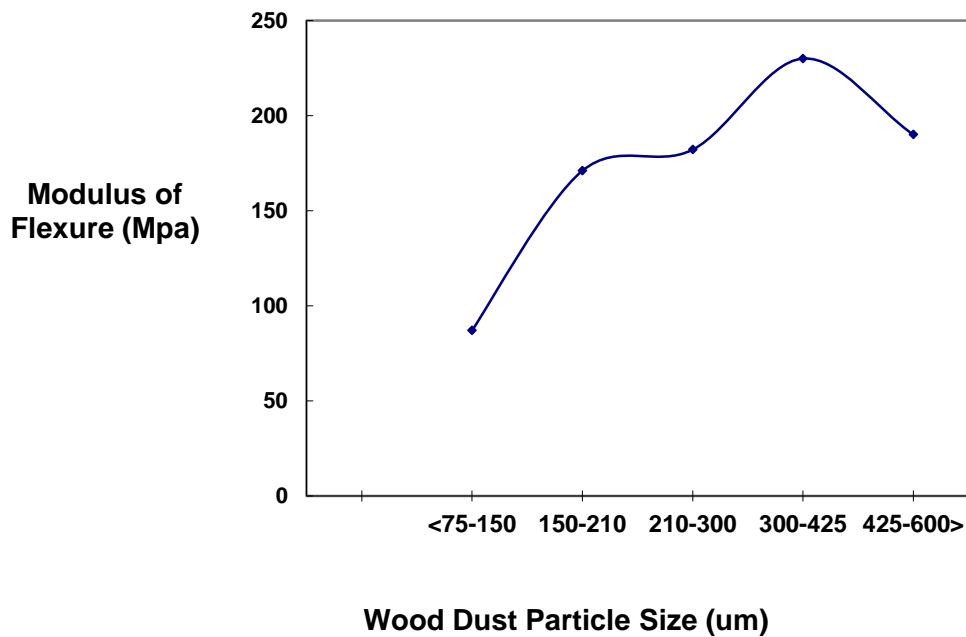


Figure 4.9 : Results of modulus of elasticity (a) tensile and (b) flexural at varying wood dust particle sizes.

Moreover, Figures 4.10a and b show that moduli of elasticity and flexure of wood polymer composites improve as wood dust content increase until it reaches 35wt%. Optimal values of adhesion and bending moduli of wood polymer composites were 68MPa and 388MPa respectively when WPCs were blended with 35wt% wood dust contents Figures 4.10a and b. It may be inferred from these findings that the values obtained from the analyses of the tensile and flexural moduli confirm the previous results which indicates that the best interfacial adhesion of the vHDPE polymer matrix and the reinforcing wood dust material can be obtained when the polymer matrix was reinforced with 35wt% wood dust content.

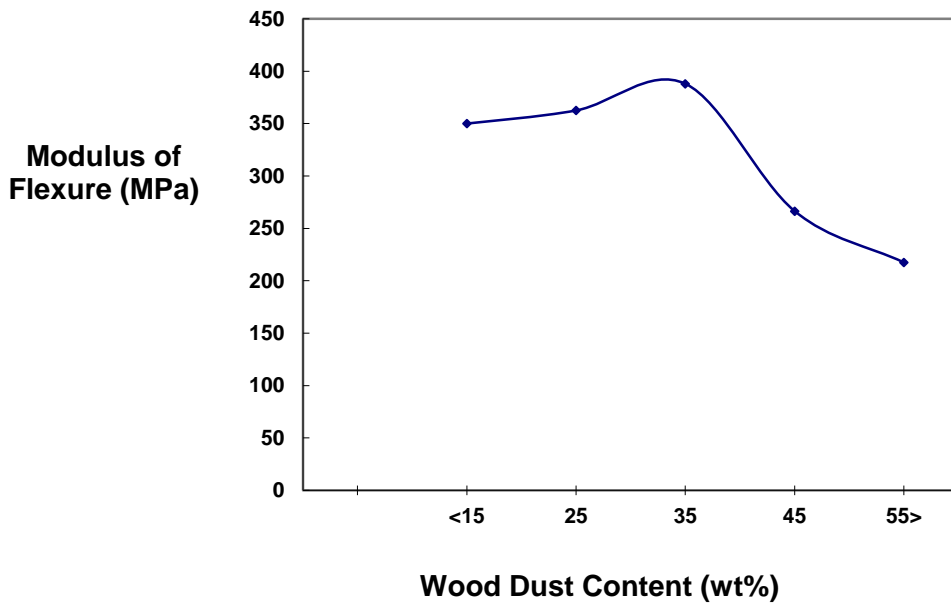
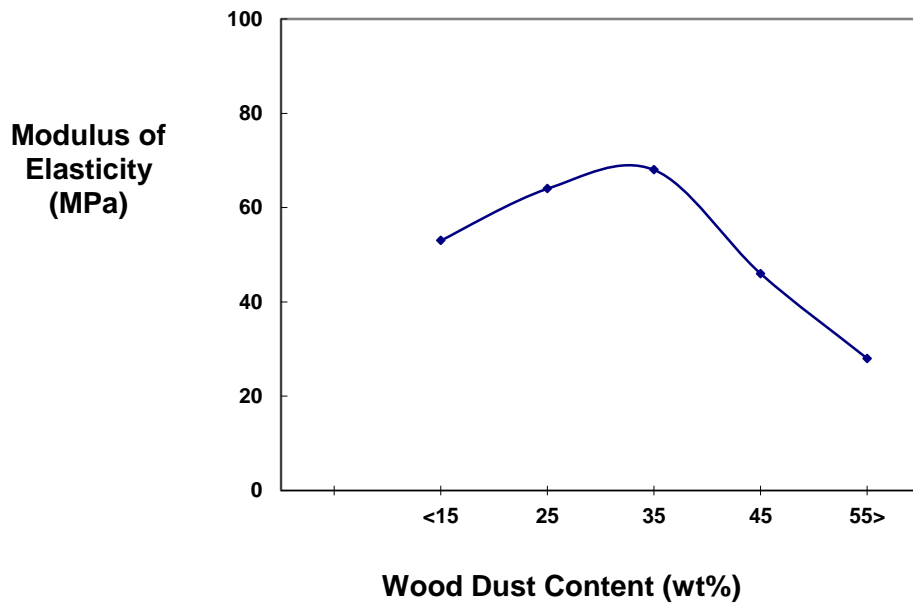
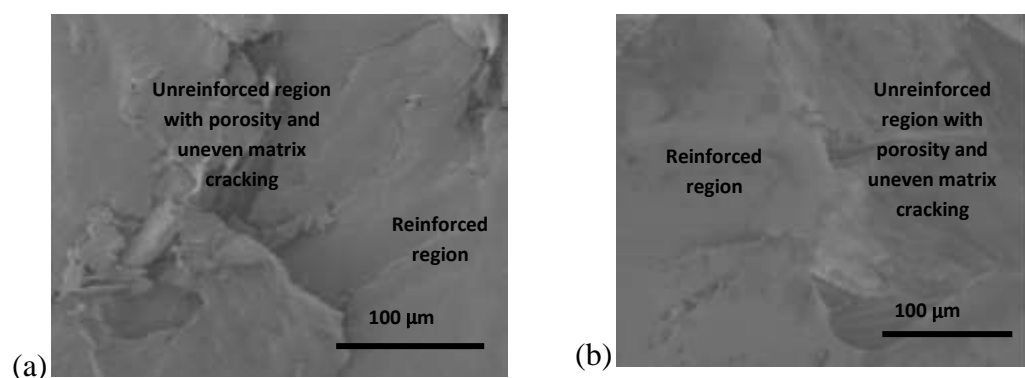


Figure 4.10: Results of modulus of elasticity (a) tensile and (b) flexural at varying wood dust contents.

4.6 Effects of Wood Dust Particle Size Distribution and Contents on the Microstructure of the Wood Polymer Composites

SEM fractographs obtained for wood polymer composite samples produced from *Daniella oliveri* wood dust of varying particle sizes and contents are presented in Plates V and VI respectively. Observation of the fractographs of the tensile test samples of the wood polymer composites were undertaken to further clarify the influence of interfacial bonding between wood fibre and polymeric materials on the mechanical test results. Comparative analysis of wood polymer composites containing modified *Daniella oliveri* wood dust of varying particle size distribution; +75-150 μm , +150-210 μm , +210-300 μm and +425-600 μm (Plates V a,b,c and e) suffered some degree of uneven particle size segregation and agglomeration leading to voids, porosity, fibre pull-outs and by extension, poor mechanical properties. However, the appropriate or optimum particle size distribution; +300-425 μm (Plate Vd) reinforced with the matrix revealed maximum level of fibre/matrix homogeneously dispersed orientation, wetting and mechanical inter-locking bonding between the particles and the matrix leading to better functional properties of the composite.



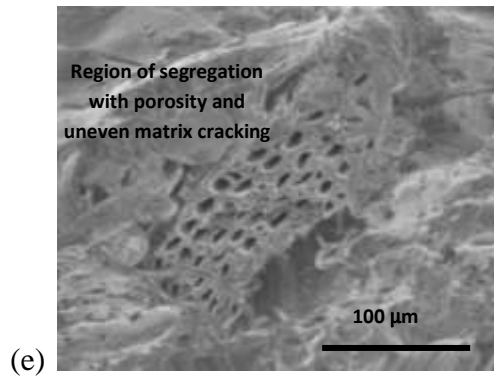
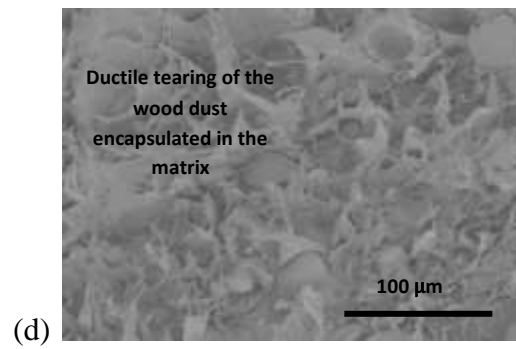
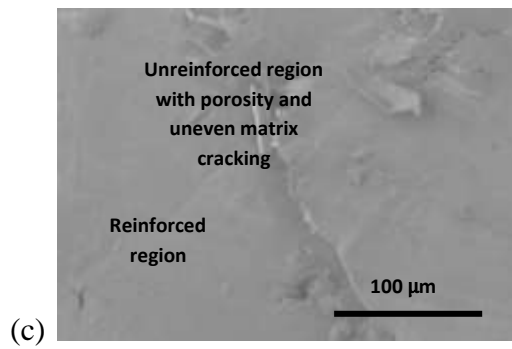
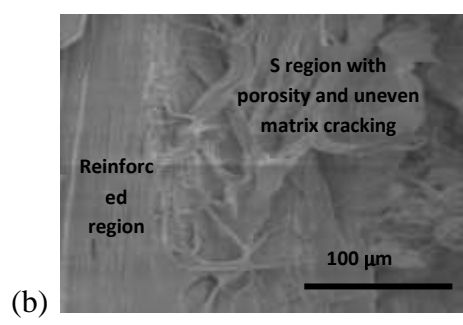
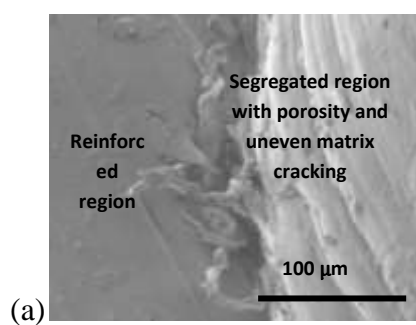


Plate V: SEM fractographs of wood polymer composites reinforced with wood dust of varying particle size distribution (a) +75-150 μm (b) +150-210 μm (c) +210-300 μm (d) +300-425μm (e) +425-600 μm.



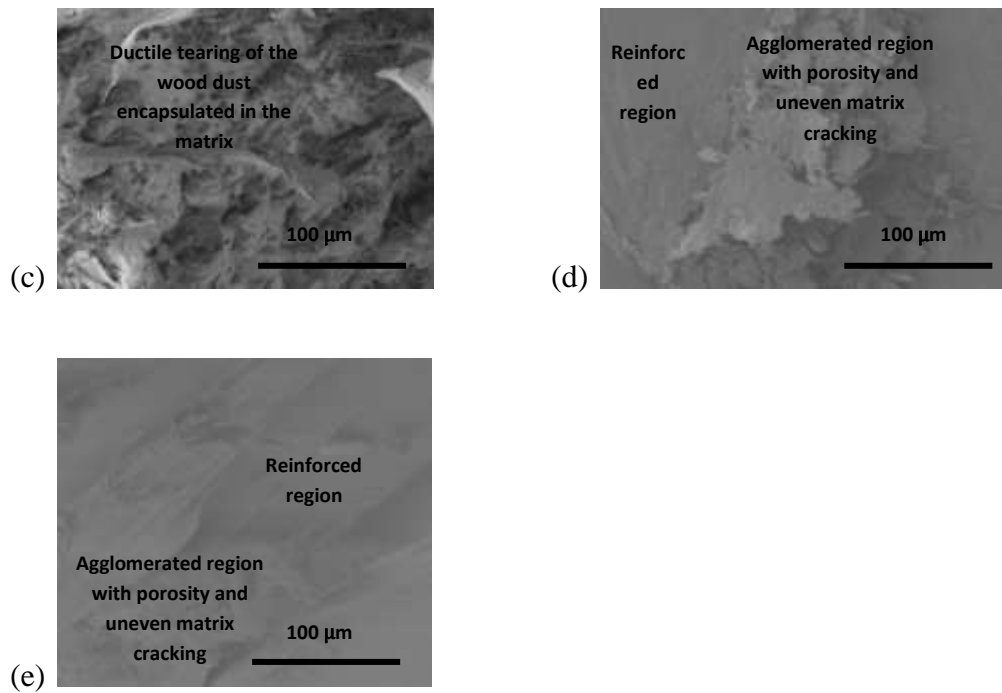


Plate VI: SEM fractographs of fractured tensile surfaces of wood polymer composites reinforced with modified *Daniella oliveri* wood dust of varying contents (a) 15 wt% (b) 25 wt% (c) 35 wt% (d) 45 wt% (e) 55 wt%.

Furthermore, a study of the SEM fractographs of wood polymer composites reinforced with varying contents of modified *Daniella oliveri* wood dust also revealed that greater degree of occurrence of uneven matrix cracking and void in composites reinforced with 15, 25, 45, and 55wt% modified *Daniella oliveri* wood dusts (Plates VI a, b, d, and e) relative to the composite sample reinforced with 35wt% wood dust (Plate VI c). The findings from the fractographs are quite in consonance with the result of the report for functional characteristics of the fibre polymer composition reinforced with varying wood dust particle size distribution and contents in which the WPC samples produced with +300μm-425μm wood dust particle sizes at 35wt% content had the most desirable physical and mechanical properties (Figures 4.2, 4.4, 4.6, 4.8, and 4.10). It is pertinent to note that the presence of voids and cracks in the

fractographs of WPC samples suggest occurrence of poor densification and the development of less than desirable mechanical properties during processing due to the blending of the polymer matrix with inappropriate choice of wood dust particle size and contents (Plates V a, b, c, and e; Plates VI a, b, d, and e).

The mechanical properties of the WPCs reached optimal values at +300-425 μm wood dust particle size distribution and 35wt% content. These could be attributed to uniform dispersion of the wood dust particles within the polymer matrix. Consequently, the fractured surface of the WPC obtained at the optimum parameters shows ductile tearing of the fibres encapsulated in the matrix (Plate V d, and VI c). Wood dust of particle size distribution less than +300-425 μm imparted less than optimum mechanical properties onto WPCs because they are unable to induce adequate dispersion and wetting of fibres within the vHDPE polymer (Plates V a, b, and c). As a result of these, excess matrix regions between fibres within the polymer matrix were created which reduces load impact between the matrix and the fibres. Due to these excess matrices, unreinforced regions made up of whole matrix were created within the composites. These resulted in the mechanical properties of those composites produced from wood dust of particle size distribution less than +300-425 μm to be less than the optimum (Figures 4.5, 4.7, and 4.9). Moreover, when the WPCs were produced through reinforcement with wood dust of particle size distribution larger than +300-425 μm , it is evident that the morphology of fractured surface of WPCs specimens is characterised by agglomerated structure (Plate V e). Figures 4.5, 4.7, and 4.9 lend credence to the claim that composites having larger particle size distribution than +300-425 μm degrade in functional properties. The explanation for the degradation of composites functional properties when reinforced with wood dust particle size distribution larger than +300-425 μm is also similar to the analyses for the reduction of WPCs' density and increase in moisture adsorption. Hence, the interfacial

adhesion between the wood dust particles and the matrix was weak when wood dust particles of larger sizes were used.

It is pertinent to note the similarity in the fractographs of WPCs samples reinforced with varying contents of wood dust (Plates VI a-e) upon comparison with the outcome of the findings on the functional characteristics of the composites (Figures 4.2, 4.4, 4.6, 4.8, and 4.10). For example, at low wood dust contents (15, 25 wt%), low wood dust particle population causes low load transfer capacity among the fibres. Therefore, segregation of wood dust particles occurred in localized areas within the matrix (Plates VI a, b). Due to accumulation of stress at localised regions of the composite microstructure, undesirable mechanical properties are imparted into the composites at low wood dust content (Doan *et al*; 2007). At higher level of wood dust contents, Plate VI d reveals that agglomeration of wood dust occurred within the matrix thereby resulting in non-uniform stress transfer capacity. Moreover, too many wood dust particles end up promoting micro-crack formations at the interface. These in turn result in reduction of strength and modulus of the composite (Doan *et al*, 2007). As the wood dust content increases within the polymer matrix, the wood dust surface area is expected to be wetted by the matrix increases. Consequently, increased surface area of the wood dust particles induces increased restriction to the molecular mobility of the matrix. This restriction of the wood dust particles on the matrix reduces effective wetting of the wood dust particles (Myers *et al*, 1992). Another reason that might have prevented effective wetting of the wood dust by the polymer matrix is the overlapping of wood dust particles as wood dust increases in content. Overlapped wood dust particles promote particle-particle contact and at the same time prevented matrix from wetting their surfaces. As the inter-particulate overlapping continues, voids were created in the composites where pull-out of wood dust from composites occurred during fracture (Plates VI d, e).

CHAPTER FIVE

5.0 CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The following conclusions can be drawn from this study:

- (1) Reinforcement of vHDPE with modified *Daniella-Oliveri* wood dust resulted in the production of WPCs with improved physical and mechanical properties.
- (2) The density, water absorption capacity and mechanical properties of the WPCs containing treated wood dust increased with increasing wood dust particle size distribution up to +300-425 μm but over +300-425 μm , the properties of the biodegradable composite decreased.
- (3) The density, water absorption capacity and mechanical properties of the WPCs containing treated wood dust improved when wood dust content attained 35wt% but above this value, the properties of the biodegradable composite decreased.
- (4) Optimal densities (0.97 and 1.04) g/cm^3 , water absorption capacities (1.5 and 0.3)%, tensile strengths (15.4 and 20.0) MPa, flexural strengths (22.1 and 24.9) MPa, modulus of elasticity (57.8 and 68) MPa and modulus of flexure (230 and 388) MPa respectively of the WPCs containing treated wood dust were obtained with wood dust parameters of +300-425 μm and 35wt% content.
- (5) The experimental results in the present work suggest that composites with multiple applications and good functional properties could be developed by exploring modified *Daniella oliveri* wood dust as a reinforcing agent for the vHDPE matrix for photo-frames, radio and television frames and other house hold applications.

5.2 Recommendations

(1) This work was carried out purely using wood dust from modified *Daniella oliveri* and vHDPE without using any coupling agents or additives and with the promising results obtained. It is thus clear that when the accurate quantity of additives and coupling agents will be introduced, a better improved results will be obtained 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 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 the same wood source.

(3) Due to time constraints simple engineering components such as photo frame, name frame could not be produced with the treatment parameters developed from this study. 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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