PRODUCTION AND CHARACTERIZATION OF RECYCLED LDPE/ PERIWINKLE *(TURRITELA COMMUNIS)* SHELL PARTICULATE COMPOSITE

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JANUARY, 2016

DECLARATION

I hereby declare that the work in this dissertation entitled "PRODUCTION AND CHARACTERISATION OFRECYCLED LDPE/PERIWINKLE SHELL PARTICULATE COMPOSITE" has been carried out by me in the Departmentof Metallurgical and Materials Engineering, under the supervision of Dr. E. T. Dauda and Dr. T. Ause. The information derived from literature has been duly acknowledged in the text and a list of references provided. No part of this dissertation has been presented for another degree or Diploma at this or any other Institution.

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Signature Date

CERTIFICATION

This dissertation entitled PRODUCTION AND CHARACTERISATION OF RECYCLED LDPE/PERIWINKLE SHELL PARTICULATE COMPOSITE by MeshackBosan BALA meets the regulations governing the award of the degree of Master of Science of the Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

In loving memory of my Grandfather (Pa KaribvuiBalaBosan).

ABSTRACT

The production and characterization of recycled LDPE/Periwinkle (*TurritellaCommunis*) shell particulate composite was carried out with a view to addressing part of environmental pollution problem. Periwinkle shells were conditioned with 5 wt% NaOH for a period of 2 hrs, after which the shells at three different particle sizes, 106µm, 75µm and 53µm were combined with recycled low density polyethylene in a two roll mill compounder at a temperature of 150ºC and time of 5 minutes to produce the composite. The samples were tested for, impact, tensile, hardness and flexural strengths, water absorption, the density and microstructures were also examined and then the soil degradability test was carried out for a period of two weeks at a depth of 10cm. The 75µm particle size exhibited the best impact strength at 25 wt% filler with a value of 1720 J/m. The best tensile strength was obtained at 106µm and 5 wt% filler with a value of 9.50 Mpa. There was a 3.3% increase in hardness for 75µm at 20 wt% and a 26.67% increase in hardness for the 106µm at 25 wt%, while a 3.3% increase in hardness at 15 wt% was obtained for the 53µm particle size representing the highest hardness for individual particle sizes considered. The flexural strength obtained for the three particle sizes presented an increase as filler content increased beyond 5 wt%, while the 75µm particle size presented the best flexural strength 25.51 Mpa at 10 wt% filler. There was an increase in water absorption at all particle sizes considered, with the 75µm particle size at 25 wt% filler with a rate of 3.77%, being the highest, while the least was at 15 wt% filler with no water absorption observed between 24 hrs to 120 hrs. Density decreased with filler increase and increased with particle size. There was no degradation for the period considered except at 15 wt% for the 53µm particle size. The results of the micro-structural analysis showed that the fractured surfaces are more of a ductile than of brittle nature, and there is a uniform distribution of filler in the matrix.

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ABBREVIATION, DEFINITIONS, GLOSSARY AND SYMBOLS

CMC – Ceramic matrix composite

CNSL - Cashew nut shell liquid

HDPE – High density polyethylene

LDPE – Low density polyethylene

LLDPE - Linear low density polyethylene

MDPE - Medium density polyethylene

MMC – Metal matrix composite

μm – micro-meter

nm– nanometre

PKS – Palm Kernel Shell

PMC – Polymer matrix composite

PP – Polypropylene

PWS - Periwinkle shell

PS – Polystyrene

PVC – Polyvinyl Chloride

RLDPE – Recycled low density polyethylene

SEM – Scanning Electron Microscopy

UV – Ultraviolent light

UHMWPE – Ultra high molecular weight polyethylene

Wks - Weeks

XLPE – Cross linked polyethylene

XRD – X-Ray Diffractometry

Symbols

% - Percentage

< - Less than

> - Greater than

CHAPTER ONE

1.0 INTRODUCTION

A composite material refers to a combination of two or more materials comprising of at least two phases bonded together to produce a material with improved properties. One of these phases is a continuous phase (matrix) while the other is a dispersed phase (reinforcement). The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed phase (Bledzki and Gassan, 1999).

Various composite materials may be formulated, targeting increase in mechanical properties such as strength, hardness, stiffness, while others may be tailored to suit other properties of interest such as thermal conductivity, toughness.

Many of our modern technology require materials with unusual combination of properties that cannot be met by the conventional metal alloys, ceramics and polymeric materials. This is especially true for materials needed for the aerospace, underwater and transportation applications (Bledzki and Gassan, 1999).

A major and new area of research in composite development is the combination of natural fibres with thermoplastics *(*Sanadi *et al*, 1994*).*

A lot of waste materials are found littered in most places around the country; some of them are polyethylene bags and agricultural wastes. These wastes are usually in very large amounts; among these are the periwinkle shells, while polyethylene bags form a major eyesore on virtually all streets of the suburbs and even the urban areas.

Agricultural wastes occur in various forms, such as clarifier sludge, bagasse, grass, sisal, pineapple, coir, jute and corn stalks. These materials are termed as hard cellulose fibers because of their high tensile modulus and tensile strength *(*Kenry *et al*, 2008*).*

Recent years have seen an increased use of biodegradable materials in form of fibres, particulates, or even laminate as filler materials in thermoplastic composite materials

manufacture. Most of these biodegradable filler materials seem to be lighter, cheaper, easily processed and recycled with less health hazards and also provide more strength per unit mass than most brittle inorganic filler materials.

These organic fillers can be easily moulded with little or no abrasive action with equipment, and are readily available and cheap (Mohanty*et al*, 2000).

Currently, material scientists around the world are engaged in the synthesis of new green polymeric materials and processes that improve the environmental quality of a number of products. In fact, the renewability, biodegradability, high filling effect, abrasion resistance, acceptable specific strength properties, availability, light weight, ease of separation, high toughness, non-corrosive nature, low cost, good thermal properties, reduced tool wear properties of natural fibres/fillers are making them part of the best reinforcing materials for the synthesis of polymer based composites.(Tudu, 2009)

Over the last thirty years, composite materials, plastics and ceramics have been the dominant emerging materials(Tudu, 2009). The volume and number of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineering materials market ranging from everyday products to sophisticated niche applications (Tudu, 2009).

The possibility of taking advantage of particular properties of the constituent materials to meet specific demands is the most important motivation for the development of composites.

In composites, materials are combined in such a way as to enable us to make better use of their virtues while minimising to some extent the effects of their deficiencies. This process of optimisation can release a designer from the constraints associated with the selection and manufacture of conventional materials. He can make use of tougher and

lighter materials, with properties that can be tailored to suit particular design requirements. And because of the ease with which complex shapes can be manufactured, the complete rethinking of an established design in terms of composites can often lead to both cheaper and better solutions (Bryan, 1999).

Composites can generally be classified based on either matrix or reinforcements as shown in Fig 1.0.

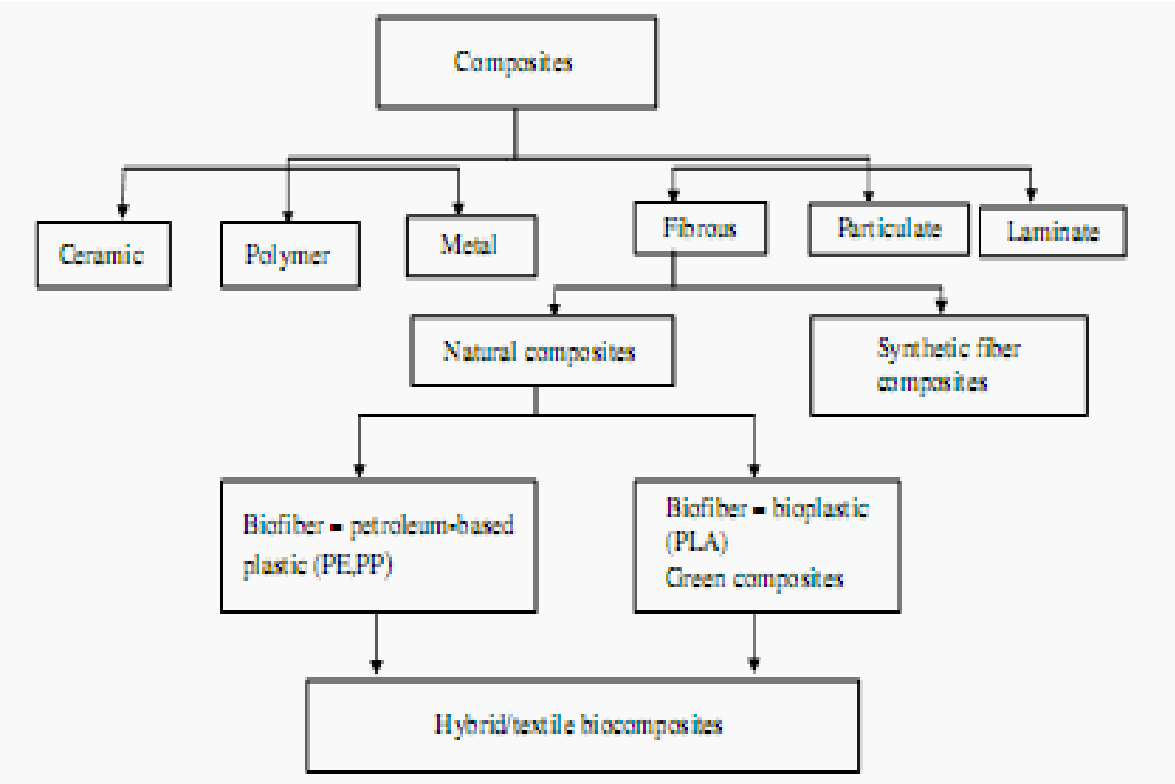


Fig 1.0 Classification of composites (Josmin *et al*, 2012)

1.1 BACKGROUND INFORMATION

The wide use of plastic products in various applications has greatly increased due to their unique properties, which include good mechanical properties, resistance to chemical attack and corrosion, ease of processing and recycling, cost effectiveness, light weight, and others. However, these properties are affected by many factors such as stress, temperature and environmentwhen they are exposed to service. In fact, this led to vehement objection of plastics in some specific applications. The attempts to overcome these obstacles led to incorporation of fillers (inorganic and organic) into plastics with a view to obtaining a plastic composite whose constituents act synergistically to withstand the challenges, thereby making plastics more reliable during use or processing. Generally, the composite properties are influenced by many factors such as filler characteristics, filler content, and interfacial adhesion and dispersion due to combination of more than one material (Abdullah*et al,* 2011).

It has been discovered over the years, that effort made to prevent indiscriminate litter of the environment with polymeric and agricultural wastes has been inefficient as these polymeric wastes such as low density polyethylene can be seen littered in most streets in Nigeria. This call for more effort into the provision of a permanent solution to the problem of polymeric waste disposal in the country and so bring about the motivation for this study.

1.2 STATEMENT OF THE PROBLEM

Waste water sachets are usually found littered on the streets of most communities in Nigeria; they constitute environmental pollution, they are not biodegradable, even if gathered and burnt, can cause air pollution.

Periwinkle shells also constitute environmental pollution especially around the coastal areas in various ways, among which are exuding bad odour, and serving as an eyesore, and can damage tyres of vehicles. It is clear that these two waste materials are problems in terms of environmental pollution. Hence, an attempt was made to address the environmental pollution effect of these two waste materials in this study by producing a composite from a combination of completely waste materials.

1.3 JUSTIFICATION/SIGNIFICANCE OF THE STUDY

Polyethylene is among the largest polymeric materials in use, especially in Nigeria. Periwinkle (marine snail) on the other hand is common seafood consumed by people mostly living in the coastal communities in Nigeria, leaving behind the shells which constitute environmental pollution.

These two products constitute a huge amount of wasteto the environment, therefore accumulating these wastes to recycle and produce composite materials, takes out a large quantity if not all of the waste from the environment, hence reducing the huge amounts spent by the Government in non biodegradable waste treatment and disposal.

Furthermore, cost effective materials could be produced from these materials which could compete favourably with materials already in use.

This research will also go a long way in reducing non-biodegradable waste in the environment, and also provide means of livelihood to people who may be interested in accumulating this waste for sale to recycling plants, hence converting waste to wealth.

1.4 AIM AND OBJECTIVES OF THE RESEARCH

The aim of this research is the production and characterisation of recycled LDPE/Periwinkle shell particulate composite, With a view to reducing some of the huge volume of agricultural and polymeric waste generated in the country.

The specific objectives targeted are:

- Conditioning of the periwinkle shells with NaOH
- To assess the effect of filler quantity and particle size on the properties of composite
- Characterizationof the composites produced using mechanical tests (tensile, impact, hardness and flexural tests), physical properties test (water absorption, density) and morphological study using Scanning Electron Microscope
- To carry out the degradation test for the composite

1.5 SCOPE OF THE RESEARCH

The scope of this work includes:

1. Surface conditioning of the periwinkle shells with 5 % NaOH solution
2. Compounding of the composite using the two roll mill.
3. Assessment of tensile, impact, flexural, hardness, water absorption and density properties
4. Assessment of microstructures of the impact fractured surfaces using SEM.
5. Assessment of the biodegradability of the composite using the weight loss method

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Composite materials

A composite may be defined as a material that consists of two or more distinct constituents materials which are bound together to form an integral unit (Choo, 1990). In composites, materials are combined in such a way as to enable us to make better use of their virtues while minimising to some extent the effects of their deficiencies. This process of optimisation can release a designer from the constraints associated with the selection and manufacture of conventional materials. He can make use of tougher and lighter materials, with properties that can be tailored to suit particular design requirements. And because of the ease with which complex shapes can be manufactured, the complete rethinking of an established design in terms of composites can often lead to both cheaper and better solutions (Bryan, 1999).

2.2 Limitations of conventional materials

The 3 basic materials in use before the advent of development of composites, are Polymers/plastics, Ceramics and Metals.

The development of composites was brought about by the need to introduce certain desirable properties that are not obtainable in conventional materials.

- **Polymers/Plastics**: Polymers are of low density. They have good short-term chemical resistance but they lack thermal stability and have only moderate resistance to environmental degradation (especially that caused by the photo-chemical effects of sunlight). They have poor mechanical properties, but are easily fabricated and joined (Bryan, 1999).
- **Ceramics:** Ceramics may be of low density (although some are very dense). They have great thermal stability and are resistant to most forms of attack (abrasion, wear, corrosion). Although intrinsically very rigid and strong because

of their chemical bonding, they are all brittle and can be formed and shaped only with difficulty (Bryan, 1999).

- **Metals:** Metals are mostly of medium to high density — only magnesium, aluminium and beryllium can compete with plastics in this respect. Many have good thermal stability and may be made corrosion-resistant by alloying. They have useful mechanical properties and high toughness, and they are moderately easy to shape and join. It is largely a consequence of their ductility and resistance to cracking that metals, as a class, became (and remain) the preferred engineering materials (Bryan, 1999).

On the basis of even so superficial a comparison it can be seen that each class has certain intrinsic advantages and weaknesses, although metals pose fewer problems for the designer than either plastics or ceramics.

2.3 Classification of composites

Composites as stated in Fig 1.0 can be classified based on matrix or reinforcement.

Based on matrix, there are the Metal matrix composites (MMCs), Ceramic matrix composites (CMCs), Polymer matrix composites (PMCs), while based on reinforcements; they can be classified as Fibrous, particulates, or laminates (Josmin et al, 2012).

2.3.1 Metal matrix composites

Large-scale research and development studies of metal-matrix composites (MMCs) date back to the 1960s, being stimulated at that time by the new availability of carbon and boron fibres, whisker crystals and, more indirectly, by successes achieved with reinforced polymer–matrix composites. The aero- space and defence industries were attracted by the prospects of a new type of constructional material possessing high

specific strength/stiffness. The fall in strength produced by rising temperature is more gradual in MMCs than with unreinforced matrix material, promising higher service temperatures. In addition, the MMC concept offers prospects of unique wear resistance and thermal expansion characteristics (Smallman and Bishop, 1999).

At present, particle-reinforced MMCs find the largest industrial application, being essentially isotropic and easier to process. Matrices based upon low-density elements have gradually come into prominence. The principal matrix materials are aluminium and its alloys, titanium and its alloys, and magnesium. Most MMCs are based on aluminium and its alloys. In recent years, cheaper particles and fibres of silicon carbide have become available, making them the commonest choice of reinforcement material (Smallman and Bishop, 1999).

2.3.2 Ceramic Matrix Composite

Reinforcement of cements and concretes with short fil aments of glass, steel or carbon is applied in the build- ing and construction industries. Costs are very closely scrutinized in these industries and the increase in cost associated with fibre reinforcement is often regarded as unacceptable unless there is promise of exceptional properties. In one example, a 2% v/v dispersion of steel wires (low-carbon or austenitic stainless) in a concrete matrix (*Wirand Concrete*2) confers improved resistance to crack propagation and greater flexural and compressive strength. These wires are typically 0.25–0.5 μ m diameter, with an aspect ratio of 100:1. The resistance of this composite to salt water and to pebble abrasion have led to its use for 40 t twin-fluked shapes (dolosse) for shore defences against heavy seas (Smallman and Bishop, 1999).

Since the 1980s there has been a sustained interest in the development of ceramic-matrix composites (CMCs) for high-temperature applications that demand exceptional strength and chemical stability (e.g. advanced engine designs, cutting tools). Ceramics

in general are inherently notch-sensitive and brittle: the introduction of fibres is primarily intended to improve the toughness of the matrix and reliability in service (Smallman and Bishop, 1999).

2.3.3 Polymer matrix composite

Polymers are generally produced as powders, pellets and liquids. In order to produce polymeric products with desired shapes, thermoplastics must be melted and cooled to a final piece, whereas thermosettings must undergo further polymerization to complete cross linking reactions to finally solidify into the desired shape. These operations are called processing. Many factors influence the processing operations. These include viscosity, orientation of heterogeneous phases, rate of reaction, volatility etc. (Ishida *et al*, 1980)

Polymer matrix composites are composed of a matrix from thermosetts (polyester, epoxyetc) or thermoplastics (PVC, Nylon, Polystyrene, polyethylene etc). These matrices can be structurally manipulated to produce a wide range of desired properties which are suitable for various engineering applications due to their low density, ease of processing, availability, less susceptibility to corrosive action by most solvents as compared with metals, and even lower cost as compared with metallic and ceramic matrices(Ishida *et al*, 1980)

Despite the enormous advantages attached to polymer matrices, limitations such as low strength, low processing temperature, formation of weakening voids, moisture absorption and a host of others are common.

2.4 Methods of composite production

A lot of methods exist in the production of composites; the various methods are a function of the class of composite.

2.4.1 Metal Matrix composites production

Metal matrix composite materials can be produced by many different techniques, of which casting is a very important one. The focus of the selection of suitable process engineering is the desired kind, quantity and distribution of the reinforcement (particles and fibres). By altering the manufacturing method, the processing and the finishing, as well as the form of the reinforcement, it is possible to obtain the different characteristic profiles, although the same composition and amounts of the components are involved. The production of a suitable precursor material, the processing to a construction unit or a semi finished material and the finishing treatment must be separated. For cost effective reasons, prototypes with dimensions close to the final product and reforming procedures are used (Ikechukwuka, 1997).

2.4.2 Ceramic Matrix Composites production

The manufacturing processes usually consist of the following three steps:

1. Layup and fixation of the fibres, shaped as the desired component.
2. Infiltration of the matrix material.
3. Final machining and if required further treatments like coating or impregnation of the intrinsic porosity.

The first and last steps are almost the same for all CMCs.

In step1, the fibres, often named roving are arranged and fixed using techniques used in fibre-reinforced plastic materials, such as lay up of fabrics, filament winding,

braiding and knotting. The result of this procedure is called fibre-preform or simply preforms (Naslain*et al*, 1989)

For the second stage, five different procedures are used to fill the ceramic-matrix in between fibres of the preform.

1. Deposition out of a gas mixture
2. Pyrolysis of a pre-ceramic polymer.
3. Chemical reaction of elements.
4. Sintering at a relatively low temperature in range of 1000-1200^{0}C (Naslain*et al*, 1989).

2.4.3 Polymer Matrix Composite production

In many cases, Polymer composite processing utilizes the same technique as polymer processing which include injection molding, compression molding, and extrusion. There are other techniques which are unique only to polymer composite processing. These include filament winding, pultrusion, and hand lay-up (Ishida and Koenig, 1980).

- The hand lay-up molding is the method of laying down fabrics made of reinforcement and painting with the matrix resin layer by layer until the desired thickness is obtained.
- Compression molding uses a press to compress either a dough of resin and fiber mixture, or the layers placed by a hand lay-up method or mechanical means, typically at an elevated temperature. With the compressive force, the void content is lower than the ordinary atmospheric pressure processing method.
- Injection Moulding is probably the most extensively used method for processing short-fibre reinforced thermoplastics. The fibre/resin mixture is fed into the hopper and transferred into the heated barrel. The material softens by heattransfer from the barrel wall. The molten material is collected in front of the

screw by rotation of the screw, and then injected with a high pressure into the mold cavity through the runner and the gate. The level of automation is the highest among many processing methods.

- Pultrusion is used only for polymer composite processing. A bundle of fibrerovings is passed through a wet resin bath, squeezed into a desired shape, passed into a heated die and cured into a final composite (Ishida and Koenig, 1980).

2.5 Thermosetting plastics

Thermosetting plastics are hard and stiff cross-linked materials that do not soften or become mouldable when heated. Thermosets are stiff and do not stretch the way elastomers and thermoplasts do. Other types of polymers are epoxy resins and other resins; unsaturated polyester resins (as in fiberglass), vinyl ester, phenolics, novolacand polyamide. Unsaturated polyesters are extremely versatile in properties and applications and have been popular thermosets used as polymer matrices in composites. They are widely produced industrially due to many advantages compared to other thermosetting resins. These include room temperature cure capability, good mechanical properties and transparency (Taj*et al.*, 2007).

2.6 Thermoplastics

Thermoplastics are polymers that require heat to make them processable. After cooling, such materials retain their shape. In addition, these polymers may be reheated and reformed often without significant changes in their properties. Some of the thermoplastics which have been used as matrix for natural fibres reinforced composites include High density Polyethylene (HDPE), Low density polyethylene (LDPE), Chlorinated polyethylene (CPE), Polypropylene (PP), Normal Polystyrene (PS),

Polyvinyl Chloride (PVC), mixture of polymers and Recycled thermoplastics (Taj*et al.*, 2007).

2.7 Particle reinforced polymer matrix composites

The term reinforced polymer is often used for composites with fibrous materials while filled polymer is the description used for polymers with particulate fillers.

Filler is a term used for materials other than continuous fibres and in general, implies that reinforcement effect is rather low. Fillers may be particulate or fibrous but the size is usually less than a millimetre. (Ishida, *et al*, 1979).

One of the major factors that govern the properties of composites is particle dispersion. It is of great importance that particles are dispersed uniformly in the polymer matrix to improve the properties of the composite.

Polymers generally possess low stiffness and hardness values as compared to the conventional metals. Therefore, reinforcement is now a necessity to improve the above properties. Particle polymer matrix interactions that lead to strengthening occur on the atomic or molecular level. The mechanism for strengthening is similar to that for dispersion hardening. Whereas the matrix bears the major portion of an applied load, the small dispersed particles hinder or impede the movement of dislocation. Thus, plastic deformation is restricted such that yield and tensile strengths, as well as hardness improve. For effective reinforcement, the particles should be small and evenly distributed throughout the matrix *(*Callister, *2007).*

Nano diamond particles have been used to reinforce polyvinyl acetate (pva) matrix. The results showed that even small additions of nano diamond led to significant enhancement in the hardness and elastic modulus of polyvinyl acetate (Maitra, *2005).*

The use of particulate reinforcements has proven to be formidable area to be explored to a greater extent in the production of composite materials of engineering applications.

2.7.1 Why Polyethylene matrix?

This thermoplastic is available in a range of flexibilities depending on the production process. High density materials are the most rigid, but this work intends to make use of the low density polyethylene (LDPE). The polymer can be formed by a wide variety of thermoplastic processing methods and is particularly useful where moisture resistance and low cost are required. Polyethylene is limited by a rather low temperature capability (200-250° F) but is manufactured in millions of pounds per year around the world.

Low density Polyethylene (LDPE) which is among the very common polymeric materials in use constitutes a major source of waste in the country, majorly in the form of packaging bags such as the commonly known "pure water" sachets. Hence this research hopes to take advantage of this waste material to produce a material of engineering applications, by recycling this waste and then reinforcing it to improve its properties.

2.7.2 Properties of Low density Polyethylene (LDPE)

LDPE is defined by a density range of 0.910–0.940 g/cm^3. LDPE has a high degree of short and long chain branching, which means that the chains do not pack into the crystal structure as well. It has, therefore, less strong intermolecular forces as the instantaneous-dipole induced-dipole attraction is less. This results in a lower tensile strength and increased ductility. LDPE is created by free radical polymerization. The high degree of branching with long chains gives molten LDPE unique and desirable flow properties.

The polymer can be formed by a wide variety of thermoplastic processing methods, and is particularly useful where moisture resistance and low cost are required.

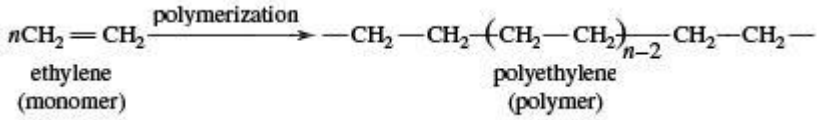
Table 2.1 gives a summary of the properties of low density polyethylene

Table 2.1: Typical Properties of LDPE

Property	Value
Density (g/cm^3)	0.92
Surface Hardness	SD48
Tensile Strength (MPa)	10
Flexural Modulus (GPa)	0.25
Notched Izod (kJ/m)	1.06+
Linear Expansion ($/°C \times 10^{-5}$)	20
Elongation at Break (%)	400
Strain at Yield (%)	19
Max. Operating Temp. (°C)	50
Water Absorption (%)	0.01
Oxygen Index (%)	17
Flammability UL94	HB
Volume Resistivity (log ohm.cm)	16
Dielectric Strength (MV/m)	27
Dissipation Factor 1kHz	0.0003
Dielectric Constant 1kHz	2.3
HDT @ 0.45 MPa (°C)	50
HDT @ 1.80 MPa (°C)	35
Material. Drying hrs @ (°C)	NA
Melting Temp. Range (°F)	220 - 260
Mould Shrinkage (%)	3
Mould Temp. Range (°C)	20 - 40

Source: http://www.gseworld.com/products/geomembranes/lldpe-smooth.html

Equation 2.1 shows the polymerization of ethylene



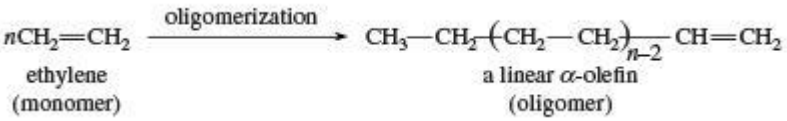
(2.1)

Figure 2.1: Low Density Polyethylene

2.7.3 Linear low density polyethylene (LLDPE)

LLDPE is defined by a density range of 0.915–0.925 g/cm^3. LLDPE is a substantially linear polymer with significant numbers of short branches, commonly made by copolymerization of ethylene with short-chain alpha-olefins (for example, 1-butene, 1-hexene and 1-octene).Because it has shorter and more branches, its chains are able slide against each other upon elongation without becoming entangled like LDPE which has long branching chains that would get caught on each other.This gives LLDPEhigher tensile strength than LDPE, it exhibits higher impact and puncture resistance than LDPE. Lower thickness (gauge) films can be blown, compared with LDPE, with better environmental stress cracking resistance but is not as easy to process as compared with LDPE.The resultant structure has a linear backbone, but it has short, uniform branches that, unlike the longer branches of LDPE,prevent the polymer chains from packing closely together. (Furumiya*et al,* 1986)

Equation 2.2 shows the formation of Linear Low Density Polyethylene



(2.2)

Figure 2.2: LLDPE

Table 2.2 gives a summary of some properties of Linear Low Density Polyethylene.

18

Table 2.2: Typical properties of LLDPE

Property	**Value**
Density	0.93g/cm^3
Surface hardness	SD48
Tensile strength	30 MPa
Flexural modulus	0.35 GPa
Notched izod	1.06+ kJ/m
Linear expansion	20×10^{-5}/°C
Elongation at break	500%
Strain at yield	20%
Max. Operating temp.	50 °C
Water absorption	0.01%
Oxygen index	17%
Flammability UL94	HB
Volume resistivity	$10^{16}\Omega\cdot$cm
Dielectric strength	25 MV/m
Dissipation factor 1 kHz	909090
Dielectric constant 1 kHz	2.3
HDT @ 0.45 MPa	45 °C
HDT @ 1.80 MPa	37 °C
Material drying	NA
Melting Temp. Range	120 to 160 °C
Mould Shrinkage	3%
Mould temp. range	22 to 60 °C

2.7.4 Medium density polyethylene (MDPE)

This has a little less branching than the HDPE. It is less notch sensitive than HDPE and has better stress cracking resistance. It has a density range of 0.926 - 0.940 g/cm^3, a summary of its properties are as presented in Table 2.3.

Table 2.3: Typical properties of MDPE

MDPE	
Density	0.926-0.940 g/cm^3
Young modulus/ tensile modulus	172–379 mpa
Tensile strength(σ_t)	12.4–19.3 mpa
Elongation @ break	100–150%
Brittleness, low temperature	-118 °c
Vicat	99-124 °c
Specific heat (c)	1.916 kj/kg.k

(Brandrup*et al*, polymer handbook fourth edition)

2.7.5 High density polyethylene (HDPE)

High Density Polyethylene (HDPE) is prepared from ethylene by a catalytic process. The absence of branching results in a more closely packed structure with a higher density and somewhat higher chemical resistance than LDPE. HDPE is also somewhat harder and more opaque and it can withstand rather higher temperatures (120° C for short periods, 110° C continuously).

High density polyethylene lends itself particularly well to blow moulding, e.g. for bottles, cutting boards, dipping baskets, dippers, trays and containers.

It has minimal branching of its' polymer chains. Because it is denser, it is more rigid and less permeable than the LDPE. It has a density of 0.941-0.965 g/cm^3.

2.7.6Ultra high molecular weight polyethylene (UHMWPE)

UHMWPE has extremely long chains, with molecular weight numbering in the millions (usually between 2 to 6 million). In general, HDPE molecules have between 700 and 1,800 monomer units per molecule, whereas UHMWPE molecules tend to have 100,000 to 250,000 monomers each. The chains of UHMWPE align in the same direction. The bonds between the chains are not very strong however, because they are so long there are more bonds holding it together than polyethylene with shorter chains. These long chains give UHMWPE high tensile strength. The longer chains serve to transfer load more effectively to the polymer backbone by strengthening intermolecular interactions. This causes the material to be very tough and gives it the highest impact strength of the polyethylenes (Christian *et al*, 2013).

2.8 Periwinkle Shell

Periwinkle shells are an external exoskeleton which protects the small edible species of medium sized sea snails of the marine gastropod molluscs from their predators and mechanical damage(Adewuyiand Adegoke, 2008).

Structurally, the wrinkle shell has several layers and is typically made of an organic matrix (conchiolin) which is bonded with calcium carbonate precipitates. These calcium carbonate-filled organic matrix shells are impervious to water and this property makes it possible for periwinkle shells and their derivatives to have very wide applicationsespecially in areas where water resistance is of priority(Painter and

Hemmer, 1979).Table 2.0 shows the XRF analysis of periwinkle shells presented in the work of (Aisien*et al,* 2014).

Table 2.0: XRF Analysis of Periwinkle Shell Particles(Source: Aisien*et al,* 2014)

Element	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	BaO	Na_2O	SiO_2	ZnO	MnO_2	TiO_2	Cuo
%	9.2	41.3	5.0	1.4	1.2	0.1	1.38	33.2	3.2	1.0	0.02	1.3

2.9 Related literature

(Aku *et al,* 2012) studied the characterisation of periwinkle shell as asbestos free brakepad material. They developed and characterized the composite using X-ray diffractometer (XRD), thermo gravimetric analysis (TGA/DTA), Fourier transform infra red Spectrometer, while the density, hardness values and wear rate of the periwinkle shell were also determined. They obtained various results which compared favourably with asbestos commonly used in brake pad production, thus showing the potency of periwinkle shells in engineering applications.

(Agbede *et al*, 2009), studied the suitability of periwinkle shell as partial replacement for river gravel in concrete production. They utilised ordinary Portland cement from Dangote as binding agent together with river Benue sharp sand, while periwinkle shell and some gravel served as reinforcement.The mix was designed using the absolute weight method. Physical and mechanicalProperties of periwinkle shells and river gravel were investigated through the following tests:Specific gravity test, bulk density, slump and aggregate impact value tests. Cube crushing tests were also carried out to determine the compressive strengths of periwinkle-gravel concretes. Sieve analysis was carried out using sieves arranged in decreasing size of opening and placed on a shaker in order to differentiate samples of the aggregate into fractions. The coefficients of uniformity, curvature and gradation were determined.

Their results showed good physical and mechanical properties of the final composite material.

(Njoku *et al*, 2011), studied the effect of particle size variation and weight fraction on the tensile and modulus of periwinkle shell reinforced polyester composite.

The hand lay-up technique was employed in producing the composite laminates.

Four different particle sizes of ground periwinkle shell were used, 400, 600, 800 and 1000μm.Appropriate quantities of reinforcing constituents were determined and mixed with proportionate amounts of polyester resin to give: 10%, 20%, 30%, 35%, 40% and 45% weight fractions of periwinkle shell particles. The "mix" was vigorously stirred to ensure homogeneous dispersion of the periwinkle particles in the resin after the additions of methyl ethylketone peroxide and cobalt nephthenate which served as a catalyst and accelerator respectively during curing of the polyester resin to give a solid laminate. They concluded at the end of their study, that addition of particles led to an increase in strength and that finer particles gave better reinforcements.

(Nwanonenyi, *et al*, 2013), studied the effect of particle size, filler content and compatibilization on the properties of Linear low density polyethylene (LLDPE) filled periwinkle shell.

The thermoplastic composite of LLDPE and periwinkle shell powder (of varying particle size of 75μm, 125μm and 150μm) was prepared by proper and thorough mixing of 200g of LLDPE with periwinkle shell powder contents of 0, 5, 10, 15, 20, 25 and 30 wt % for each composite samples without compatibilizer. Then, the resulting mixture of the composite constituents of each sample was melt-blended and homogenized in an injection moulding machine at 170ºC, and the resulting composites were produced as sheets. In the second set of composites preparations, 200g of LLDPE with periwinkle shell powder content of 20% wt and compatibilizer contents of 0.5, 1.0, 1.5, 2.0, and 2.5 wt % for each composite samples were mixed, fed into an injection moulding machine and processed as stated above.

They also found that the tensile strength of the composite produced increased with increase in periwinkle shell powder filler for the entire particle sizes investigated.

(Michealand Muneer, 2012), carried out a research on the effect of filler content on the mechanical properties of periwinkle shell reinforced cashew nut shell liquid(CNSL) resin composites. They considered, three particle sizes (400μm, 600μm and 800μm) of milled periwinkle-shell (PWS) filled (CNSL) resin composites were developed by compression moulding technique with varying filler content (10% to 40% by weight). The developed PWS filled CNSL composites were then characterized by tensile, flexural and impact strength tests. The effect of filler content and particle size on the mechanical properties of the PWS filled CNSL composites was investigated. Results showed that, there was an improvement on the mechanical properties as the filler content increases while properties decrease as filler size increases. Properties of tensile strength, flexural strength, % elongation, tensile modulus and impact strength slightly increased as the filler loading increases. While % elongation, tensile and flexural strengths decrease as particle sizes increase, tensile modulus and impact strength increase as particle sizes increase. The optimum properties were observed at 30% filler content and 400μm particle size.

Now, in the light of all these literatures that have been reviewed, it is obvious that the potentials of periwinkle shells as a filler material in polymer matrix composite are enormous.

Hence, this research is aimed at exploring the possibility of producing a composite from completely waste materials, that is, combining recycled low density polyethylene (RLDPE) and another waste (periwinkle shells), at varying particle sizes to assess the effects of the change in particle sizes and filler quantity on the material to be produced. With respect to the works reported above, it shows the feasibility for the use of periwinkle shells in polymer matrix systems.

CHAPTER THREE

3.0 MATERIALS AND METHODS

3.1 Materials

The periwinkle shells were sourced locally from IkotEtetuk community in Ikot-Abasi L.G.A of Akwa-Ibom state, Nigeria.The polyethylene matrix was obtained by recycling waste Water sachet picked from Danraka, Samaru in Zaria, Kaduna state, Nigeria. Other materials were Aluminium foil paper, distilled water, beakers, Sodium Hydroxide (NaOH) pellets.

3.2 Equipment

The equipment that were used in this research includes:

- Two roll mill by Allen-Bradleywith model no 802T-WS1P
- Muffle furnacewith model no 110706
- Scanning Electron Microscope(SEM) model JEOL JSM-6480LV
- Hounsfield Monsantotensometer with serial no 386083-W9
- Charpy impact testing machine with model no 412-07-15269C
- Durometer hardness tester with model no DIN 53505
- Digital weighing balance with precision of 0.002
- Carver hydraulic press with model no 3851-0

3.3.0 Periwinkle shell preparation

The Periwinkle shells were washed, weighed, sun dried, followed by oven drying at 105°C for 3 hours until the moisture was ensured to have greatly reduced (Aku *et al,* 2013).

3.3.1 Surface conditioning of the periwinkle shells

The periwinkle shells were immersed in the 5% NaOH solution,for every 5wt% NaOH concentration, 100mls of distilled water was used to produce the mixture; this process was repeated until desired quantity was achieved. It was immersed for a period of 2 hours, after which it was removed from the solution, washed with distilled water and dried naturally.

The periwinkle shells were crushed, milled and sieved unto a set of sieves of 106µm, 75µm, and 53µm particle sizes.

3.4 Synthesis of the recycled LDPE and periwinkle shell composite

The LDPE was recycled in the Nigerian Institute for Leather and Science Technology (NILEST), Zaria.

The composite samples were produced by a compounding process involving the addition of the shredded recycled LDPE while the rolls were in counter clockwise motion for a period of 10 minutes and at a temperature of 150°C,upon achieving a paste like matrix, the filler material was introduced by gently applying manually as the rolls rotate at a rate of 500 rpm. The percentage of periwinkle shell was varied from 5 vol% to 25 vol% with an interval of 5 vol% for the following particle sizes 106µm, 75µm, and 53µm. The compounding was carried out at a temperature of 150°C, while curing time was for a period of 5 minutes.

The samples were produced after compounding by inserting the paste into various moulds of dimensions 100×100×5 mm, then subjecting the covered moulds to the

hydraulic press for 5 minutes to enable it cure. Sectioning according to ASTM standards for physical and mechanical tests were carried out as further discussed in sections 3.5 and 3.6.

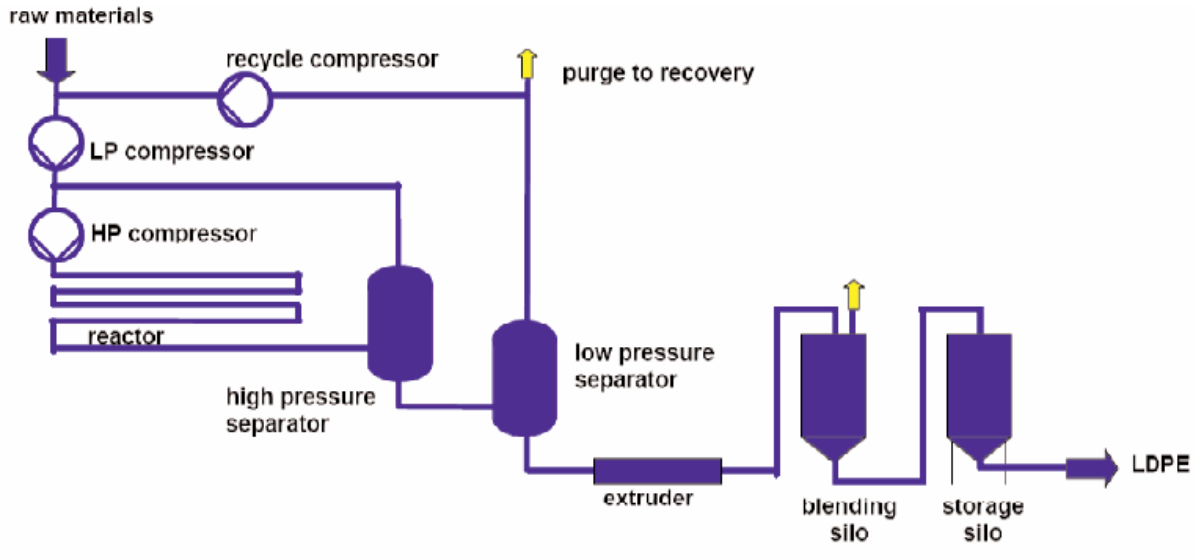


Fig 3.1: Set up for the recycling of low density polyethylene (source: *http://www.plasticseurope.org retrieved 15*march, 2014)

3.5 PHYSICAL PROPERTIES TESTS

3.5.1 Water absorption test

The specimens were prepared from a 5mm thick plate with dimension 100mm width and 100mm length. Water absorption test (ASTM D570) was done by total immersion of specimens in distilled water at room temperature for 14 days. The water absorbed was determined by weighing the samples at regular intervals of 24 hrs,with the aid of digital weighing balance. The water adhering on the surface of the specimen was carefully wiped off using filter paper. The water uptake in the composite was measured by the weight gain method of the material using the formula;

$$\% \text{ Water absorbed } = \frac{W_t - W_1}{W_1} \times 100 \qquad (3.1)$$

W_t= weight at time t (hrs)

W_1= weight before immersion

3.5.2 Determination of density

The densities of the samples were determined using ASTM D-792.

The mass and volume were obtained by first taking a clean sample and weighing it in air using a digital weighing balance in the laboratory, and then suspend it in water. The volume of the sample when immersed in water was determined by measuring the volume of the water displaced.

The density was then calculated from the expression:

$$Density = \frac{Mass}{Volume} \qquad (3.2)$$

3.5.3 Soil degradability test

The soil degradability test for the composite was carried out according to ASTM D 5338 standard.

$$\%\ \text{Weight loss} = \frac{W_1 - W_2}{W_1} \times 100 \qquad (3.3)$$

W_1= Mass before burial

W_2= Mass after burial

The soil location used for this test is at Silver Jubilee quarters, Zaria, and the soil pH was determined to be 5.7, which is slightly acidic, while the nature of the soil was confirmed to be sandy-loam.

3.6.0 Mechanical property tests

The tensile test, 3-point bending test, and impact tests were carried out following the prescribed ASTM D-638, D-790, and D-256 standards respectively, while the Durometer hardness ASTM D-2240 shore D was used for the hardness, to ascertain the relevance of the composite material as an engineering material. All the mechanical tests were done at room temperature.

i. **Impact Test:** The impact energy test of the composite samples was determined using Charpy Impact testing machine with model no 412-07-15269C. The procedure used was in accordance with that recommended by ASTM D-256. Samples were sectioned to 100mm×10mm×5mm dimensions after which 3 samples were each were prepared, mounted on the machine, and a swinging pendulum released, under gravity, to hit the sample(s). The energy at impact was read directly from a dial indicator of the machine and the average values of the results obtained are given in Fig 4.1 and Table A3 (Appendices).

ii. **Tensile Test:** The tests were performed in accordance with standard procedure specified in ASTM D638**,**using the Hounsfield MonsantoTensometer in the Department of Mechanical Engineering, A.B.U, Zaria. Samples were sectioned to 100mm×15mm×5mm, with gauge dimensions 40mm×10mm×5mm. 3 samples each were then tested and the average values obtained maximum stress and strain values recorded for tensile strength computation. The Tensile values are given in Fig 4.2 and Tables A.4 – A.6 (Appendices).

iii. Hardness Test: The hardness tests were carried out using the Durometer Hardness tester with model no DIN 53505, on shore D scale according to ASTM D-2240.

The hardness test results are as presented in Fig 4.3 and Table A7 (Appendices).

iv. **Flexural Test**: Flexural tests were carried out on a HounsfieldTensometer. Samples wereprepared to dimensions of 100mm×40mm×5mm. 3 samples eachwere subjected to bending by supporting them at both ends and a midpoint load applied until failure as recommended in ASTM D790.The results obtained are presented in Figure 4.4 and Table A8 (Appendices).

3.7 MICROSTRUCTURAL ANALYSIS

The Scanning Electron Microscope (SEM) with model noJEOL JSM-6480LV was used to study the morphology of material produced. For the SEM analysis, the impact fractured surfaces of the best samples in terms of impact properties in each of the particle sizes considered, was viewed to reveal the failure mode of the composite material.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSIONS

This chapter presents and discusses the results obtained from the mechanical tests: such as impact, tensile, flexural and hardness tests carried out on the composite samples. It equally explains the micrographs of the samples obtained from SEM.

4.1 Results

The results for the mechanical and physical properties of the Recycled Low Density Polyethylene/Periwinkle Shell (RLDPE/PWS) composite with varying filler quantity for the three particle sizes are presented in tabular form: Tables A.1 – A.15 (Appendices). These results are represented graphically, Figures 4.1 – 4.9. The morphologies of the composite are shown, Plates I to VIII.

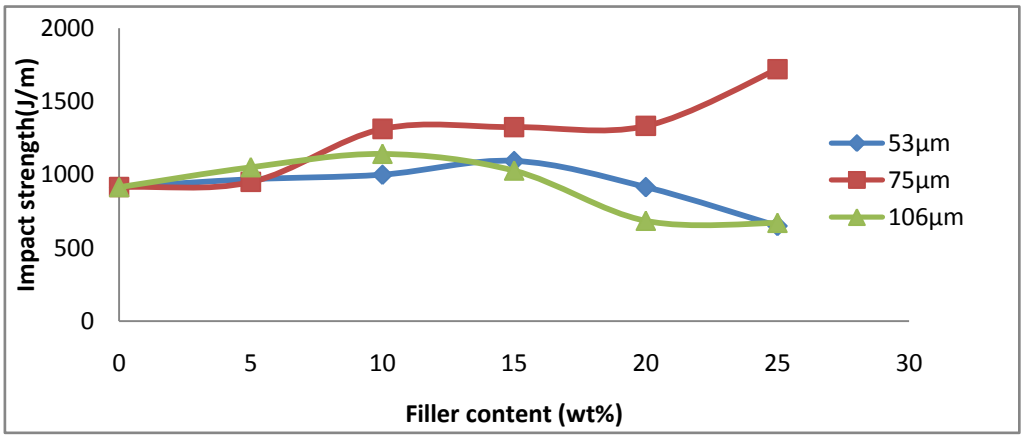


Figure 4.1: Impact energy profile for recycled low density polyethylene/periwinkle shell composite at varying particle sizes and filler contents

4.1.1 Impact Strength

Figure 4.1 shows the impact strength profile for the produced RLDPE/PWS composite. From figure 4.1, it can be deduced that there was a progressive increase in impact strength for the 75µm particle size as the filler content increased from 5wt% to 25wt% with 950J/m and 1720J/m respectively while that of the recycled low density polyethylene was 915.63J/m showing an 87.9% increase in impact strength. On the other hand, a slight increase in impact strength was observed with increase in filler content from 5wt% to 10wt% for 106µm sieve grade, and later falls from15wt% filler content to 1028.57J/m and a subsequent decrease to 25wt%. Furthermore, for the 53µm particle size, a gradual increase in impact strength was observed from 968.75J/m at 5wt% to 1093.75J/m at 15wt% after which a decrease to 915.63J/m was observed.

The highest impact strength was observed at 25wt% filler content with 1720J/m and at 75µm particle size, while the least impact strength was observed at 25 wt% filler quantity for the 53µm particle size. An increase in filler was observed to have improved impact energy beyond 915.63J/m reported for the control sample (RLDPE).

The increase in impact energy of the composite upon increase in filler content may be attributed to the presence of the filler material in the matrix, thus, increasing the

restriction to deformation processes and hence improving ability to absorb and dissipate energy, while a decrease may have been as a result of particle to particle interaction, hence giving rise to zones of weakness which may induce failure at lower stress values.

This is in agreement with the work of Bigg(1987),Nwanonenyi *et al* (2013),andMicheal and Muneer (2012); who studied the mechanical properties of particle filled polymer systems and reported an increase in impact strength with increase in filler loading, and is also in line with the work of Guo *et al (2005),* who investigated carbonate/polypropelene systems and found that an increase in filler content gave rise to an increase in the ability to absorb and dissipate energy and hence improved impact strength, but decreased with further filler content addition.

These results are also shown, Table A3 to A5 of the Appendices.

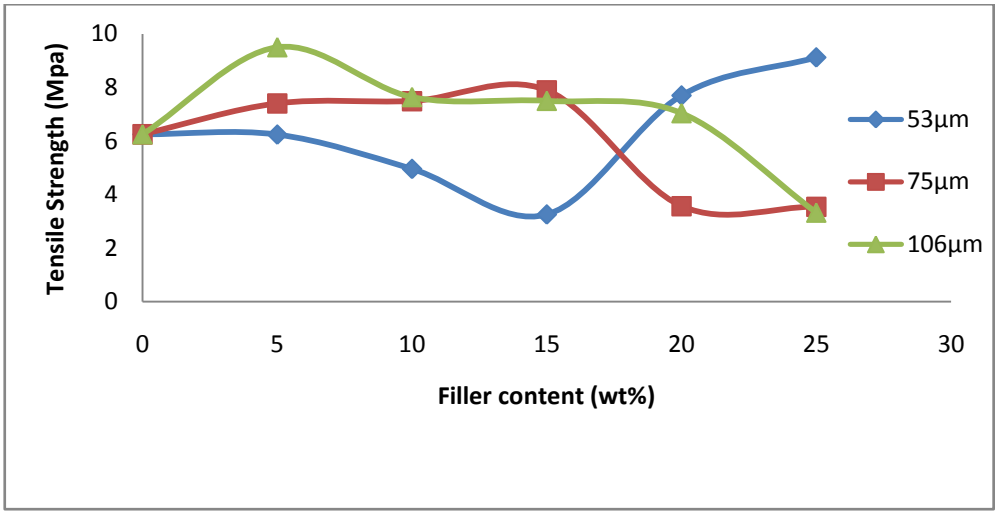


Figure 4.2: Tensile strength profile for RLDPE/PWS composite at varying filler content and particle size

4.2.1 Tensile Strength

Figure 4.2 shows the tensile strength of the composite material produced, the profile shows a progressive decrease as filler quantity increased from 5 wt% to 25 wt% for 106µm, with 9.50 Mpaand 3.33 Mpa respectively. For the 75µm particle size, there was an increase in tensile strength as the filler quantity increased from 5wt% to 15wt% with values of 7.40 Mpa at 5 wt% and 7.89 Mpa at 15 wt%, beyond which there was decrease in tensile strength, followed by a decrease to 3.57 Mpa at 20 wt% filler content, and 3.55 Mpa at 25 wt% filler content. When we compare this with the control sample with a value of 6.25 Mpa, we can deduce that at finer particle sizes, tensile strength decreases upon addition of filler, but increases at a point where there is enough volume to provide better stress transfer between filler and matrix, and hence improved tensile strength.

At 53µm particle size, there was also a decrease in tensile strength from 6.24 Mpa at 5 wt% to 3.56 Mpa at 15 wt% filler quantity, but at 20 wt% filler, a sudden increase to 7.70 Mpa was observed, with the highest value of 9.12 Mpa for 53µm particle size at 25

wt% filler content. This sudden increase in tensile strength may be due to increased surface area, which enhanced load transfer between RLDPE matrix and PWS filler. This is in agreement with the work of Romanus *et al (*2012), who carried out a research on the effect of alkali treatment on coir reinforced cashew nut shell liquid, and reported an increase in tensile strength with a subsequent decrease in tensile strength.

The highest tensile strength of 9.50 Mpawas observed with 5 wt% filler content at 106µm, while the least value for the tensile strength was 3.26 Mpa at 15 wt% filler and 53µm size.

The increase in tensile strength of the composite at 75µm could be attributed to an improvement in stress transfer between matrix and filler material, caused by improved interfacial adhesion. Furthermore, an increase in surface area and bonding surface has been known to improve tensile strength as reported by Romanus *et al* (2012), and Micheal and Muneer (2012), but a decrease could be attributed to a decrease in wettability, which could be as a result of the increase in filler-filler interaction. On the other hand, the progressive decrease in tensile strength of the composite at 106µm may be attributed to improper distribution of the filler in the matrix, and a decrease in interfacial adhesion between filler/matrix of the composite, thereby decreasing its tensile strength.

The tensile results are also presented, Tables A6 to A8 of the Appendices.

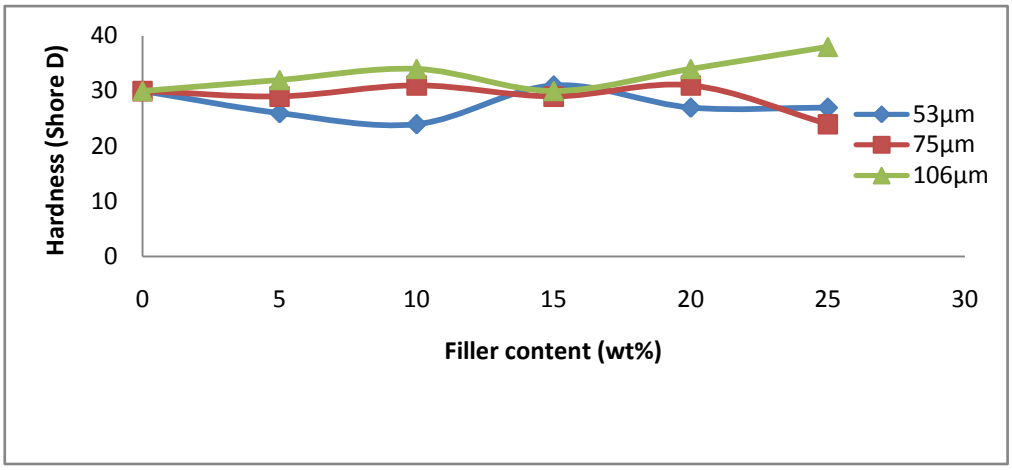


Figure 4.3: Hardness profile for recycled low density polyethylene/periwinkle shell composite at varying filler content and particle size

4.3.1 Hardness property

Figure 4.3 shows the hardness profile for the RLDPE/PWS composite at different filler quantity and particle size.

The figure shows a gradual increase in hardness for the 106µm and 75µm particle sizes at 5 wt% (32 shores) and 10 wt% (34 shores) filler quantity, but there was a decline in hardness at 15 wt% (30 shores) filler quantity followed by an increase. The increase can be attributed to the increase in the hard and brittle phases of the periwinkle shells in the polymer matrix, but at the point of decline, an uneven distribution of phases may have been responsible for the decrease in hardness. Furthermore, the presence of the filler may have improved the matrix surface resistance to indentation. This is in agreement with the work of Agunsoye *et al* (2012), who studied the effect of palm kernel shell on the microstructure and mechanical properties of recycled polyethylene/palm kernel shell particulate composite.

The 53µm particle size indicated the least increase in hardness of the three particle sizes considered at three different filler quantities of 5 wt% (26 shores), 10 wt% (24 shores) and 20 wt% (27 shores), which may be due to the fact that the fine particles could not

provide much resistance to surface indentation. Furthermore, the indenter may rest on the surface of the filler material at some point and on the matrix surface at other points, thus giving such behaviour in hardness at certain compositions as shown on Figure 4.3.

The highest hardness was observed at 25 wt% filler with 38 shores in the 106µm particle size, while the least hardness was seen at 25 wt% filler with 24 shores in 75µm particle size, and also at 10 wt% filler (24 shores) in 53µm particle sizes, as opposed to the control sample with a value of 30 shores.

The results are further reflected, Tables A9 to A11 of the Appendices.

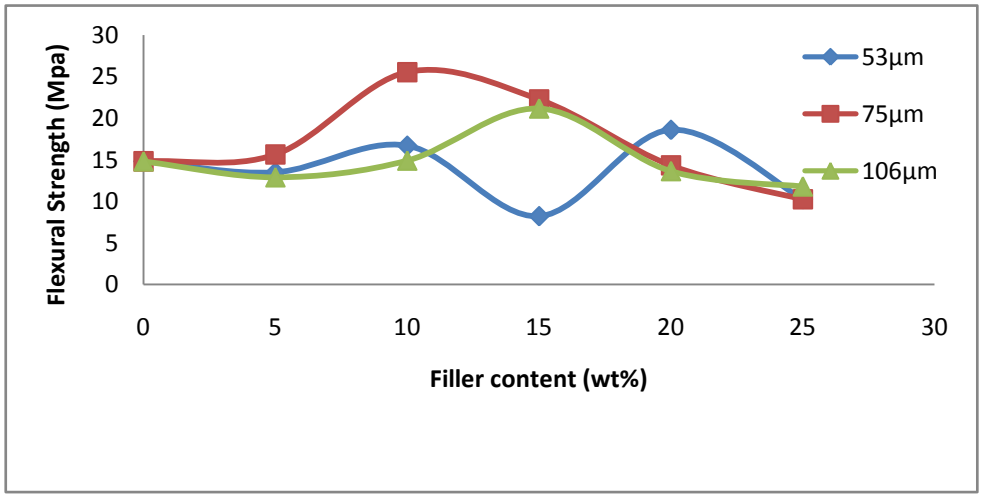


Figure 4.4: Flexural strength profile for RLDPE/PWS composite at varying particle size and filler content

4.4.1 Flexural strength

Figure 4.4 shows the flexural strength of the RLDPE/PWS composite.

From the figure, it can be observed that there was an increase in flexural strength at 75µm particle size considered with an increase in filler up to 10wt% with 25.51 Mpa which was the highest, but beyond 10 wt% filler, a decrease was observed. A decrease at 5 wt% was observed for the 106µm particle size, but a sudden increase followed up to 15 wt% with 21.14 Mpa, beyond which a decrease was observed. On the other hand, initial increase and decrease, were observed with a sharp decline in flexural strength from 16.67 Mpa at 10 wt% filler to 8.19 Mpa at 15 wt% for the 53µm. The control sample had a flexural strength of 14.79 Mpa, which shows that the increase in filler produced an increase in flexural strength but decreased as the filler content increased to a point where there was extensive interaction between particles of filler material.

The increase in flexural strength upon addition of filler material may be due to the improved interfacial adhesion between matrix and filler material, caused by the alkali conditioning of the filler, hence, increasing surface area available for matrix to filler interaction. This improves stress transfer between matrix and filler,while a decrease

may be attributed to extensive interaction between filler materials in the matrix, thus, giving rise to inefficiency in bonding surface, and hence, reduced stress transfer between matrix and filler material. This is in agreement with the work of Obasi*et al* (2014) who asserted that influence of alkali treatment on properties of oil palm press particles, leads to an increase in flexural properties, and subsequent decrease with increase in filler content. This behaviour is also supported by the work of Isiaka and Temitope (2013), where they studied the influence of cow bone particle distribution on mechanical properties of cow bone reinforced polyester composite.

Values are reflected, Table A12 of the Appendices.

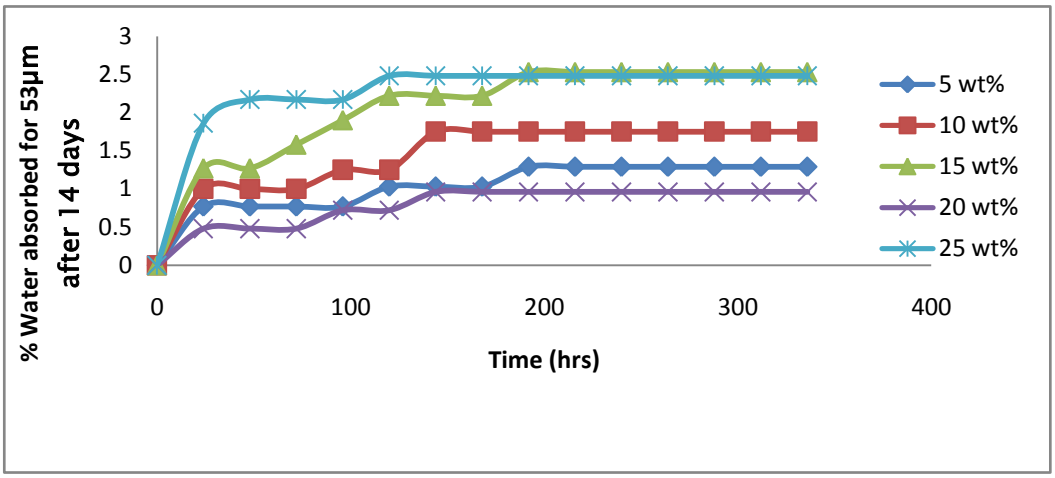


Figure 4.5: Water absorption profile for 53µm particle size composite at varying filler contents

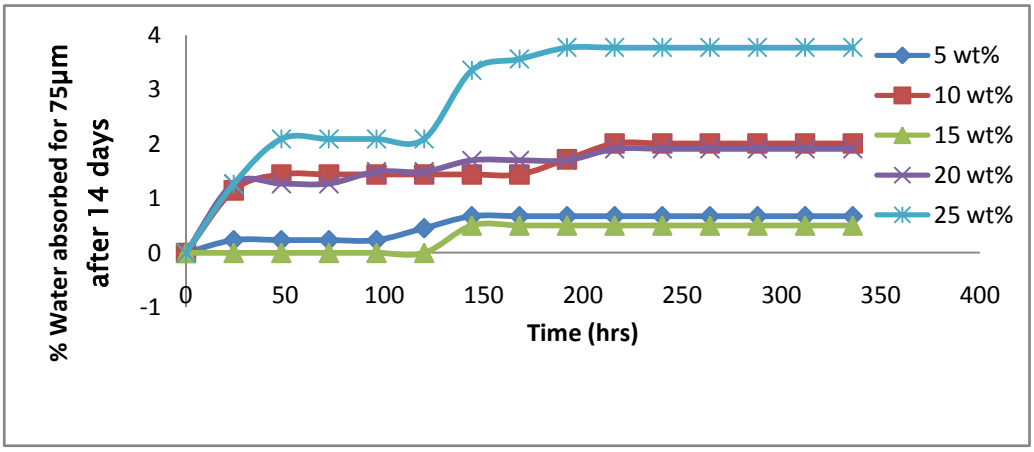


Figure 4.6: Water absorption profile for 75µm particle size composite at varying filler contents

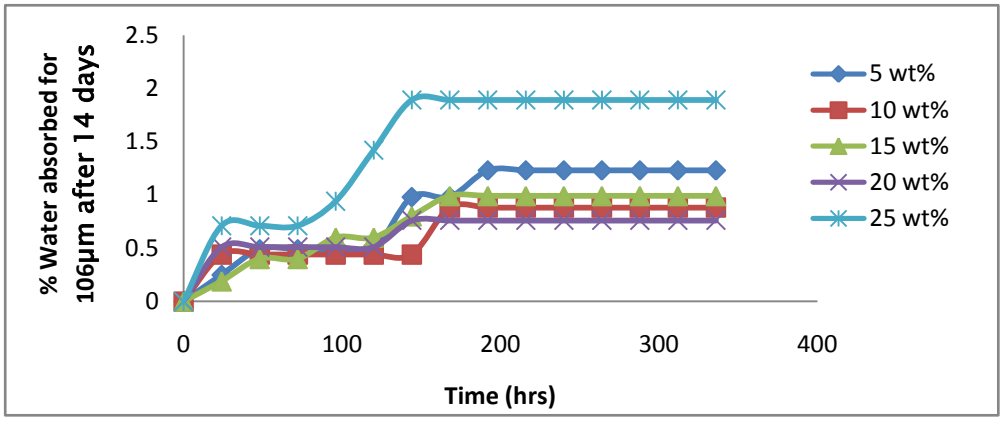


Figure 4.7: Water absorption profile for 106µm particle size composite at varying filler contents

4.5.1 Percentage Water Absorption

From Figure 4.5, it can be observed that water absorption increases with time and also with filler content, with the highest water absorption at 2.48% for 25 wt% filler content and at 53μm particle size, while the least absorption is at 20 wt% filler with a 0.24% absorption rate. The increase may be attributed to increased surface area of filler in matrix, hence, leading to increased number of pores.

Figure 4.6, shows the water absorption profile for the 75μm particle size. It can be deduced that there was a slight increase in water absorption with time, with the highest water absorption rate being 3.77% at 25 wt% filler content, while the least water absorption was observed at 15 wt% filler, where there was no water absorption recorded for 5 days, beyond which only a 0.5 wt% water absorption rate was recorded for the remaining days. The behaviour at 15 wt% may be attributed to improved interfacial adhesion, thereby decreasing number of voids created.

Figure 4.7 shows the water absorption profile for 106μm particle size. We can observe that this particle size presented a water absorption rate that was much less, as compared with other particle sizes considered, which may be due to the decrease in surface area of filler in matrix, thus creating less number of voids as compared with other particle sizes. The highest water absorption of 1.89% was observed at 25 wt% filler, while the least water absorption of 0.19% was obtained at 15 wt% filler.

Generally, it was also observed that beyond 192 hrs (8 days), at all particle sizes, there was no further increase in water absorption, which could be attributed to the fact that the pores created may have been saturated with water, thus giving rise to such behaviour as also shown in Tables A13 to A15 in the Appendices . This is in agreement with the work of Agunsoye*et al* (2012), who studied the effect of palm kernel shell on the microstructure and mechanical properties of recycled polyethylene/PKS composite, and

reported an increase in water absorption rate as particle filler increases, but reported a constant trend at a certain point.

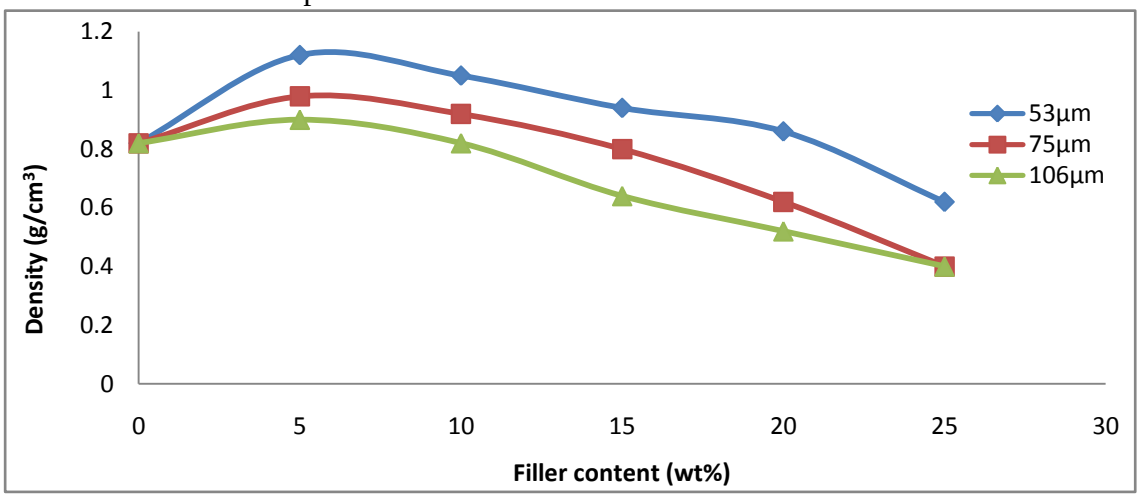


Figure 4.8: Density profile for recycled low density polyethylene/periwinkle shell composite at differing particle sizes and filler content

4.6.1 Density of the composite

From Figure 4.8, it can be deduced that the density of the composite initially increased with addition of filler, while a decrease in particle size presented an increase in density, with the highest density at 5wt% for the 53µm particle size with a value of 1.12g/cm^3, while the least value was obtained at 25wt% for the 106µm particle size with a value of 0.4g/cm^3.

It can be observed that upon addition of filler material, an increase in density was observed at 5wt% filler in all particle sizes considered, beyond which a decrease in density as filler content increased was observed. This may be attributed to the increased number of voids created by the periwinkle shell particles, hence decreasing the overall density of the composite. This is in agreement with the work of Naidu *et al* (2013), who worked on the study of the mechanical properties of groundnut shell reinforced polymer composite, where they reported a decrease in density with increase in filler content and an increase in density with a decrease in particle size.

Table 12 of the Appendices also shows the values obtained for the densities.

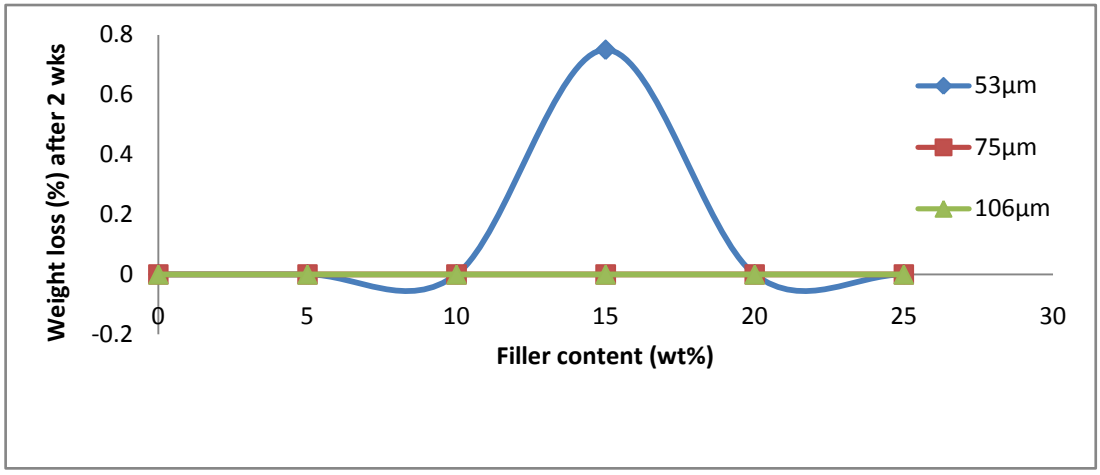


Figure 4.9: Degradation profile for composite material

4.7.1 Degradation of the composite

Figure 4.9 shows that the composite generally has a very low degradation rate in the soil, and this is seen in the profiles shown for the 75µm and 106µm particle sizes, where there was no weight loss observed in the material. The 53µm particle size presented slight weight loss of 0.75% at 15 wt% filler content. This may be due to voids in the composite, thus providing openings for micro organisms to act on the material. The low rate of degradation may be attributed to the low activity of micro organisms, caused by the acidity of the soil. The nature of the soil is sandy-loam, and sandy soils have been reported to be porous, and so do not favour high microbial activity. This is in agreement with the work of Cooke (1990), on the biodegradability of polymers and fibres.

In addition, microbial activity decreases with depth, and very high microbial activity have been reported to occur within 0 to 10cm depth (Cooke, 1990). Furthermore, the time of exposure plays a role, the greater the time of exposure, the greater the time for micro-organisms to act on the material. This is in agreement with the work of Pagga*et al* (1995), involving the determination of the aerobic biodegradability of polymeric materials in laboratory controlled composting environment.

Tables A17, A18 and A19 in the Appendicesshows values obtained for soil burial test.



Plate I: Surface morphology of impact fractured surface of 100wt% RLDPE (×20)

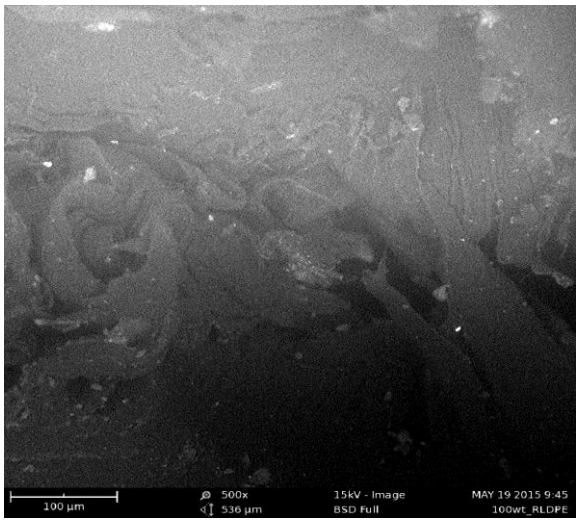


Plate II: SEM morphology of impact fractured surface for 100 wt% RLDPE indicating the entangled nature of the polymer (×500)

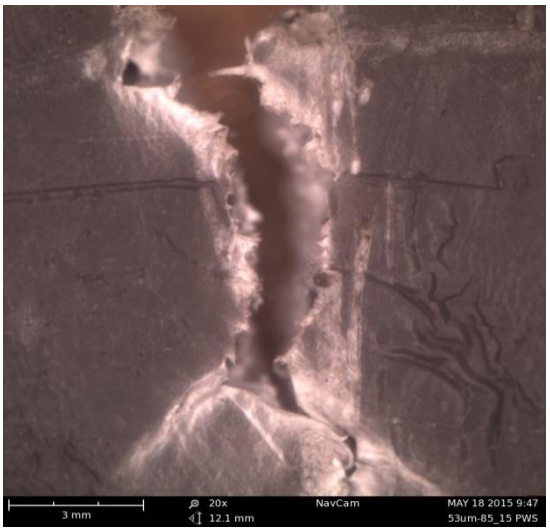


Plate III: Surface morphology of impact fractured surface for 53µm particle filled RLDPE showing the fibrous appearance indicating ductile failure mode (×20)

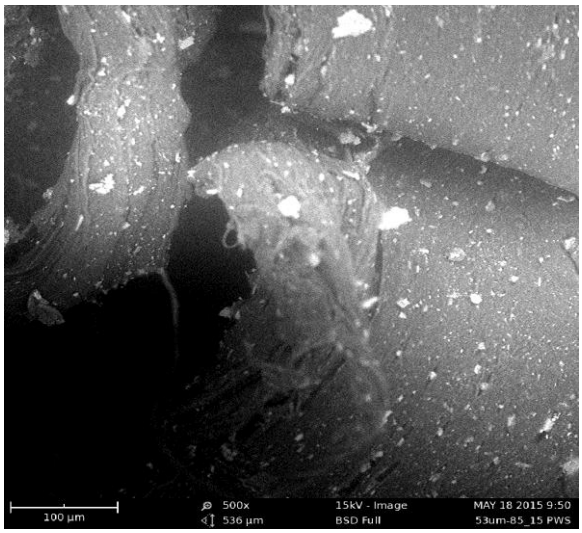


Plate IV: SEM morphology of impact fractured surface for 15wt% PWS filled RLDPE at 53µm particle size (×500)

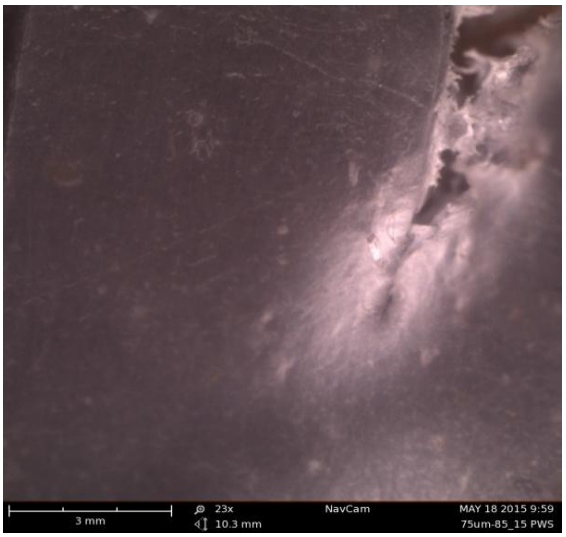


Plate V: Surface view of the impact fractured surface of composite at 75/25 PWS composition and 75µm particle size indicating less damage as compared with 53µm particle size (×23)

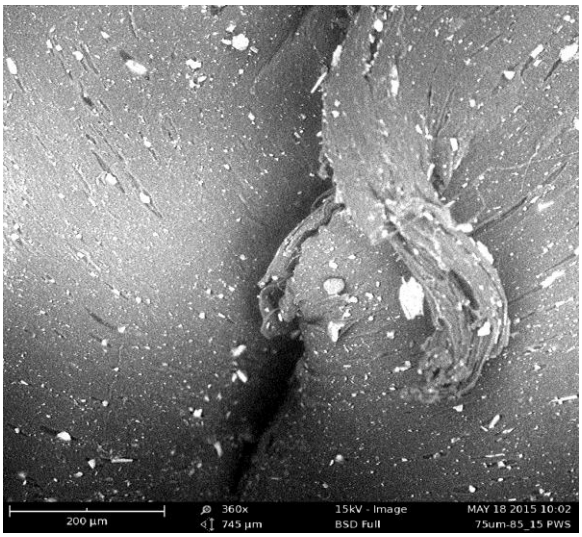


Plate VI: SEM morphology of impact fractured surface at 75µm particle size and 25wt% filler quantity indicating the uniformly filled RLDPE with periwinkle shell particles (×360)



Plate VII: Surface view of 90/10wt%PWS and 106µm particle size (×20)

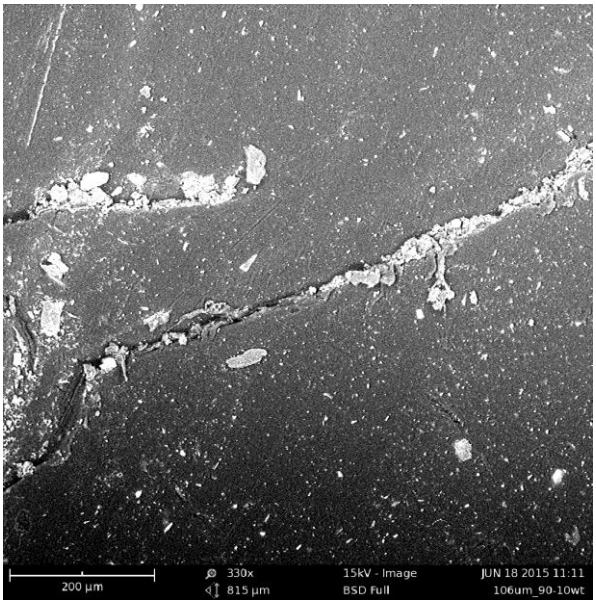


Plate VIII: SEM morphology of composite at 106µm and 90/10 wt% PWS showing the fractured surface with particles of periwinkle shells along the Fault line (×330)

4.8 Correlation between the properties and microstructure of the composite

The SEM morphology of the control sample of RLDPE are shown in Plates I and II. It can be observed from the micrographs that the RLDPE is fairly homogenous. This is due to the fact that there is no filler addition. In the absence of the filler material, deformation processes are likely to be extensive. This explains the low properties associated with the RLDPE material.

Plates III and IV show the impact fractured surfaces of the composite at 53µm particle size and 15wt% filler content, indicating the fibrous appearance of the surfaces, thus suggesting that the failure mode is more of a ductile than of brittle nature. In addition, it can be observed that the particulates are uniformly distributed, even though there are little clusters in the matrix.

Plates V and VI show the impact fractured surfaces for the 75µm/25wt% filler content which presented the best impact properties, and shows a fibrous surface also with particulates uniformly distributed in the RLDPE matrix eventhough some particles can be observed to be larger as compared with others, which may be due to coagulation of the finer particles when in contact with a fluid.

This is in agreement with the work of Atuanya*et al* (2011) who worked on potentials of using recycled low density polyethylene in wood composite boards.

Plates VII and VIII show the impact fractured surfaces for the 106µm/10wt% filler content, which presented the best impact strength in this category. As can be seen on the micrograph, there is also a uniform distribution of the particles in the reinforcement, which may have been responsible for the improvement in impact strength, as compared with other composites in this category. It can also be observed from Plate VIII that the periwinkle shells in white can be seen along the fault line, which may also have

prevented a progress in deformation processes, thus improving its energy absorption capability.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

In this research, the conversion of waste materials to a valuable product was targeted, it showed that periwinkle shells can be used as a reinforcing material on polymeric matrices either in virgin state or recycled state.

From the observations and analysis of results obtained, the following conclusions could be drawn;

i. The alkali treatment with sodium hydroxide aided the interfacial adhesion through the removal of impurities from the surface of the shells as shown with the 52% increase in tensile strength for the 106µm composite and an 87.85% increase in impact strength.

ii. The filler content produced an increase in impact strength up to 25 wt% in the 75µm particle size with a value of 1720 J/m, while for the particle size, we realized that the 75µm produced the best properties and is recommended for the subsequent production of this composite for engineering applications.

iii. The 75µm particle size presented the best impact strength at 25 wt% filler with 1720 J/m and best impact strength of the entire sizes considered, which when compared with the impact strength for the control sample which was 915.63 J/m showed improvement.

The water absorption rate for all particle sizes considered was observed to increase as time of exposure increased, but beyond 192 hrs, there seemed to be no further increase in water absorption.

iv. The soil degradability test showed that there was no weight loss for the period considered except at 15 wt% filler content for 53µm particle size with a 0.75 wt% loss.

The composite material could be used for some interior automobile applications and areas where high strength is not a critical requirement.

5.2 RECOMMENDATIONS

1.0 Finer particles such as nano-sized particles should be considered for use in recycled low density polyethylene matrix systems at different filler contents, to assess the effect this particle sizes may cause in the composite.

2.0 Other methods should be considered for the production of the composite, at filler contents higher than those used for this study (preferably at 10wt% increment), to see if the method of production of the composite would provide a better composite material as compared with the compression moulding method used for this work.

3.0 The degradability test should be carried out for a period of 6 to12 months to determine the level of degradation that may occur over an extended period of time.

4.0 The work of adhesion should be determined to better assess the effect of interfacial adhesion on the composite.

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APPENDICES

Table A.1: Tensile strength for RLDPE

% composition of PWS	Maximum load (KN)	%Elongation	U.T.S (Mpa)
100	174	47	6.25

Table A2: Summary table for mechanical properties of RLDPE

Composition (wt%) RLDPE	**Tensile Strength(Mpa)**	**Flexural Strength (Mpa)**	**Charpy Impact energy (J/m)**	**Hardness (Shore D)**
100	6.25	14.79	915.63	30

Table A3: Impact strength values for composite at 106µm particle size

Composition of PWS (Wt %)	**1st reading (J/m)**	**2nd reading (J/m)**	**3rd reading (J/m)**	**Average (J/m)**
5	1023	1075	1052.43	1051.43
10	1140	1086	1194.86	1142.86
15	900	1092.86	1092.85	1028.57
20	778.56	500	778.56	685.71
25	607.15	800	607.15	671.43

Table A4: Impact strength values for composite at 75µm particle size

Composition of PWS (Wt %)	**1st reading (J/m)**	**2nd reading (J/m)**	**3rd reading (J/m)**	**Average (J/m)**
5	980	970	900	950.00
10	1000	1400	1536	1312.50
15	1500	975	1500	1325.00
20	1200	1398	1398	1332.50
25	1160	2300	1700	1720.00

Table A5: Impact strength values for composite at 53µm particle size

Composition of PWS (Wt %)	1st reading (J/m)	2nd reading (J/m)	3rd reading (J/m)	Average (J/m)
5	906.25	800	1200	968.75
10	700	900	1400	1000.00
15	1000	1081.25	1200	1093.75
20	746.89	1000	1000	915.63
25	950	570	430	650.00

Table A.6: Tensile strength at 106µm

% composition of PWS	Maximum load (KN)	%Elongation	U.T.S (Mpa)
5	288	48	9.50
10	246	42	7.64
15	234	36	7.50
20	204	46	7.04
25	126	36	3.33

Table A.7: Tensile strength at 75µm

% composition of PWS	Maximum load (KN)	%Elongation	U.T.S (Mpa)
5	246	44	7.40
10	186	66	7.49
15	222	35	7.89
20	96	63	3.57
25	174	47	3.55

Table A.8: Tensile strength at 53µm

% composition of PWS	Maximum load (KN)	%Elongation	U.T.S (Mpa)
5	180	44	6.24
10	144	49	4.96
15	120	33	3.26
20	222	48	7.70
25	228	47	9.12

Table A9: Hardness values for composite at 106µm particle size

Composition of PWS (Wt %)	1st reading (Shores)	2nd reading (Shores)	3rd reading (Shores)	Average (Shores)
5	39	29	33	34
10	33	33	29	32
15	29	27	33	30
20	31	37	34	34
25	35	39	41	38

Table A10: Hardness values for composite at 75µm particle size

Composition of PWS (Wt %)	1st reading (Shores)	2nd reading (Shores)	3rd reading (Shores)	Average (Shores)
5	29	29	29	29
10	30	30	32	31
15	27	30	31	29
20	31	30	33	31
25	25	23	24	24

Table A11: Hardness values for composite at 53µm particle size

Composition of PWS (Wt %)	1st reading (Shores)	2nd reading (Shores)	3rd reading (Shores)	Average (Shores)
5	20	29	29	26
10	20	33	19	24
15	29	34	29	31
20	23	30	29	27
25	25	26	30	27

Table A12: Flexural strength values of RLDPE/PWS composite at differing particle sizes

	Flexural strength, Mpa		
% Composition of PWS	106µm	75µm	53µm
5	12.78	15.61	13.47
10	14.89	25.51	16.67
15	21.14	22.23	8.19
20	13.63	14.30	18.58
25	11.76	10.23	10.23

Table A 13: Water absorption values for composite at 53µm particle size for 14 days

Composition Of PWS	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	144 hrs	168 hrs	192 hrs	216 hrs	240 hrs	264 hrs	288 hrs	312 hrs	336 hrs
0	3.76	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77
5	3.91	3.91	3.91	3.91	3.92	3.92	3.92	3.93	3.93	3.93	3.93	3.93	3.93	3.93
10	4.03	4.03	4.03	4.04	4.04	4.06	4.06	4.06	4.06	4.06	4.06	4.06	4.06	4.06
15	3.20	3.20	3.20	3.21	3.22	3.23	3.23	3.23	3.24	3.24	3.24	3.24	3.24	3.24
20	4.20	4.21	4.21	4.21	4.22	4.22	4.23	4.23	4.23	4.23	4.23	4.23	4.23	4.23
25	3.27	3.30	3.30	3.30	3.31	3.31	3.31	3.31	3.31	3.31	3.31	3.31	3.31	3.31

Table A.14: Water absorption values for composite at 75µm particle size for 14 days

Composition of PWS	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	144 hrs	168 hrs	192 hrs	216 hrs	240 hrs	264 hrs	288 hrs	312 hrs	336 hrs
0	3.76	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77
5	4.46	4.46	4.46	4.46	4.47	4.48	4.48	4.48	4.48	4.48	4.48	4.48	4.48	4.48
10	3.52	3.53	3.53	3.53	3.53	3.53	3.53	3.54	3.55	3.55	3.55	3.55	3.55	3.55
15	4.01	4.01	4.01	4.01	4.01	4.03	4.03	4.03	4.03	4.03	4.03	4.03	4.03	4.03
20	4.77	4.77	4.77	4.78	4.78	4.79	4.79	4.79	4.80	4.80	4.80	4.80	4.80	4.80
25	4.84	4.88	4.88	4.88	4.88	4.94	4.95	4.96	4.96	4.96	4.96	4.96	4.96	4.96

Table A.15: Water absorption values for composite at 106µm particle size for 14 days

Composition of PWS	24 hrs	48 hrs	72 hrs	96 hrs	120 hrs	144 hrs	168 hrs	192 hrs	216 hrs	240 hrs	264 Hrs	288 hrs	312 hrs	336 hrs
0	3.76	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77	3.77
5	4.07	4.08	4.08	4.08	4.08	4.10	4.10	4.11	4.11	4.11	4.11	4.11	4.11	4.11
10	4.58	4.58	4.58	4.58	4.58	4.58	4.60	4.60	4.60	4.60	4.60	4.60	4.60	4.60
15	5.04	5.05	5.05	5.05	5.06	5.07	5.08	5.08	5.08	5.08	5.08	5.08	5.08	5.08
20	3.98	3.98	3.98	3.98	3.98	3.99	3.99	3.99	3.99	3.99	3.99	3.99	3.99	3.99
25	4.27	4.27	4.27	4.28	4.30	4.33	4.32	4.32	4.32	4.32	4.32	4.32	4.32	4.32

Table A.16: Density of RLDPE/PWS composite at differing particle sizes

% Composition of PWS	g/cm^3		
	106µm	75µm	53µm
5	0.90	0.98	1.12
10	0.82	0.92	1.05
15	0.64	0.80	0.94
20	0.52	0.62	0.86
25	0.40	0.40	0.62

Table A.17: Soil burial test for composite at 53µm after 14 days

Composition of PWS	**Mass beforeburial,M_1**	**Mass after burial, M_2**	**% weight loss**
5	2.25	2.25	0.00
10	2.14	2.14	0.00
15	2.67	2.65	0.75
20	2.62	2.62	0.00
25	3.29	3.29	0.00

Table A.18: Soil burial test for composite at 75µm after 14 days

Composition of PWS	**Mass before burial,M_1**	**Mass after burial, M_2**	**% weight loss**
5	4.95	4.95	0.00
10	2.05	2.05	0.00
15	2.95	2.95	0.00
20	2.55	2.55	0.00
25	3.11	3.11	0.00

Table A.19: Soil burial test for composite at 106µm after 14 days

Composition of PWS	**Mass before burial,M_1**	**Mass after burial, M_2**	**% weight loss**
5	2.07	2.07	0.00
10	2.03	2.03	0.00
15	4.99	4.99	0.00
20	3.90	3.90	0.00
25	2.52	2.52	0.00