PROCESSING AND CHARACTERIZATION OF A SORGHUM STEM-BASED FIBRE POLYMER COMPOSITE FOR BUILDING INSULATION APPLICATION

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Declaration by Candidate

"I hereby declare that the work submitted in the write up for consideration for the partial requirements for the degree, MEng: Mechanical and Energy Engineering, at Botswana International University of Science and Technology is my own original work and has not previously been submitted to any other institution of higher education. I further declare that all sources cited or quoted are indicated and acknowledged by means of a comprehensive list of references".

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Date: 15 September 2021

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1

Abstract

Agricultural residue-based fibrous (ARF) materials have been identified to be a commercially viable solution for energy-efficient building insulation application. ARFs have various advantages over synthetic fibers including environmentally friendly, light weight, low cost, and competent mechanical properties. Meanwhile, in many African countries including Botswana, the expertise to manufacture building insulators by using ARFs is scarce and consequently building insulation material is imported. Furthermore, undesirable characteristics of hydrophilic ARF such as poor mechanical properties, water absorption, poor thermal stability, and poor adhesion with hydrophobic polymers have posed challenges to the development and application of agricultural residue-based fibers reinforced polymer composites (ARFRPCs). Hence, the need to engage a critical literature review to identify appropriate surface medication methods, materials, and processing parameters to manufacture ARFRPCs for building insulation. This thesis investigates the production of building insulation composites by using ARF derived from sorghum stalk incorporated into polymeric matrices to develop local expertise and reduce importation. To achieve this goal, samples of ARF extracted via water retting technique and then treated for five and ten days using thermo-alkali and thermo-laccase surface modification techniques. Samples of ARRFPC from both treatments were manufactured by compounding ARF with recycled low-density polyethylene (rLDPE) and later on processed via compression moulding technique. The samples were then dried and cured in a conditioning room for 10 days. Analytical techniques such as water absorption, thermal conductivity tests, thermo-gravimetric analysis (TGA), Scanning Electron Microscope(SEM), X-Ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR) were engaged to provide insight into the thermal degradation, microstructural features, interfacial adhesion mechanisms as well as mechanical and chemical characteristics of the

ARFRPC modified with thermo-alkali and thermo-laccase treatments at different time durations. Both thermo-alkali and thermo-laccase treated ARFRPCs were found to be stiff and well compacted. It was established that modified fibres improved the functional performance of the composites in comparison to untreated composites. Moreover, thermo-alkali reinforced ARFRPCs exhibited the most desirable mechanical characteristics with a tensile strength of 28.57 MPa, improved microstructure/interfacial adhesion, and reduction in porosity between the filler/matrix relative to untreated and thermo-laccase treated samples. The thermo-alkali treatment increased the interfacial bonding between the plastic and the fibrous filler for the composites as observed in SEM micrographs of the fractured surfaces of ARFRPCs. The thermal properties revealed that the thermal conductivity values ranging in between 0.223-030 W/m.K are higher than other commercially available insulation materials, such as glass fiber blanket (0.039 W/m.K), cork board (0.043 W/m.K) and expanded polystyrene (0.029 W/m.K) to mention few, but they are comparable to those obtained from ARFs reinforced polymer boards. Therefore, a conclusion is made that the composite materials produced in this study require extrusion moulding which is better suited to produce foamy insulation material, and a provisional application given the quality properties of the composite boards produced is wall and floor panelling applications in buildings.

Keywords: Agricultural residue-based fibers (ARF); Building insulation; Polymer composite; Quality characteristics; Surface modification.

Table of Contents

Declaration by Candidate ii

Acknowledgements iii

Abstract iv

1 List of Figures x

2 List of Tables xii

3 List of Symbols and Abbreviations xiii

1 CHAPTER 1: INTRODUCTION 1

1.1 Chapter Summary 1

1.2 Background of the study 1

1.3 Problem Statement 5

1.4 Objectives 6

1.5 Scope and Limitation of the Work 6

1.6 Dissertation Thesis Outline 8

1.7 Conclusion 9

2 CHAPTER 2: LITERATURE REVIEW 10

2.1 Chapter Summary: 10

2.1.1 The nature of fibers derived from agricultural waste residues. 10

2.2 Extraction of Fibers derived from agricultural residues. 15

2.2.1 Water Retting 16

2.2.2 Dew Retting 17

2.2.3 Mechanical extraction 19

2.3 Processing challenges in polymer composites reinforced with ARFs derived from agricultural waste residues. 21

2.3.1 Hydrophilicity of Natural Fibres in the Polymer Matrix 22

2.3.2 Thermal Stability 23

2.3.3 Durability and degradation 24
2.4 Approaches for improving thermal insulation properties of polymer composites reinforced with ARFs derived from agricultural waste residues. 26
2.4.1 Chemical modification of natural fibres 26
2.4.2 Biological Modification of Natural Fibres 31
2.4.3 Hybrid Treatments 32
2.5 Polymer matrices 35
2.6 Effects of materials parameters on the functional performance of agricultural waste-based fibers reinforced polymer composites (ARFRPCs) 38
2.6.1 Particle Size and Distribution 38
2.6.2 Fibre content 39
2.6.3 Processing of Fiber Reinforced Thermoset Composites 41
2.6.3.1 Hand Lay Up Method 42
2.6.3.2 Injection Molding 43
2.6.3.3 Compression Molding 44
2.6.3.4 Extrusion moulding 45
2.7 Behaviour and characterization of thermal insulator developed from ARFRPCs. 47
2.7.1 Thermal characterization 47
2.7.2 Thermal Characterisation 49
2.8 Conclusion 50
3 CHAPTER 3: METHODOLOGY 51
3.1 Chapter Summary 51
3.2 Materials 51
3.2.1 Sorghum stalk Residues 51
3.2.2 Recycled Low Density Polyethylene (rLDPE) 53
3.3 Water Retting 54

3.4 Surface Modification of Natural Fibres 55
3.4.1 Thermo-alkali treatment method 56
3.4.2 Thermo-laccase Treatment 57
3.5 Pulverisation of the Natural Fibre Samples 58
3.6 Compounding 60
3.7 Compression Moulding 61
3.8 Characterisation of composites 64
3.8.1 Mechanical Properties 65
3.8.2 Water absorption studies 67
3.8.3 Thermal Properties 68
3.8.4 Physical and Chemical Characterisation 71
3.9 Conclusions 73
4 CHAPTER 4: RESULTS AND DISCUSSION 75
4.1 Chapter Summary 75
4.2 Density 75
4.3 Moisture Absorption 76
4.4 Thermal Conductivity 79
4.5 Thermogravimetric Analysis 82
4.6 Fourier transform infrared Analysis 85
4.7 X-Ray Diffraction Analysis 86
4.8 Tensile Testing 89
4.9 Scanning Electron Microscope Analysis 93
4.10 Conclusion 95
5 CONCLUSION AND RECOMMENDATIONS 96
5.1 Chapter Summary 96
5.2 Conclusions 96

5.3 Recommendations for Future Study 98

6 Appendices 99

7 References 100

List of Figures

Figure 2-1: Classification of Agriculture Waste-Based Fibers 11
Figure 2-2: Structure of biofibre [34] 12
Figure 2-3: Water Retting at (a) 0.5 days, 3.5 days, and 4.5 days (b) 7 days and 9 days [77] 16
Figure 2-4: Experimental setup of Dew Retting [72] 18
Figure 2-5: Schematic diagram of hybrid yarn manufacturing through DREF III spinning[73] . 19
Figure 2-6: Sketch of simple fiber decorticator[73] 19
Figure 2-7: SEM micrographs of WPCs reinforced with wood fiber of varying contents from 15 wt.% to 55 wt.% [84] 23
Figure 2-8: Effect of water on fibre/matrix interface [73] 25
Figure 2-9: SEM micrographs showing the surface morphologies of untreated and sodium hydroxide treated fibers [94]**.** 28
Figure 2-10: Hand Lay Up Method [73] 42
Figure 2-11: Injection Moulding Process [153] 43
Figure 2-12: Compression Molding [153] 45
Figure 2-13: Extrusion Moulding Model [153] 46
Figure 2-14: (a) "Sea-island" in low fillers loading; (b) Thermal conduction paths in high fillers loading; (c) Percolation phenomenon; (d) Thermoelastic coefficient theory [166]. 49
Figure 3-1: Sorghum Plant Stalks 52
Figure 3-2: Process flow for extracting fibers from Sorghum plant fibers 54
Figure 3-3: Water Retting of sorghum stalks 55
Figure 3-4: Neutralising Kit 56
Figure 3-5: (a) Termite Mounds (b) Laccase Enzyme 58
Figure 3-6: Fritsch Planetary Mill Pulverisette 5 59
Figure 3-7:The round vibratory sieves (b) the sieves with Sorghum AR particle fiber 59
Figure 3-8: (a) Compounding machine (b) Fiber particles and LDPE granules inside machine 61
Figure 3-9: (a) Compression moulding mould (b) Removal of extra moisture 62

Figure 3-10: (a) Composite board after pressing (b) Cooling and compression of composite board. 62
Figure 3-11: Composite board 63
Figure 3-12: ARFRPCs Analysis Samples cutting scheme 63
Figure 3-13: (a) Cutting boards into sample sizes (b) Boards stacked for conditioning. 64
Figure 3-14: Tensile testing sample 65
Figure 3-15 Jeol JSM-7100F Thermal field emission Scanning Electron Microscope 66
Figure 3-16 Q 150T ES 67
Figure 3-17: Linear heat conduction unit H112A 68
Figure 3-18: (a) Thermal conductivity sample (b) Measuring sample thickness using veneer callipers. 69
Figure 3-19: Thermal Conductivity Experimental setup 69
Figure 3-20 TGA 701 71
Figure 3-21: D8 Advance Diffractometer 73
Figure 4-1: Composite Samples Density Graph 76
Figure 4-2: ARFRPCs Water Absorption Line Graph 77
Figure 4-3: ARFRPCs Thermal Conductivity (W/m.K) Line graph 79
Figure 4-4: Comparison of Insulation Materials Thermal Conductivity Values 81
Figure 4-5: ARFRPCs Thermograms 83
Figure 4-6: IR Spectra of Treated and Untreated ARFRPCs 86
Figure 4-7: XRD Patterns of the treated and Untreated ARFRPCS 87
Figure 4-8: Stress-Strain Curves for ARFRPCs 90
Figure 4-9: ARFRPCs Samples Tensile Strength 91
Figure 4-10: ARFRPCs Samples Elongation at break 92
Figure 4-11: ARFRCPS SEM analysis (a) Untreated sample ,U (b) Thermo-laccase treatment for 5 days (c) Thermo-laccase treatment for 10 days (d) Thermo-alkali treatment for 5 days (e) Thermo-alkali treatment for 10 days 93
Figure 6-1: Methodology Workflow 99

List of Tables

Table 2-1: Sugar Cane Bagasse cell wall constituents weight composition 13

Table 2-2: Comparative analysis of various extraction techniques for ARF 21

Table 2-3: Comparison of ARFs Treatment Methods..... 34

Table 2-4: Thermal Conductivity and Water absorption coefficient values of PALF reinforced latex composites [150]. 40

Table 3-1: Mechanical properties and processing data of Recycled LDPE – from the supplier's technical data sheet 53

Table 3-2: Surface Modification Treatment Formulations..... 58

Table 4-1: ARFRPCs Water Absorption..... 78

Table 4-2: ARFRPCs Thermal Conductivity , k (W/m.K) 80

Table 4-3: ARFRPCs TGA Analysis Results..... 84

Table 4-4: Crystallinity index (CI) and Crystalline size (CS) values of ARFRPCs...... 88

List of Symbols and Abbreviations

Δx_{int} = Composite sample thickness

A10- 10 days Thermo-alkali treated composite

A5- 5 days Thermo-alkali treated composite

AAP – Acetyl acetone peroxide

A_{int} = Cross section are of composite sample

ARF- Agriculture residue fibers

ARFRPC- Agriculture residue-based reinforced polymer composites

BMC –Bulk molding composites

BPO – Benzoyl peroxide

C.I- Crystallinity Index

C.S- crystal size

d – Fiber diameter

E_f – Modulus of fiber

E_m – modulus of matrix

FTIR- Fourier Transform Spectroscopy

I_{200} - maximum intensity of the (200) lattice diffraction

I_{am} - intensity diffraction of the amorphous band

k = Thermal Conductivity

L10- 10 days Thermo-laccase treated composite

L5- 5 days Thermo-laccase treated composite

m_i - weight of the dry sample

m_t -weight of the sample at time t

NaOH –Sodium hydroxide

PMCs – Polymer matrix composites

PVAc – Polyvinyl acetate

rLDPE – Recycled Low density Polyethylene

SEM – Scanning electron microscopy

SMC – Sheet molding composites

$T_{coldface}$ = Temperature difference across cold surface

T_{deg} – Degradation Temperature

TGA – Thermo gravimetric analysis

$T_{hotface}$ = Temperature difference across hot surface

T_{onset} – Onset Thermal Degradation Temperature

TS – Tensile strength

U- Untreated Composite

V – Volume

V_{crit} – Critical volume fraction of fiber

V_f –Fiber volume fraction

V_m – Volume fraction of matrix

V_{min} – Minimum value of fiber volume fraction

W – Mass

XRD- X-Ray Diffraction

ρ– Density of the composites

σ_f – Fiber strength

σ_m – Matrix strength

CHAPTER 1: INTRODUCTION

1.1 Chapter Summary

A brief history about the origin of natural fibres and what has led to the adoption of natural fibre polymer composites for building insulation application are presented. Also explored are the reasons leading to increased interest in natural fibre in the academic research and the industry at large. The potential of natural fibre polymer composites in Botswana is discussed to show relevance of the technology locally. A background of building insulation in Botswana, its current state and potential, is explored. This chapter also introduces the objectives, the scope, and the outline of the dissertation.

1.2 Background of the study

In recent decades, the building industry has become increasingly focused on environmentally friendly and energy efficient structures [1]. The building industry creates various job possibilities and aids in the development of a country's infrastructure. However, the building industry is responsible for forty percent of greenhouse gas (GHG) emissions and a third of carbon dioxide emissions [2], [3]. Moreover, according to international green building experts, the building and construction sectors will emit more than 40 billion tons of carbon dioxide by year 2030 [4]. Numerous international organisations, authorities, and research groups now believe that energy efficient buildings are necessary to alleviate pollution and environmental challenges [5]. As a result, several attempts to develop sustainable building materials have lately been recommended. Investigating green building materials and eco-products, as discussed in this thesis, is an effective

way to achieve this goal. For a country's development to be sustainable, it must use construction materials that have the least amount of environmental impact. Majority of energy consumption in buildings is caused a number of factors such as heating, ventilation and air conditioning system (HVAC) [6], window resistance and door thermal insulation, losses of thermal bridge, wall envelope, and thermal performance [7]. Thermal insulation materials can effectively minimize heat losses to the environment while also providing a pleasant indoor atmosphere. Alternative strategies for reducing annual energy usage in buildings, which have been demonstrated in preliminary investigations, include installation of thermal insulation materials in the walls and roofs. Wall insulation is critical for improving a building's thermal comfort while also lowering its cooling load [8].

The utilisation of natural fibers, as fillers in manufacturing polymer composites for thermal insulation application in buildings, has gained a lot of interest in recent decades. Natural fiber is one of the most environmentally friendly materials available, with superior properties to synthetic fiber [9]. Natural fibers are fibers that are not synthetic or man-made, obtained from plants and animals [10]. Natural fiber reinforced polymer composites (NFRPCs) have been used in the building and construction industry for both structural (load bearing walls, staircases, roof systems, and sub-flooring) as well as non-structural (outdoor decking, window and door frames, ceiling tiles, and furniture) purposes [11]. Non-structural applications, on the other hand, do not necessitate the same high mechanical qualities as structural applications [12]. Composite panelling boards for building partition walls are also made from wood-based composites. NFRPC panels are cost-effective because natural fibers are readily available as wastes [13], and they are relatively robust and lightweight [14]. Belonging to the natural fibers category is agricultural residue-based

fibers (ARF). They are the remains from the agricultural field after the crop/product is harvested from the agricultural plants [15]. Residues include bagasse, stalk, seeds, leaves, roots etc. Approximately 20% of agricultural waste is currently used for producing biofuels, paper, fertilizers, and animal feed, while the rest is either burned, absorbed into the soil, or used as mulch for the following crop after harvest [16]. Nonetheless, waste buried into the soil decompose slowly and may host harmful bacteria and illnesses, whilst burning the agricultural residues is unsustainable and environmentally unfriendly due to widespread atmospheric pollution [17], which includes greenhouse gas emissions and smoke. Waste or Residue-to-energy is one of the recommended solutions for managing waste, and industry innovations have presented a variety of eco-friendly approaches to reduce the carbon footprint caused by landfills, with variable degrees of success [18]. The process of constructing the structure - operating heavy machinery, moving materials - as well as the process of living in the building, i.e. operation and maintenance - all place a strain on the environment through energy expenditures [19]. Waste-to-energy facilities are one option to supplement energy needs while reducing energy waste during a building's life cycle [18].

Bagasse, sorghum, rice fibers and banana leaves are different examples of agricultural waste-based fibers (ARF) that could be used as reinforcements in polymeric composites [20], [21]. Various researchers have taken steps to study the economic and social feasibility of applications for agricultural residues such as residues from the rice production cycle used for ARFRPCs production, as discussed by Goodman [19]. Bagasse fibers derived from sugar cane were initially used to make composite panels in the United States in the 1920s [22]. In the Philippines, Jamaica and Ghana, construction panels and roofing sheets manufactured from bagasse fibre reinforced phenolic composites are presently used in homes [9]. Due to their low density, cereal straws are

also used in the construction of panels, producing panels which are more durable and resistant to earthquakes [23]. Rice husks or ash are also utilized in the manufacture of fiber cement blocks and other cement products. Rice husks are used in construction items to improve acoustic and thermal qualities [24]. Ismail *et al*.[25] also explored the potential use of rice straw for the production of biofuels and a variety of other items. Several agriculture waste-derived products have been developed in the past few years, such as polymeric composite resins and polymeric timber as a natural wood alternative, which are often made by blending crushed agriculture waste-based fibers (ARFs) and polymer, either solid or liquid. Ramlee *et al* [17] reviewed the potential of sugarcane bagasse for the use of building application and concluded that the agriculture waste based fibers are effective thermal insulator, that are renewable, safe and economically sustainable because of the abundantly available resources. As a result of the excellent characteristics and improved advantages of natural fiber over synthetic fibers in terms of its comparatively low weight, low cost, minor damage to processing equipment, good relative mechanical properties such as tensile modulus and flexural modulus, and an increased surface finish of moulded parts composite, a natural fiber reinforced polymer matrix has received much interest in a variety of applications [26], [27]. By incorporating a strong and light-weight agricultural waste-based fiber into a polymer (thermoplastic or thermoset), ARFRPCs characterised with high specific stiffness and strength can be manufactured [28]. The tensile strength of ARFRPC is dependent on the properties of polymer matrix, whereas its elastic modulus is dependent on the fibre characteristics [29]. The composition of ARFs approbates moisture absorption from the environment, resulting in poor fiber-polymer bindings [30]. Furthermore, considering that the chemical structures of both the fibers and the matrix are dissimilar, couplings between agricultural waste based fiber and polymer are considered a challenge [31]. Interface bonding that exists between the matrix and the filler is significant in

transmission of stress from the matrix into the fillers across the interface [32]. The interface bonding between the matrix and the ARF can be enhanced by utilizing coupling agents [33]. The coupling agents impart a bond between the ARF and the matrix via the enhanced compatibility and forming a mechanical/chemical bonding.

In addition, previous research has shown that recycled polymers have similar mechanical qualities to virgin polymers but are significantly less expensive. The mechanical behavior of recycled high-density polyethylene (rHDPE) from post-consumer milk jugs were found to be similar to those of virgin resin, allowing it to be employed in a variety of applications [34]. rHDPE pellets and granules, on the other hand, are 31-34 percent cheaper than virgin HDPE (vHDPE) [35]. As a result, if recycled polymers are used to make ARFRPCs without sacrificing the quality properties, ARFRPC products will have a cost-competitive edge in the industry.

1.3 Problem Statement

The lack of good-quality, affordable building insulation for low- and middle-income people is a major issue for Botswana as the currently used and available insulation materials are imported from outside Botswana. Furthermore, the building and construction industry is currently focused on energy efficiency to reduce the environmental impact of buildings by adopting environmentally friendly materials and manufacturing processes, necessitating the development of low-cost, long-lasting building insulation materials. The above problem will be solved by developing high-quality building insulation materials using ecologically friendly sorghum stalks (as agro-waste is available in generous quantities locally) as a ARF's source to produce ARFRPCs has not been widely exploited yet for reinforcing agent in bio composites.

1.4 Objectives

The overall objective of this study is to produce, analyse and characterise the mechanical, chemical, thermal, physical, and microstructural properties of an agriculture waste-based fibre reinforced polymer composites (ARFRPCs) produced from sorghum plant agricultural residues derived from Botswana for building insulation application. To achieve the overall objective, specific objectives are defined as follows:

1. To identify appropriate (i) environmentally friendly extraction and surface modification techniques for preparing sorghum stalk fibers, (ii) materials parameters (reinforcement particle size, polymer matrix) and (iii) compounding technique and parameters for manufacturing ARFRPCs derived from sorghum stalk fibers for building insulation application.

2. To manufacture ARFRPCs derived from sorghum stalk fibers based on the selected appropriate environmentally friendly extraction and surface modification technique/parameters, materials parameters, and compounding technique/parameters.

3. To analyse the physical, chemical, mechanical, thermal, and microstructural properties of the ARFRPCs produced in (2) above with a view to identifying appropriate environmentally friendly surface modification technique/parameters to produce building insulators.

1.5 Scope and Limitation of the Work

This thesis focuses on the development of new ARFRPCs reinforced with sorghum stalk fibers (an agro-based waste). The composite materials were characterised with low environmental impact, high compostability and suitable for building insulation. The analysis of the available agricultural

residues is limited to Botswana, although the results and findings presented in this thesis are valid for other regions of the world where similar raw materials are available. To assist manufacturers in selecting appropriate processing and materials parameters to produce ARFRPCs for building insulation application, this study reviews relevant literature from 2000s to 2021. To achieve the goal of this study, various surface modification techniques, manufacturing techniques/parameters and material parameters were identified, discussed and their likely influences on the quality characteristics of ARFRPCs were compared.

The thesis is mainly based on experimental laboratory work which is focused on the characterization, evaluation, and comparison of the performance of the experimental materials proposed. The aspects investigated in the characterization are related to physical properties (mainly geometry and density), thermal characteristics (thermal conductivity and stability), mechanical properties (bending and tensile strength), microstructure, chemical analysis, and hygroscopic properties (moisture content and absorption). It is beyond the scope of this thesis to analyse other relevant aspects such as the fire retardancy, mould growth resistance, and the aggregate or the life cycle analysis of the proposed composites.

In buildings, insulation materials are integrated into complex construction systems, the configuration of which determines the service performance of the materials; thus, it is crucial not only to study the performance of such materials individually, but also as part of the specific construction systems. The mechanical aspects concerning the applicability of the material in such a system as well as the construction details necessary for its implementation are beyond the scope of this thesis. However, parametric studies were conducted to identify the influences of several key parameters on the structural of composites fiber treatment. The fibers matrix interaction by

Scanning Electron Microscopy (SEM) was investigated. Moreover, the effects of water absorption on the suitability of sorghum fiber rLDPE composites materials were studied.

1.6 Dissertation Thesis Outline

The following text is organized into five Chapters: (1) introduction, objectives, and thesis scope; (2) literature review; (3) materials and methodology; (4) results and analysis (5) conclusions and recommendations for further research.

The introduction, a broad overview of the thesis, and the state of the art are presented in chapter 1. The study's problem and scope, which includes the features of the existing building materials, and the state of the insulation materials, are presented in Chapter 1. Published literature on natural fiber composites is reviewed and critiqued in Chapter 2. The origin and classification of ARFs are discussed as well as their structure and chemical composition. The most prevalent composite production processes will be reviewed as well as well as the issues and challenges experienced when producing ARFRPCs. Chapter 2 also covers the methodology and standards used to investigate surface modification techniques including chemical, biological, and physical methods employed. The experimental setups and procedures employed in this study are explained in Chapter 3. In Chapter 3, methods, equations, and standards used to study Physical, thermal, and hygroscopic qualities are outlined. The agriculture residues employed as raw materials in this thesis is sorghum stalk residues whilst the polymer used is recycled low density Polyethylene (rLDPE). There are three stages to the experimental work. Water retting and treatment of sorghum residues fibers is the initial step. Furthermore, two different types of treatment , thermo-alkali and thermo biological are used as fiber surface modification techniques in this study. The second stage is the composite fabrication stage which involves compounding and producing the composite

materials via compression moulding. The last stage analyses the physical, mechanical, thermal, and chemical characteristics of ARFRPCs, such as strength, Young's modulus, interfacial adhesion, thermal conductivity, and thermal stability.

The main focus of Chapter 4 is on the physical, chemical, mechanical, and thermal properties of ARFRPCs. Experiments were conducted to determine the density, water absorption, tensile strength, and Young's modulus of the ARFRPCs. The effects of thermo chemical and thermo biological treatment on composite properties were examined and contrasted to the untreated composite sample using tensile test, TGA, FTIR, XRD and SEM analysis. Finally, in Chapter 5, conclusions are taken from the above-mentioned work. The recommended composite's technical feasibility as a building insulation material is assessed by evaluating its various properties. Further study possibilities are also discussed.

1.7 Conclusion

In closing, the durability and endurance of ARFPCs developed from the local natural fiber of interest, sorghum, are still not completely grasped, and the influencing factors are unknown, providing ample research opportunities for formulation and processing optimization. The performance of ARFPCs can be improved by experimenting with a variety of composite compositions and analytical processes. The dimensional stability, durability, mechanical, and thermal properties of fibers and composites will be studied.

CHAPTER 2: LITERATURE REVIEW

2.1 Chapter Summary:

This chapter presents a review of relevant literature on ARF's, its surface modification and challenges associated with its processing. The first section deals with the definition of ARF's, its classification, structures, and properties as they affect their performance as thermal insulators when incorporated into agriculture waste-based fibres reinforced polymer composites (ARFRPCs). The processes of extracting ARF's are reviewed and the most suitable fiber extraction method is determined. Challenges involved in extraction and surface modification of ARF's are discussed with a view to proffering solutions to the treatment of ARF's. This chapter also dwells on how various surface modification methods and treatment parameters influence the quality characteristics of ARFRPC's such as the microstructure, surface morphology, chemical, thermal, mechanical, and physical properties.

2.1.1 The nature of fibers derived from agricultural waste residues.

Plant fibers and animal fibers are two types of natural fibers that can be categorized under natural fibers based on their origin [36]. As mentioned in the Section 1.2 ARFs which belong to natural fiber category are further divided into various classes as leaf, fruit, stalk, and root fibers as shown in Figure 2-1 [37]. Leaf fibers are usually seen running lengthwise across the leaves of most monocotyledonous plants [38]. Banana, agave, pines, and aloe are all examples of leaf fibers [21]. These fibers are rough in nature compared to other ARFs and are commonly utilised in the preparation of boards, rugs and mats, among other things [39].

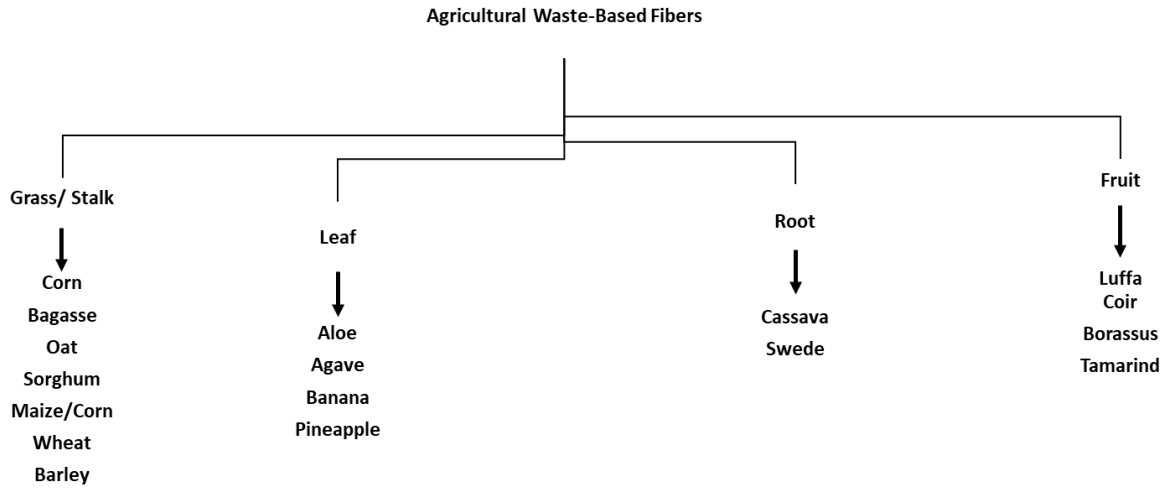

Figure 2-1: Classification of Agriculture Waste-Based Fibers

Grass ARFs are typically obtained from monocotyledonous plants' stems [40]. Bamboo, *Saccaharum cilliare*, esparto, sugar cane, and sabai grass are all examples of grass ARFs [41], [42]. Similar to grass fibers, we have stalk fibers which are a common bye-product of cereal farming and are manufactured in large quantities all over the world [43]. Stalk ARFs are often regarded as residue, and approximately half of the stalk is disposed of by burning on farming fields [44]. Attempts to utilise stalk fibers as filler materials in polymer composites have recently been made. Mulinari *et al* [45] researched the properties of sugarcane bagasse ARFs and the influence of surface modification treatments for the production of polypropylene composite materials. Their findings revealed that pre-treated bagasse was efficient in improving the mechanical properties of the reinforced polypropylene composite material. It also transformed the brittle cement composite as its tensile modulus of elasticity showed an improvement of 61% compared to the untreated fibers. The outcome of their study proved the feasibility of the composite industrial applications

as the treatment removed the fiber's essential constituents namely: cellulose, hemicellulose, and lignin which make up the structure of cellulosic fibers [46].

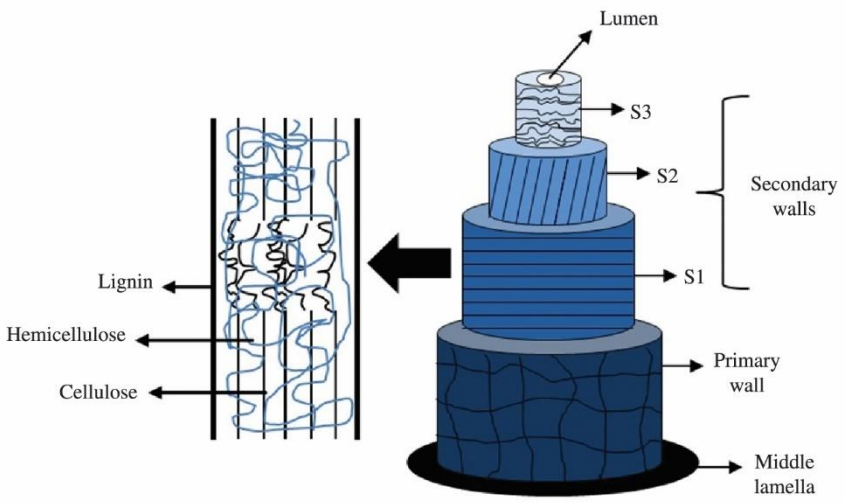

Figure 2-2: Structure of biofibre [34]

Agriculture residue fibres possess a composite-like build and entail rigid crystalline cellulosic microfibrils as reinforcement apprehended together through lignin, hemicelluloses and waxes (Figure 2-2) [47], [48], [49]. The typical chemical makeup and weight composition of the amorphous constituents of ARFs found from agricultural crop sources is presented in Table 2-1. The physical characteristics of cellulosic fibres are chiefly a result of factors such as the cellulose content or crystallinity [50], [51]. The fist outer layer is the primary cell wall which shelters the secondary cell wall which consists of three layers (S1, S2, and S3). ARF microfibril orientation differs from one another and lies in the series 5 to 10 degree for bast fibers, whereas the microfibril angle in wood fibres are from 10 to 60 degrees [52]. It was acknowledged that the three layers have dissimilar microfibril orientations leading to different structures and roles they play in the cell wall [53]. The secondary cell wall is made up of cellulose and hemicelluloses. Cellulose is the key structural element that delivers strength and steadiness to the plant cell wall [54]. Cellulose

occurs naturally as a semi- crystalline macromolecule which displays non-homogeneity molecular weight in the body [55]. The composition of bagasse fiber as an example of ARFs is shown in Table 2-1. Fibres from wood celluloses have been found to possess a lesser molecular weight in comparison to agricultural residue fibre sources [56]. Furthermore, agriculture residues fiber's cellulose is vastly crystalline and comprises mostly homogeneous cellulose with a high molecular weight, while wood cellulose have a less crystalline and amorphous cellulose [55], hence the increased interest to study ARFs for the production of polymer composite as shown by Labidi *et al* who studied a comparison between alfa fibers and wood fibers [57].

Table 2-1: Sugar Cane Bagasse cell wall constituents weight composition

Cellulose content (%)	Hemicellulose (%)	Lignin (%)	Ash (%)	Extractive (%)	Reference
51.00	30.10	12.50	2.00	–	[58]
41.00	25.08	20.10	1.54	5.63	[59]
44.10	29.01	24.03	1.43	–	[60]
33.00–36.00	28.00–30.00	–	–	–	[61]

The research proved that the polymer composite reinforced with alfa fiber's young modulus was superior with a value of approximately 4 GPa compared to the wood fiber-based composite with approximately 3 GPa. The second constituent of ARFs is hemicellulose which has a higher moisture absorption capacity and is easily biodegradable [62]. The presence of high moisture

content in turn influences the thermal degradation and stability of reinforced polymer composites. Thermal analysis of date palm fiber reinforced epoxy resin composite revealed, that as the hemicellulose content increases water absorption capacity of the composite correspondingly increase resulting in poor thermal stability [63]. In contrast to hemicellulose which exist in many life forms, lignin is only found in vascular plants [64]. Lignin is responsible for the water transportation, stiffness and resistance to microbial degradation in vascular plant cell walls [65], [66].

Naturally, lignin is found on the outer surface of plant fibers covering the cellulose and hemicellulose components (Figure 2-2) [34]. Similar to hemicellulose, removal of lignin has a critical positive impact on composite materials [67] as it aids formation of high density composite materials with high crystallinity and improved mechanical characteristics such as Young's modulus [27]. In addition to ARFs constituents, fiber size, structure, flaws, crystallinity, variability, and cost are all important physical qualities to consider when selecting ARFs for use in composites [10]. When choosing a fiber for composites reinforcement, mechanical qualities are even more crucial [68]. It is critical to use strong reinforcing fibers when making composite materials. However, fiber strength is not the only component that contributes to composites' strength; strong fiber-matrix bonding, appropriate fiber orientation, and adequate fiber dispersion are all required [69]. Sorghum stalk residue fibers were selected for this study because it is an annually grown crops in Botswana and has the potential to be used as reinforcing fiber in polymer matrix. The ARF used in polymer composite applications is determined by the availability of the fiber in the area as well as the final composite qualities required for the building insulation application. Furthermore, in comparison to conventionally used softwood, ARFs generally contain a less lignin content, a greater cellulose content, and a low hemicellulose concentration [10] and

will produce an excellent fiber-matrix adhesion resulting in better thermal and mechanical properties compared to wood. The length of sorghum stalk fibers is shorter towards the bottom and longer at the top of the stalk (Rowell et al. 1999). Similar to kenaf fibers, the transition from bottom to top is not smooth, but rather S-shaped [70]. Sorghum stalk fiber length increases early in the plant cycle and then decreases as the plants develop [71]. ARFs geometry and characteristics are influenced by a variety of factors, including species, growth conditions, cambium age, harvesting, defibration, and processing [72]. This variety makes analyzing the influence of ARFs and their interfaces on the composite material's mechanical properties more complex. These challenges necessitate the development of new methodologies for assessing the ARFs influence as reinforcements of polymer composite. Various parameters, especially chemical composition, and intracellular fiber structure, ultimately affect the mechanical properties of ARFRPCs, and these parameters vary between various portions of a plant as well as sorghum plants variety.

In addition to the fiber structure and its constituents, the final properties of a reinforced composite are affected by the way fibers are extracted as discussed in the next section.

2.2 Extraction of Fibers derived from agricultural residues.

In addition to its structure and composition, the method used for extraction of AFR could also affect the quality of fibers and composites made from it. Therefore, the use of appropriate methods of extraction for AFR is considered critical. AFR from different sources require different approaches of extraction [73]. For instance, cotton fibers are extracted from seeds via mechanical extraction while sugarcane bagasse fibers are obtained from the stalk through either water retting or mechanical extraction. Dew retting, water retting and mechanical extraction techniques [74]

will be considered in this section with a view to identifying the most suitable method of extraction in terms of process economics, extraction time and quality characteristics of ARFs. These techniques are considered in this section because the methods use little to no chemicals in addition to being cheap, affordable, and accessible for research in a middle-income country such as Botswana.

2.2.1 Water Retting

Water retting involves submerging stems from agricultural residue in water [75] as shown in Fig. 2-3. Water will be absorbed by the core section of the stem resulting in swelling of the inner cells, thereby, causing the surface layer of the plant to rupture [76].

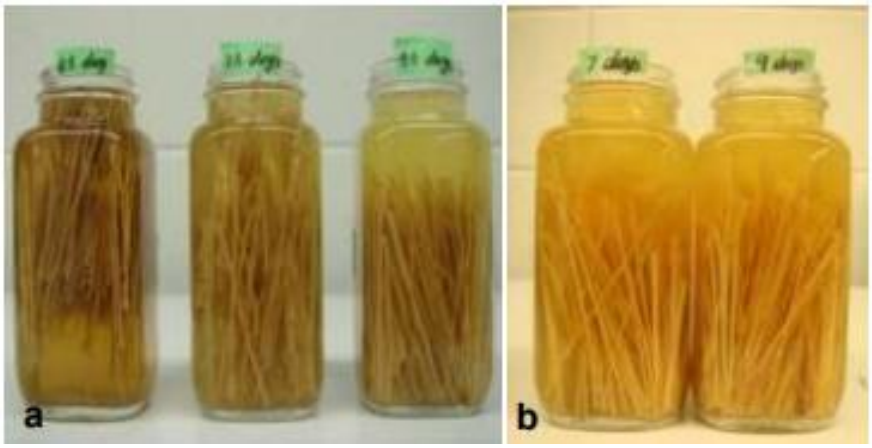

Figure 2-3: Water Retting at (a) 0.5 days, 3.5 days, and 4.5 days (b) 7 days and 9 days [77]

This process requires between 10 to 14 days for optimum fiber extraction to ensure quality characteristics such as density, moisture absorption, physical appearance etc. as well as to ensure partial degradation of hemicellulose, lignin and impurities [76] [78]. The presence of bacteria and moisture in the stem during the retting process causes significant sections of the cellular tissues and adhesive substances that cover the fibers to be broken down, allowing degradation of the fibers as reported by Rozyanty *et al.*, [79]. Bodies of water, sluggish streams, as well as rivers can all be

used for natural water retting. As pointed out, there exists bacteria that may be beneficial or harmful in water retting method [80], therefore, understanding the dynamics and types of the microflora found in water retting necessitates an in depth study of water retting on a microbiological level. Water retting uses significant amount of water therefore it may be argued that it serves as a wastage of water where clean water is used or contaminates water where the retting process is carried out in natural bodies of water such as rivers or lakes.

2.2.2 Dew Retting

Dew retting is a process where the stems of the agricultural residue are trimmed and uniformly dispersed throughout the plains, where bacteria, sunlight, ambient air, including dew enable the cellular tissues and binding agents that envelope the fibers to disintegrate. In areas with strong night dew and hot days, dew retting is favoured. This method is both cost-effective and commonly utilised in the development of blast fibers as seen in Figure 2-3 where dew retting was used to extract hemp fibers [81].

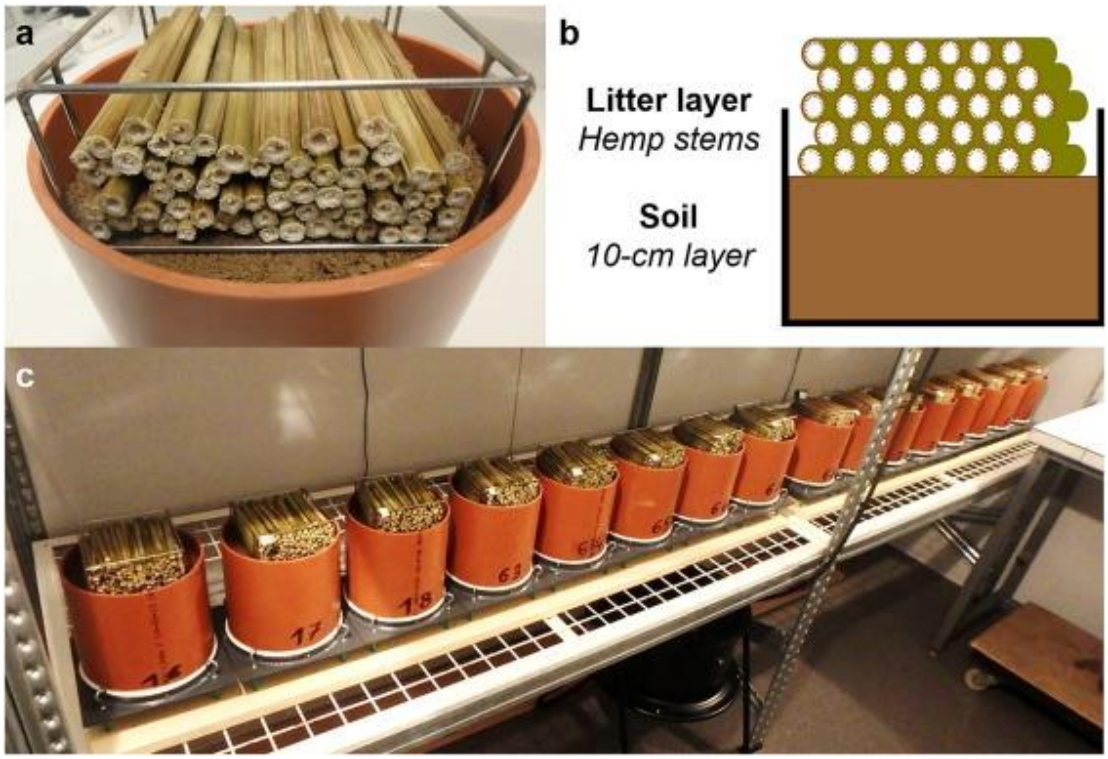

Figure 2-4: Experimental setup of Dew Retting [72]

The study [81] confirmed the breaking down of bast tissue parenchyma and middle lamella fibre bundles by the microbial action during dew retting. Dew retting is popular in the European continent, however dew-retting is not possible in the Semi Desert climate in Botswana, as a result of the high temperatures and low humidity in the summer, which severely limit the development of microbial action [82]. Furthermore, the use of water to mitigate for the lack of dew, is not economically feasible due to the enormous number of operations necessary. Even when the climatic conditions are ideal, changes in weather and fiber erosion by fungi may cause uneven retting [81]. The process requires a careful observation of temperature, soil type, microbes present, water as well as duration of retting to monitor the yield and quality of the fibers. Between the two types of retting, water retting has been found to produce quality fibers with superior breaking strength and elongation at break rates compared to dew retting [78].

2.2.3 Mechanical extraction

Mechanical extraction method produces fibers in greater quantities at one go with a reduced processing time than both dew and water retting techniques. Mechanical extraction is typically performed using a decorticator and a leaf crushing mechanism as seen in Figures 2-4 and 2-5.

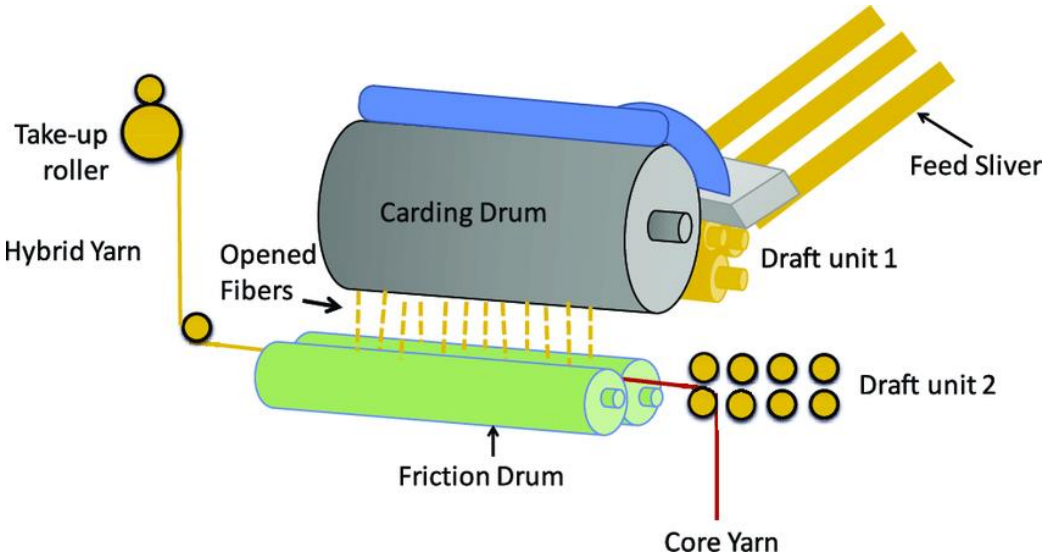

Figure 2-5: Schematic diagram of hybrid yarn manufacturing through DREF III spinning[73]

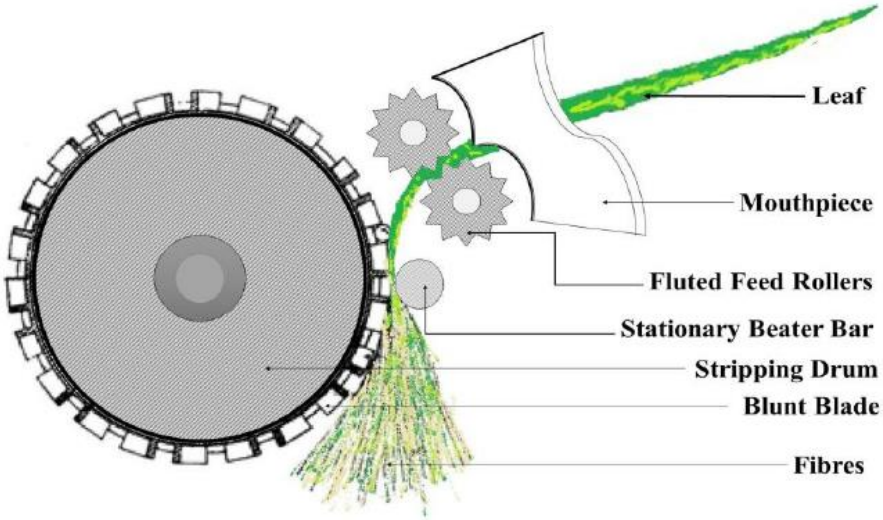

Figure 2-6: Sketch of simple fiber decorticator[73]

Scutching is the process of physically separating fibre bundles from one another after they have been collected [83]. Breaking and swinging are the two key stages in scutching. Fibre bundles are rotated between fluted rollers to extract the leftover wood core, known as shives, in the breaking process. Following breaking is the swingling which is the process of removing shives by scraping the damaged stems tangentially [69]. Long and short fibers are extracted via the scutching method. Mechanical extraction is seen to produce fibers with a rougher surface morphology which is essential and contributes to an exceptional fiber/matrix interfacial adhesion [84]. It also produces fibers with a lower quality characteristics compared to water retting as reported by Rozyanty et al [79]. The results of the study concluded that unsaturated polyester (UPE) composites reinforced with water retted kenaf fibers possessed enhanced interfacial adhesion between the fiber and the matrix compared to the mechanically extracted fibers reinforced UPE composite. The water retted fiber's surface morphology analysis also revealed less damage in comparison to mechanically extracted fibers. Therefore, the latter showed the existence of pores in the microstructure composite. The pores compromise properties such as water absorption and mechanical properties as a result of poor fiber/matrix adhesion [85].

Table 2-2 compares how each extraction techniques discussed in this section influences the process economics, time of extraction and quality characteristics of the extracted ARFs. Data for Table 2-2 was obtained by summarising findings from various literature cited in sections 2.2.1 to 2.2.3. According to Table 2-2, water retting method of extraction is recommended for ARFs in this study because it appears to have a significant impact on considerable fibre properties, including:

(i) the degradation of undesirable substances including sugars and cuticle [86],

(ii) the efficient detachment of the fibres from other stem tissues,

(iii) the fibre quantity, and

(iv) the residual pectin and hemicelluloses [82]

Table 2-2: Comparative analysis of various extraction techniques for ARF

Criteria	Water retting	Dew retting	Mechanical extraction
Processing time	10 to 14 days[87]	14-21 days [75]	Few hours
Cost of equipment	Low	Low	Medium-high
Processing cost	Low	Low	Medium
Breaking strength	High	Medium	High
Elongation at break	High	Medium	High
Interfacial adhesion	Stronger	Strong	Strongest

2.3 Processing challenges in polymer composites reinforced with ARFs derived from agricultural waste residues.

As mentioned above, ARFs possess some shortcomings which include excessive moisture adsorption, and unsatisfactory compatibility with the polymer matrix in composite materials, as a result of their hydrophilic nature and thus require surface modification techniques to modify surface chemical makeup and morphology of natural fibres. In this section, the challenges are discussed in detail.

2.3.1 Hydrophilicity of Natural Fibres in the Polymer Matrix

Lignocellulosic fibres are hydrophilic in nature due to the occurrence of hydroxyl groups which make them susceptible to microbial degradation [32], [88]. The incompatibility of fibres to the hydrophobic matrix causes poor mechanical performances of the resulting composites [89]. Mohamed *et al* [90] found that in general, the banana polystyrene composites are well compounded and made a plausible cheap and sustainable thermal insulating material. However, there is a development of small spaces or gaps inside the banana-polystyrene composites which may be explained by a degree of separation between the banana leaves powder particles and the polymer. The weak compatibility of banana fibers and polystyrene, as well as the aggregation of natural fibers causes the production of these voids seen between powder and the polymer. Diverse physical and chemical treatments are therefore applied to impart fibre dispersion and reduce moisture absorption of fibres in the matrix [1], [55]. Surface modification of fibers and compatibilization are effective ways that have been proven to solve the challenge of fiber-matrix incompatibility [91], [92]. Chemical surface modification has the ability to enhance cellulose's hydrophobicity by modifying the surface morphology of the fibers, removing impurities, roughening the fiber surface in preparation for a strong interfacial relationship, promoting mechanical interlocking, and producing excellent cellulose dispersions in hydrophobic matrix [93].

Figure 2-6 demonstrates that alkaline treatment of wood fibres improves interfacial bond and functional properties of WPCs through the lessening of fibre hydrophilicity [94]. The bond between the wood fibre and the polymer matrix after treatment of fibres is a result of nature of the chemical bond between the ARF and the polymer matrix and mechanical bonding [95], [96]. This

behaviour governs the load transfer between the ARF and the matrix and the functional properties of the composite such as thermal stability which is discussed in the next section.

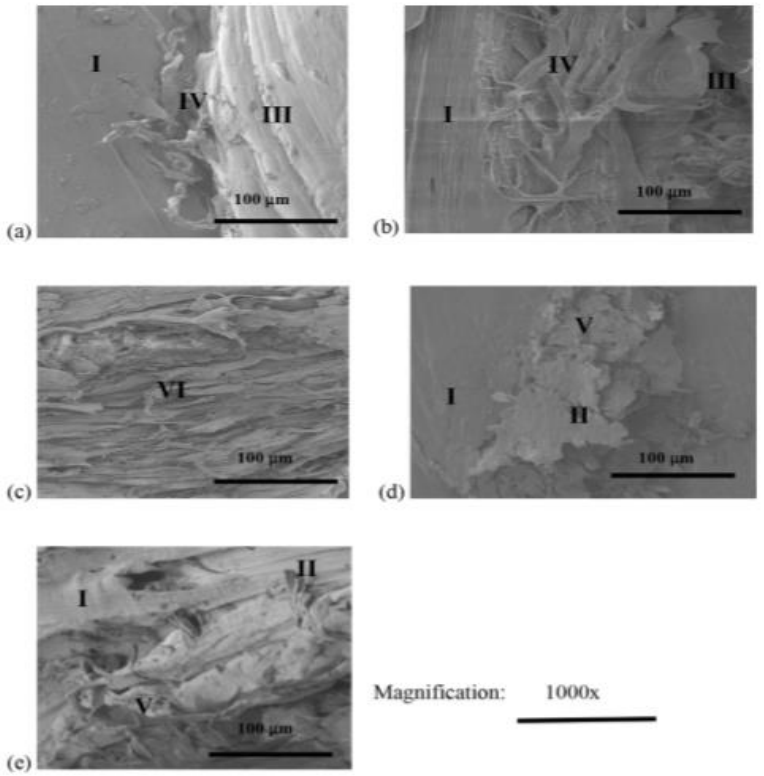

Figure 2-7: SEM micrographs of WPCs reinforced with wood fiber of varying contents from 15 wt.% to 55 wt.% [84]

2.3.2 Thermal Stability

Since ARFRPCs are used in a variety of applications, their thermal stability has become one of the most significant challenges in recent times [97]. Thermal degradation of ARFs is a concern when producing natural fibre polymer composites as lignocellulosic fibres experience chemical and physical changes when subjected to heat at temperatures between 100–250°C [98]. When ARFRPCs are exposed to fire or other sources of high-intensity heat, they undergo thermal

decomposition and combustion [99]. Since various types of ARFs have varying and unique chemical compositions and microstructures, the thermal stability and flammability of ARFRPCs varies. The performance of ARFRPCs at elevated temperatures has been investigated by numerous researchers. According to some reports, ARFs begin to degrade at around 240 degrees Celsius [99], [100], [101]. However, there is a difference among the different components of fiber. Thus lignin begins to degrade at about 200 degrees Celsius, whereas hemicellulose and cellulose begin to degrade at higher temperatures of 220–315℃ and 300–400℃ respectively [102]. The thermal stability of natural fiber composites can be improved by lowering the lignin and hemicellulose content thus improving the interfacial adhesion composites [103]. Research by Sayeed & Paharia [104] detailed that surface modification of jute fibres using chemical treatment such as the alkaline treatment has the capability to improve the thermal stability and other physico-mechanical characteristics.

2.3.3 Durability and degradation

Gama *et al* [105] reviewed fibers from recycled ARFs such as bagasse, rice hulls and pine leaves and noted that water can penetrate into the polymer matrix even in the core regions of lignocellulosic materials because they are hydrophilic, boosting water absorption.

Increasing the hydrophilicity of composites can enhance the material's resistance to degradation process. Consequently, the resulting ARFRPCs also absorb water in humid conditions or when exposed to water on its surface. In a composite, water diffuses through as a result of pores and cracks that develop in the fibre/matrix as imperfections during processing [106]. When water is absorbed by the fibres, it results in the fibre swelling causing further cracks in the fibre/matrix structure. This further weakens the fibre/polymer adhesion and debonds the fibres from the matrix [107] as shown in Figure 2-7. When the fibre/matrix adhesion is compromised, it results into an ineffective stress-transfer, and hence forth, to the reduction of the mechanical characteristics of the ARFRPCs leading to pre-mature aging of the composite materials [108]. This affects durability of the composite material. Diverse physical , biological chemical and hybrid treatments are therefore applied to improve the fibre dispersion and reduce moisture absorption of fibres in the matrix [1].

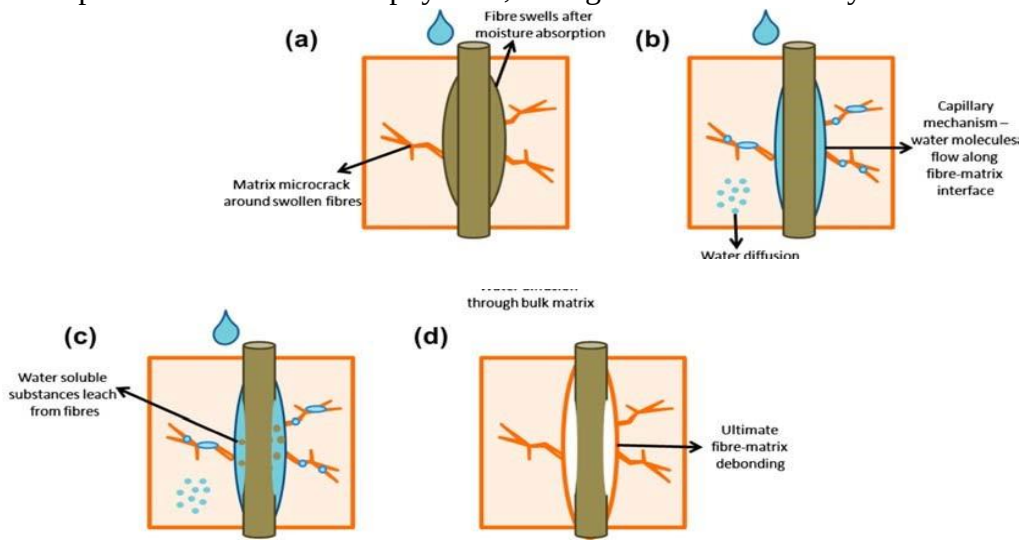

Figure 2-8: Effect of water on fibre/matrix interface [73]

Treatment of cellulose fibres improves interfacial bond and functional characteristics of natural fiber polymer composites through the lessening of fibre hydrophilicity [109]. To lessen the hydrophilic nature of fibers, surface and structural modification techniques have been implemented to minimize prolonged exposure times. A thorough analysis of data on the overall challenges of processing ARFRPCs show that there is insufficient data on the effects of ARFs properties on thermal stability and moisture behaviour. This involves exploratory investigations in order to find the use of relevant ARFRPCs qualities that could lead to cost-effective optimization of these functional capabilities. It's also crucial to analyse the effects of ARFs qualities on the life cycle of ARFRPCs, with the goal of defining and comparing to those of conventionally used composite materials. Furthermore, while determining the best ARFs qualities for producing ARFRPCs, life cycle projections must be factored into the analysis and comparison of the environmental benefits. Analytical or numerical methods could be used in life cycle prediction models that examine the influence of ARFRPCs properties. Working mechanism and effects of different surface modification treatments are discussed in detail in the next section.

2.4 Approaches for improving thermal insulation properties of polymer composites reinforced with ARFs derived from agricultural waste residues.

2.4.1 Chemical modification of natural fibres

Chemical surface modification methods of ARFs involve salinization, alkalization, acetylation, cyanoethylation, benzoylation, dewaxing, esterification, etherification, and graft copolymerization

etc. [93]. This review focusses on alkaline, acetylation, benzoylation and silane treatment as the most economic and less environmentally invasive methods for low-income countries.

2.4.1.1 Alkaline treatment

Alkaline treatment is one of the most established and utilised chemical treatment methods for reinforcement of natural fibre with polymers. This surface modification treatment eliminates lignin, hemicellulose and waxes casing the fibre cellulose [110]. The chemical reaction is shown in Equation 2-1 below [111].

$$Fibre - OH + NaOH \rightarrow Fibre - O - Na + H_2O \qquad 2\text{-}1$$

The NaOH applied reacts vigorously with the fiber. While reacting with the water molecules contained in the fiber, the hydroxyl groups between the molecules are broken apart [112]. As a result, the fiber's hydrophilic nature reduced. Kathirselvam *et al*. [113] examined the effect of NaOH treatment on *Thespesia populnea* bark fibers and proposed that the alkali treatment results in a rough surface of the fibre which aids mechanical interlocking while simultaneously increasing the content of cellulose uncovered on the fibre surface, consequently leading to increased reaction sites. The study concluded that alkaline treatment affects the mechanical properties of fibres, specifically improving fibre strength and stiffness. Figure 2-8 below shows the improvement of fibre surface roughness as the waxes are removed upon treatment with 4 wt.% NaOH concentration up to 150 min from a study by Olakanmi *et al*. [94]. The alkaline treatment caused the wood fibre to get softer and subsequently aids mechanical interlocking.

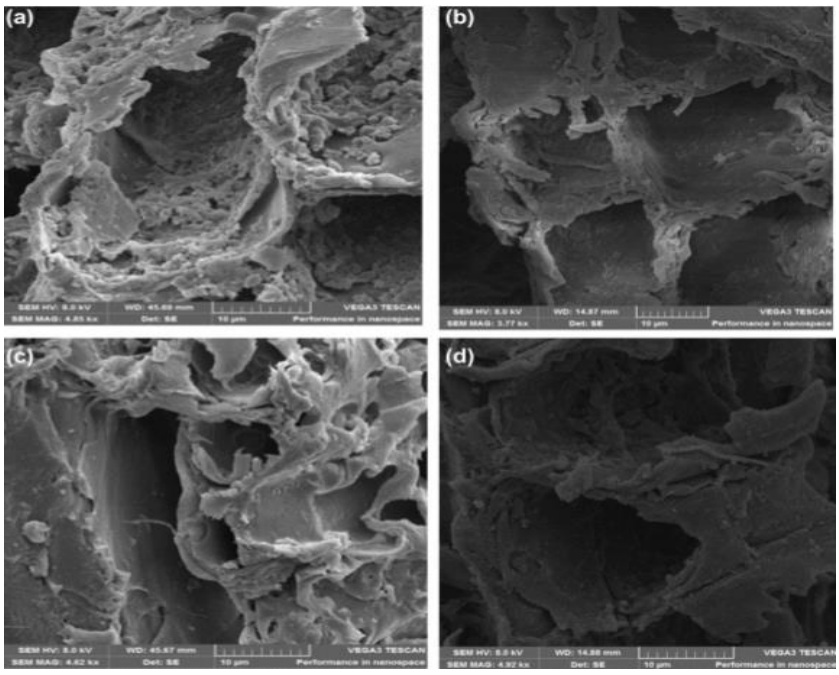

Figure 2-9: SEM micrographs showing the surface morphologies of untreated and sodium hydroxide treated fibers [94]**.**

Bui *et al* [114] also investigated the effect of alkali treatment on coconut fibers and found that when fibers are treated with alkali , their absolute density increases dramatically. The treatment eliminates pectin and contaminants, resulting in a reduction in volume and an increase in absolute density. An increased density is useful when manufacturing ARFRPCs because the more dense fibers leads to less voids formation in the fiber/matrix composite and consequently improved thermal and mechanical properties [115].

2.4.1.2 Silane Treatment

In this process, silane is incorporated as a coupling agent used for fibre surface modification. It is one of the most efficient coupling agents used on fibres. It is dependent on various factors such as pH, temperature and hydrolysis time [27]. Upon contact with water, the hydrolysable alkoxy group

forms silanols, which subsequently bond with the fiber's hydroxyl group to produce a solid covalent bond on the cell wall [116]. As a result of the covalent bond, the hydrocarbon chains from the application of silane prevent swelling of the fiber by establishing a cross connected network [117]. This reaction is shown by Equation 2-2 below.

$$CH_2CHSi(OH)_3 + Fibre - OH \rightarrow CH_2CHSi(OH)_2O - Fibre + H_2O$$

Equation 2-2

Salit [118] compared the outcome of silane surface modification treatments on kenaf/PLA fibres to the effects of a treatment that mixed both saline and alkali treatment (a hybrid treatment). The results proved that salinization improved mechanical properties of fibres, however the hybrid alkali/silane treatments presented the best improvement. Fibers that have been chemically treated with silane have better bonding between their interfaces and the polyurethane matrix, resulting in a more homogeneous structure. The well-distributed fibers in the cell struts operate as diffusion barriers, preventing heat transfer by blocking radiation. As a result, heat radiation is suppressed by scattering or absorption mechanisms, enhancing thermal insulation of the resultant eucalyptus fibers polyurethane composite foams researched by Czlonka *et al* [119].

2.4.1.3 Acetylation treatment

Acetylation involves reaction of the acetyl group (ACH_3COO) with the hydrophilic hydroxyl groups of natural fibres, thus, removing the moisture. During the reaction shown in equation 2-3, acetic anhydride compound replaces the cell wall's polymer hydroxyl groups with acetyl groups [120]. The characteristics of the polymers are altered during this process, and the fibers develop hydrophobicity.

$$Fibre - OH + CH_3 - C(=O) - O - C(=O) - CH_3 \rightarrow Fibre - OCOCH_3 + CH_3COOH$$

Equation 2-3

This results in reduction of the hydrophilic nature of the fibre which advances the dimensional stability and the distribution of natural fibres in the polymer matrix [121]. Silva *et al* [122] researched the impact of acetylation on sugarcane bagasse fiber composition and revealed that cellulose content and acetylated hemicellulose coating on the fiber surface declined steadily until it reached 14% due to cellulose degradation. The results demonstrated that acetylation was efficient and resulted in increased thermal stability. Untreated bagasse depicted a deterioration temperature of 266°C, however the modified fiber had a degradation temperature of 322°C, which is higher than raw bagasse.

2.4.1.4 Benzoylation Treatment

Benzoylation treatment is when benzoyl chloride is utilised for ARF surface modification. The benzoyl (C_6H_5CO) group reacts with the hydroxyl group that exist in ARFs therefore reducing the hydrophilic nature of the fibre as shown in equation 2-4 [123].

$$Fibre - OH + NaOH \rightarrow Fibre - O - Na + + H_2O$$

Equation 2-4

Making natural more hydrophobic fiber like the polymer matrix with benzoyl groups improves the interfacial bonding between the ARF–polymer matrix., thereby, improving the behaviour of the ARFRPCs. A study by Nair *et al.* [124] concluded that benzoylation of the short sisal fibre enhanced fibre–polymer interfacial bonding. This therefore significantly improves the composite mechanical quality properties[125].

2.4.2 Biological Modification of Natural Fibres

Although chemical treatments have been proven to successfully eliminate lignin, hemicellulose, and waxes, thereby, causing the fibre surface to achieve a rough surface of the fibre which aids mechanical interlocking of the fibre/matrix, chemical treatment techniques have disadvantages of residue generation, land and water pollution, high energy use and high cost of some chemicals. This led to the interest in biological methods of surface modifications which are more environmentally friendly. Biological methods include utilising microorganisms (fungi and bacteria) and enzymes to remove the lignin, hemicellulose and waxy components of fibers [126]. Biological methods have already been adopted for surface modification in the textile industry.

Deng *et al*. [127] analysed the reactions of cellulose fibers to biological treatments for the purpose of reinforcing polymer composites. The study investigated the outcome of fungal treatment on the fibre surface morphology characteristics, mechanical properties, and moisture absorption of hemp fibers blended with an unsaturated polyester resin matrix. Analysis of the results revealed an improvement of properties in the treated fibers such as increased tensile strength and resistance to moisture absorption [127]. Recently lipase and laccase enzyme have been used to eliminate lignin, waxes, and hemicellulose to lessen the hydrophobicity and increase the molecular weight of paper and pulp factory waste. A study by Narinthorn *et al*. [110] considered reinforced composites that were treated with lipase and laccase enzyme respectively. Fibers treated with lipase enzyme were imparted with an inferior tensile strength in comparison to fibers treated with laccase.

2.4.2.1 Laccase Enzyme

Laccase is an enzyme that exist and is usually found in plants and fungi [110]. White rot fungi which naturally degrade lignin are one of the main sources of laccase. The mechanism that involve delignification by laccase involves oxidation of the numerous phenolic polymers that make up the lignin polymer, with an accompanying reduction of oxygen to water [126]. Currently, laccase enzyme is mostly used in the paper production industry for the purpose of increasing the wet strength of fibers [128]. Laccase treatments typically comprise of the application of laccase enzymes to stimulate the lignin in fibers for any period of hours to days depending on the concentration of the enzyme as well as other parameters such as temperature and pH. Samples treated during this study, were treated for 5 and 10 days. Since laccase enzymes are too bulky to infiltrate the cell wall (50 to 100 kDa), treatments are limited to surface modification only [129]. Although, biological treatments such as laccase enzyme are cheaper, less energy-intensive and more environmentally friendly unlike chemical treatments, they have their own challenges when using them such as low scalability and problems associated with control of the processes.

2.4.3 Hybrid Treatments

Incorporating more than one type of treatment into ARFs surface treatment is one of the ways to achieve improved ARFRPCs properties as an alternative to one type of treatment. Various types of surface modification treatment can be utilized to improve the interaction between ARFs and the matrix, increase ARFs characteristics, and introduce new qualities into the ARFRPCs. Combination of chemical treatments such as silane and alkali have been used on sugar palm fibers (SPF) to study the treatment's effects of mechanical and interfacial interlocking between the sugar palm fibers with thermoplastic polyurethane (TPU) [130]. For a duration of 3 hours, the sugar palm fibers were modified with a 6% sodium hydroxide solution, 2% silane, and a combination of

6% NaOH and 2% silane (NaOH-silane). The results showed that physical, tensile, and microstructural qualities are less improved in the untreated, and silane/alkali compared to combined alkaline, and silane treated SPF. Treating SPFs with silane and alkali improves its characteristics and improves fiber-matrix bonding in SPF/TPU composites proving that hybrid treatments present a better chance of eliminating hemicellulose and voids in the ARFRPCS structure. To reduce the amount of chemicals used, one might consider the combination of chemical treatments and physical treatments such as plasma, UV, or heat treatment. The effects of treating sisal fibres with a mixture of alkali and high-intensity ultrasound (HIU) on their reinforced PP composites were investigated by Krishnaiah *et al* [131]. Following the hybrid treatments of alkali and ultrasound, FTIR and SEM data indicated the elimination of amorphous components including hemicellulose, lignin as well as waxes and oils. The thermal stability of sisal fibres achieved with a hybrid of alkali and ultrasound surface modification was raised by 38.5 °C when compared to untreated fibres. In addition to using less chemicals, so as to use less costly processes research recommends incorporating heat treatment as a supplement to chemical treatments. Moumakwa *et al* [132] studied the effects of chemical and thermo-alkali surface treatments on the characteristics of water reed and mokolwane plant fibers. In comparison to chemical treatment, thermo-alkali treatment improved the thermal resistance and mechanical properties of the fibers more effectively. Chemical treatment of the fibers resulted in the degradation of hemicellulose, which expanded the interfibrillar region and thermal treatment further enhanced the breakage of links connecting cellulose and hemicellulose. The above cited literature prove that hybrid treatments are better at improving the fiber's compatibility with the polymer matrix. Furthermore, in comparison with other to numerous physical processes, heat treatment is a simple, low-cost, and environmentally friendly way for improving the mechanical properties of ARFs. In addition to the

filling and compatibility features, a combination of chemical treatments with heat treatment can improve the ARFRPCs material's mechanical properties, enhancing the end product's quality. Cao *et al* [133] conducted heat and alkali treatments to kenaf fibers with the goal of increasing mechanical characteristics of natural fiber for the purpose of reinforcing polymer composites. The results concluded that the tensile strength of kenaf fiber heated at 140 degrees Celsius has the greatest value.

Other examples might be given, but the purpose remains the same: the use of hybrid treatments in the fabrication process of ARFRPCs as an emerging technique for producing a variety of multifunctional ARFs composites. Table 2-3 compares and summarises the effects of hybrid treatments in comparison to treatments discussed in Section 5.

Table 2-3: Comparison of ARFs Treatment Methods

Criteria	Chemical Treatments [89],[104],[52]	Biological Treatments [26]	Hybrid Treatments [32]
Processing time	Ideally >3 hours	12 hours >	Few hours
Cost of equipment	Low	Low	Medium
Processing cost	Low	Low	Medium
Breaking strength	High	Medium	High

Elongation at break	Medium	Low-Medium	High
Interfacial adhesion	Stronger	Strong	Strongest

All of the treatments presented in Table 2-3 show varying effects in reducing ARFs hydrophilicity, removes surface impurities, increasing surface roughness, all of which affect the properties stipulated in the table. Hybrid treatment remains the most efficient method, followed by chemical methods, and biological methods which are gaining in popularity. Biological treatment of ARFs, on the other hand, is mostly unexplored. Temperature, concentration, and period of immersion of ARFs in various chemical reagents all affect the properties of ARFRPCs. Most of the literature reviewed studied only one or two variables namely the concentration and period of chemical reagent treatment. With the goal of optimizing various surface modification procedures and maintain minimal use of hazardous chemicals and reagents, it is necessary to conduct a thorough evaluation of hybrid biological treatments in comparison to hybrid alkali methods and study how period of fiber exposure affects ARFRPCs qualities.

2.5 Polymer matrices

Whilst the polymer matrix possesses a low density it is characterised by a comparably low material strength, a disadvantage that is corrected by the fibre filler, which is very strong and stiff. Nevertheless, the polymer matrix provides a medium to protect the surface of the fibers and transfer loads among them [14], [134]. When compounded together, the two materials improve

each other's weaknesses making a lightweight, strong, and durable composite material. In addition, the combination of the fibers from agricultural residue and matrix provides opportunities to modify the final product, proportions, and geometry of the component so as to obtain optimum and required properties of a material [123]. A polymer matrix also serves the purpose of holding the fibers together to make a structured solid material [54]. Additionally, the matrix helps to shield the fibre surface from harsh environmental effects and abrasion especially during composite processing [32]. Another purpose the matrix serves is to retain the reinforcing fibers in the proper orientation and position to aid them to carry the intentional loads, allocate the loads consistently between the fibers and afford resistance to crack propagation and damage [135]. During processing of the composite, the matrix largely controls the overall service temperature restrictions of the composite, as well as the composite's environmental resistance [136]. To choose a matrix for composite production, the properties of the matrix are selected carefully to complement the fiber. First-rate toughness in a matrix for example balances the tensile strength of the ARFs [137]. When the two are mixed together, the product has a high strength and stiffness as a result of the fibers and resistance to crack propagation [138].

Polymer matrices consist of two types namely: thermoplastics and thermosets. Plastics that are thermosetting do not soften when heated after being heated once. They are employed where heat resistance is crucial (e.g. kettles, plugs, laptop chargers etc.)[139]. Thermosets polymers are stiff and do not stretch the way that elastomers and thermoplastics do [140]. Polyester is the most used thermoset polymer in composite. Examples of combinations of polyester-ARF composite include: polyester/coir, polyester/banana/cotton, polyester/straw, polyester/pineapple leaf, and polyester-cotton [141]. On the other hand, thermoplastics are plastics that soften while being heated and may be reshaped. They can reheat once again and re-moulded without affecting the material's

properties [142]. This makes it easy to recycle them, a property that brands them popular in the market. However, for the purpose of ARFRPCs production, thermosets are more suited for the role because of their temperature resistance [23]. Selecting a matrix is also dependent on the processing temperature. ARFs degrade at temperatures between 160 to 180 °C [143]. Therefore, if temperature and chemical resistance are a required composite property, a thermoset matrix is used. Polylactic acid (glass transition temperature of 58°C and melting point of 170°C) and other polymers such as LDPE and HDPE have comparatively low processing temperatures, making them suitable for compounding with ARFs [144].

Recycling industrial plastic waste plastic has the advantages of minimizing the issue of waste disposal, decreasing the usage of virgin plastics, and reducing production costs. A number of strategies for obtaining recycled plastics that may be used or substituted for virgin plastics in a variety of applications have been developed [64]. With regards to development of ARFRPCs, recycled thermoplastics that have been exposed to a variety of storage and reprocessing have also been thought to have considerable potential. ARFRPCs made from recycled and virgin LDPE were compared in terms of physical, chemical, thermal, and mechanical properties [60]. The mechanical characteristics of composites manufactured using recycled LDPE were found to be comparable to those of virgin LDPE composites. Even so, the exposure of recycled polymers to various storage and reprocessing cycles has an impact on their functional qualities. Therefore, for this research rLDPE is used as a matrix for ARFRPCs as any recycled polymers that can melt and be treated below the deterioration point of ARFs can be utilized to make ARFRPCs in the same manner as virgin plastics can. The recycled polymer provides a larger potential for producing low-cost ARFRPCs goods without sacrificing quality characteristics.

2.6 Effects of materials parameters on the functional performance of agricultural waste-based fibers reinforced polymer composites (ARFRPCs)

2.6.1 Particle Size and Distribution

It is common knowledge that particle size and geometry of the agricultural residue-based fibers greatly affect the development of ARFRPCs' properties in relation to bonding quality between the particles, compared to tensile strength characteristics of the fibers [33]. Setswalo *et al*., [95] and Guo *et al*. [143] have reported that smaller particles enhanced the qualities of a natural fiber polymer composite board with a superior internal bond strength. Zhang *et al*. [145] studied the effect of particle size on the insulation properties of wood-phenolic composites. The investigated particle sizes were 250–380, 180–250, and 120–180 µm. Scanning electron micrograph (SEM) analysis showed the formation of porosity in samples fabricated with fiber's particle size and distribution of 250-380 and 180-250 µm, hence, a weaker adhesion between the fiber and polymer. The 120-180 µm size particle board depicted an even fiber-matrix particle distribution, hence, a superior flame retardancy when compared to the other particle sizes.

This is due to the fact that smaller particles were squeezed more tightly with reduced overlapping areas, resulting in uniform homogenous cells with fewer pores [23]. In comparison to larger particles, this results in higher dimensional stability and a cleaner surface for composites boards [94], [146]. Smoother finishes and aesthetics also save energy because no sanding stage is necessary. Smaller particles, especially those in powder form, require more energy to produce and are more difficult to manipulate during the manufacturing process [23]. In relation to thermal properties, it appears that particle size influences thermal conductivity in the sense that as particle

size increases, conductivity values drop [147]. Khalaf *et al* [148] compared to the conductivity of recycled miscanthus/chitosan bio-composites of size 1-2 mm with a thermal conductivity of 0.084 $W.m^{-1}.K^{-1}$ in comparison to 0.07 $W.m^{-1}.K^{-1}$ for longer (2-6 mm) particles. Additionally, Khalaf *et al* 's findings from bending and compressive strength show that composites have the highest stiffness and stress, which decreases dramatically when larger particles are included.

2.6.2 Fibre content

Mechanical properties have been proven to improve with an increase in fibre content in the composite [14]. A high amount of fibre in the composite results in a high specific strength and stiffness of the composite given that an optimum fibre/matrix adhesion is achieved. The fibres provide high strength and provide resistance to twisting and breaking when exposed to stress. Therefore, it is critical that a proper balance between fibre content and wettability is achieved to ensure a good stress transfer [93]. The volume fraction of the fibers (V_f) is also a significant material parameter which influences the composite's mechanical characteristics [149]. Kelly and Tyson [150] recommended the theory of mixtures model for predicting a composite material's modulus and strength. The rule is:

$$Ec = E_f * V_f + E_m * V_m \quad \text{(Equation 2-5)}$$

where E_c represents the strength of a reinforced composite material, E_m and E_f constitute the modulus of matrix and fiber correspondingly whereas V_m and V_f are the volume fractions of matrix and fiber, respectively.

There exists a range of the volume fraction from 10% to 60% for which functional performance of composite is optimized because volume of fibrous reinforcements below 10% imparts poor

mechanical properties into composite due to occurrence pores in the matrix whereas volume of fibre fraction above 60% result in poor wetting due to a deficient fibre/matrix ratio [151]. Zimmermann & Wang [152] explored the influence of fiber volume fraction as well as fiber length on flexural characteristics of a composites reinforced with kenaf fiber in biodegradable resin and manufactured by hot-press forming. They discovered that as the volume fraction of fiber increases, the flexural modulus reached a maximum value of 3991 MPa at 62vol%. The fiber fragmentation seen at the surface was responsible for increased flexural modulus. Ramaniah et al [153] studied the thermo-physical properties of biodegradable borassus fruit (BFF) fiber-reinforced polyester composites and found that composites' thermal conductivity diminishes as fiber concentration increases. At 0.15, 0.26, 0.34, and 0.41 volume percentages of fiber, the thermal conductivity of composite reduced by 4.1 percent, 12.8 percent, 19.8 percent, and 23.5 percent, respectively. The addition of BFF lowers the thermal conductivity of polyester resin, thereby, increasing the insulating capabilities of the composite. Satish *et al* [154] also investigated the effect of fiber content on thermal conductivity of pine apple leaf fiber–latex composites and reported that when the fiber content of the composite increases, its thermal conductivity falls.

Table 2-4: Thermal Conductivity and Water absorption coefficient values of PALF ARF reinforced composites [150].

Volume Fraction of the fiber (%)	5	10	15	20	25	30
Thermal Conductivity (W/m.K)	1	0.79	0.64	0.59	0.39	0.33
Water Absorption Coefficient	5.69	11.44	13.62	16.21	18.64	24.14

This is because the air is contained in the hollow region of the reinforced fiber. The percentage of air volume rises as the fiber content rises. The results also showed that as the volume proportion of fiber grows, so does the water absorption coefficient value. The percentage of resin, that is natural rubber, drops as the volume fraction of the fiber increases, resulting in increased water absorption. Because there is less resin and more fiber available, the water absorption capacity increases as the fiber content increases.

A review of the literature concludes that properties of ARFRPCs improve with diminishing fiber content, and as a result more polymer matrix content in the composite. This poses a drawback as the aim is to use less polymers in the development of environmentally friendly building insulation materials. Therefore, there is a necessity of more studies to find processing methods to fully utilize residue fiber content quantities that could still lead to a cost-effective and efficient composite material. It is also crucial to examine the impact of ARFs content on the life cycle of ARFRPCs, with the goal of evaluating and comparing their environmental effects to those of conventional composite materials.

2.6.3 Processing of Fiber Reinforced Thermoset Composites

Fabrication techniques suitable for fabricating ARF-reinforced polymer composites such as the hand lay-up technique for unidirectional ARFs, sheet molding (SMC)/ bulk molding (BMC) for short and chopped fibers, filament winding, and pultrusion for continuous fibers. These techniques are selected for this literature review as they are suitable for short and pulverized fibers, as well as continuous fibers. It’s worth noting that, unlike synthetic fibers, ARFs do not normally have

continuous fibers. Some natural fibers (single) can be up to 4 meters long, and if a bundle of fibers is joined in the length direction, the fiber length can be even longer [103].

2.6.3.1 Hand Lay Up Method

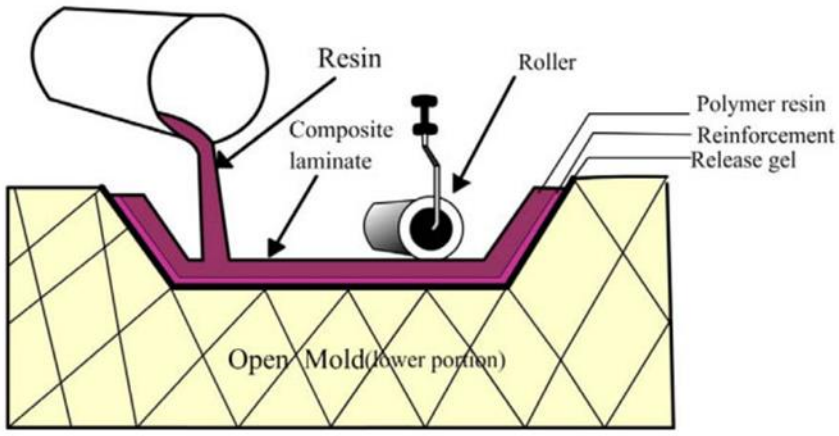

Figure 2-10: Hand Lay Up Method [73]

Hand lay-up is by far the most popular, easiest, and least expensive composite manufacturing technique [155]. In this approach, a release agent is first applied to the open mold as an anti-adhesive agent, and then the fibers are inserted in the mold as shown above in Figure 2-9. Resins are poured and brushed onto the fibers with a roller or brush. Layer upon layer of lay-up is applied until the ideal thickness is achieved. Squeegees or rollers are used to manually release trapped air in the laminate. After that, the laminates are allowed to cool under normal atmospheric conditions [156]. Because of its use of normal atmospheric temperatures and pressure, the hand lay- up is limited to polymers in liquid form such as epoxy resin. A hardener is also usually used to help the composite set [157].

The hand lay-up method performs well with long fiber ARFRPCs since it does not require the use of heat. This approach, on the other hand, requires for a low fiber content (less than 20 percent wt.

percent) therefore does not allow much flexibility in relation to varying fiber content and length and limits the composite's quality characteristics [158].

2.6.3.2 Injection Molding

Injection moulding (IM) is a continuous procedure with two stages: plasticizing plus injection[159]. IM is among the most essential polymer fabrication techniques for manufacturing polymer-based composite, as per Muhammad *et al* [160]. The stages of the IM process are cyclic and can be summarized as follows: (i) filling, in which molten plastic is introduced under pressure into a sealed, cooled mold; (ii) packing, in which the pressure is retained high such that the molten plastic remains in the interior of the mould cavity and deformation is avoided during solidification; and (iii) cooling/plasticating, which entails cooling the item within the mold till it is stiff enough to be evacuated, as well as melt preparation for the next cycle (plasticating stage).

The screw revolves during the plasticizing phase, and the solid polymer is transported and melted within the injection barrel until it reaches the liquid state, ready to be injected in the following cycle. All of these stages take place throughout the injection and clamping processes [23]. A schematic of IM and the described process is shown in Fig. 2-10.

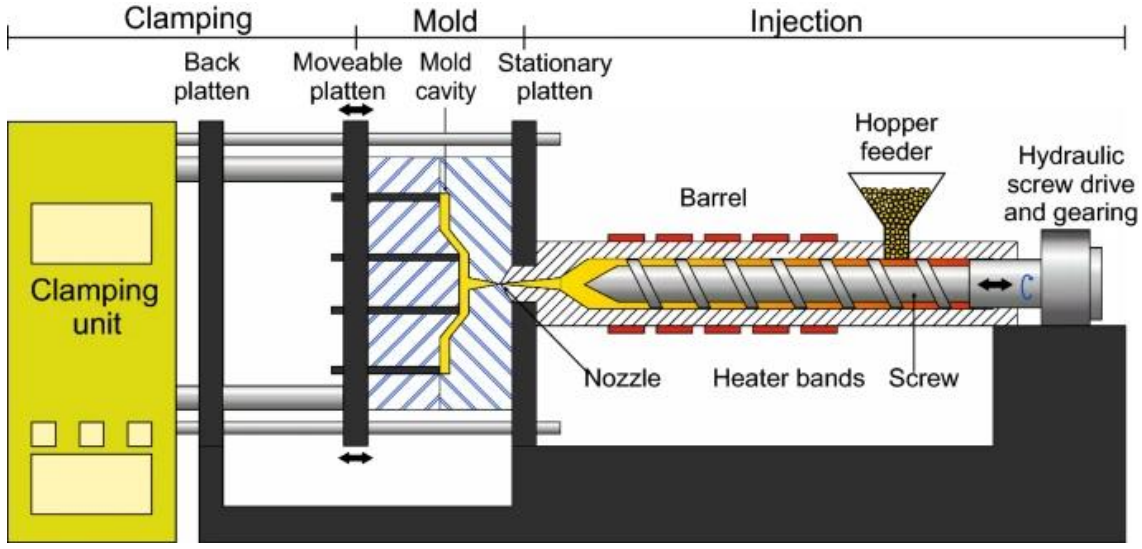

Figure 2-11: Injection Moulding Process [153]

IM usually requires optimization of parameters that suit the polymer and type of fiber. The injection molding screw extruders have greater fibre dispersion and mechanical qualities than compression molding. However, injection molding for such composites is often regulated to less than 40% fiber content because of viscosity constraints [160]. For the study on marble waste characterization and reinforcement in low density polyethylene composites via injection molding, Khan *et al* [161] the produced composites which outperformed neat LDPE in terms of mechanical strength, and by using marble debris, the amount of polymer (plastic) used was lowered because of the fabrication method as compared to compression molding which is a wastage problem. A detailed critique and review of compression molding is mentioned in the next section.

2.6.3.3 Compression Molding

The most popular fabrication technique of casting thermosetting materials including SMC (sheet molding compound) and BMC (bulk molding compound)is compression molding. The cold press and hot press procedures [23] include pressuring materials containing a thermal catalyst in a preheated matching metal die utilizing a vertical press. Approximately 70 percent of fibers can be integrated with this process, and the resultant thickness can range from 1 to 10 mm. Curing temperatures for cold press and hot press molding are typically 40-50°C and 80-100°C for 1-2 hours, correspondingly.

Compression molding is the most common and cheapest following the hand lay-up method shown in figure 2-11. The process requires a pre-compounding equipment to mix the fiber and polymer before fabrication [162]. The method also requires optimization of parameters such as amount of dry material, pressure, and compression temperature. As a result, there is wastage of material that

occur while performing trials [21]. Large volumes parts, lower temperature conditions, and the minimization of thermomechanical deterioration are all advantages over other techniques mentioned previously [163]. The compression during processing is often affected by the structure and composition of fibres, as well as lesser degrees of fibre alignment. ARFRPCs, on the other hand, are easily

compactable than glass fiber composites [109]. This technique, which is ideal for low-volume manufacturing runs, can produce ARFRPCs with optimal component strength.

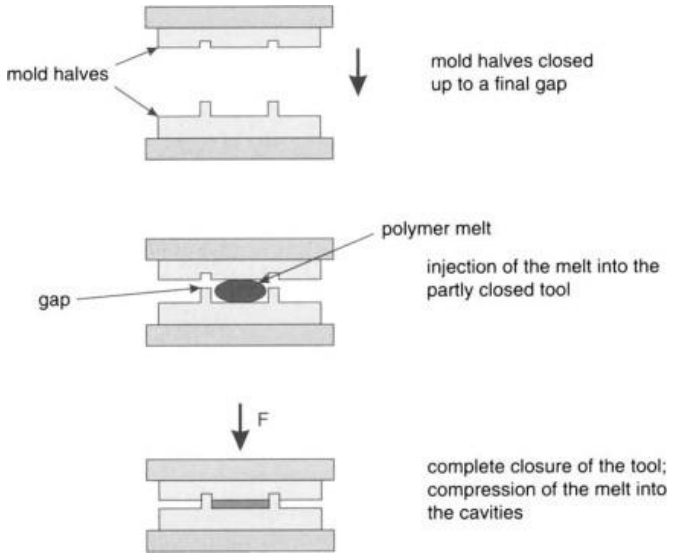

Figure 2-12: Compression Molding [153]

2.6.3.4 Extrusion moulding

Extrusion moulding is amongst the extensively utilized processes in the natural fiber composites production techniques. This method begins with the thermoplastic material being stored in a hopper as pellets or grains as depicted in Figure 2-12. After that, they are moved into a heated

barrel to melt. The melted plastics are then employed moulded into the desired shape. The product must be cooled at the end.

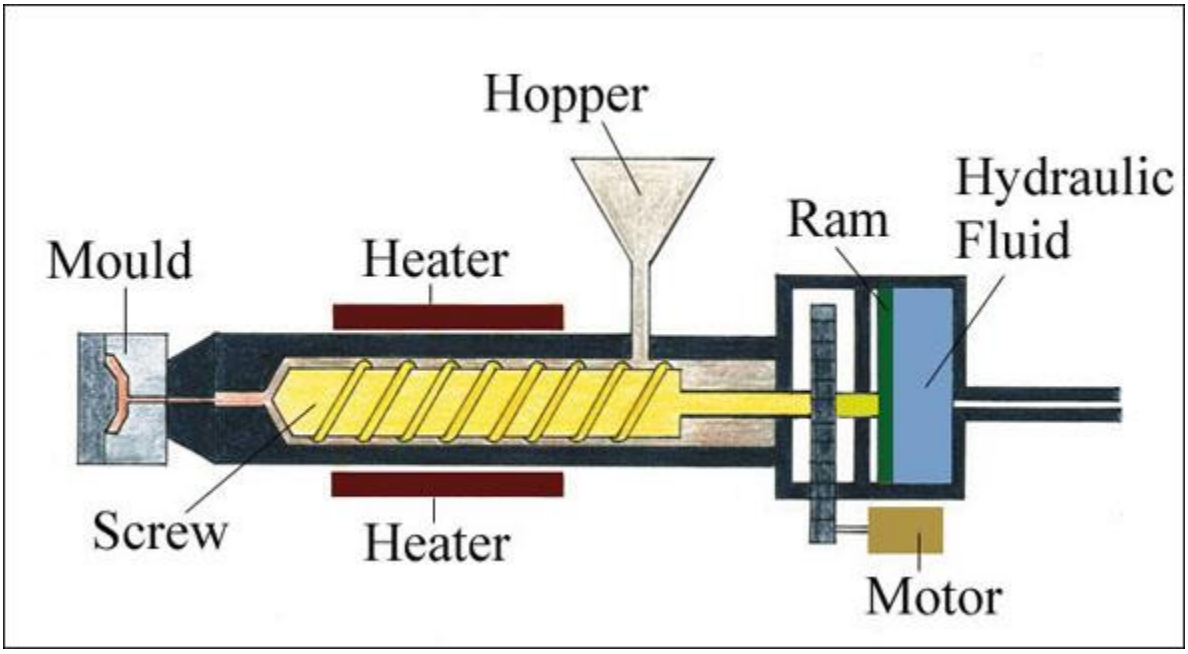

Figure 2-13: Extrusion Moulding Model [153]

Extrusion molding is one of the best methods in producing insulation materials, as the method can produce foam insulation materials which are light weight, and insulative against heat and sound [164]. The process also ensures effective compounding of the fiber and polymer hence consistently even distribution of particles is observed, as well as a smooth appearance. Jiang *et al.* [165] combined extrusion and compression to achieve production stability and a high quality reinforced composite material. The authors pointed out that the properties of the composite were improved by the continuous feed function offered by the extruder. This also helped to maintain the same parameters i.e., temperature and pressure hence obtaining products with exceptional mechanical and structural properties with reproduceable results.

In conclusion of section 2.8, ideally ARFRPCs production techniques should provide uniform dispersion of fibers inside the matrix, limit thermal stresses, and achieve complete and uniform composite make-up. Meanwhile, industrial optimization techniques have only been used in a limited way to discover the elements that affect quality features during the fabrication of ARFRPCs. This has resulted in material, resources, and chemical waste, as well as an increase in other production-related expenditures. The shape and properties of composites are influenced by the processing procedures and operating circumstances. For this study, compression molding is selected to produce the ARFRPCs as the composite sample the process involves an initial quick press closing, and ultimate cooling under pressure [88]. Previous research has shown that the hot compression molding technology can create flat thermoplastic and ARF filler composite panels.

2.7 Behaviour and characterization of thermal insulator developed from ARFRPCs.

2.7.1 Thermal characterization

Among all feasible measures, building insulation, both new and existing, plays a critical role, as it is well known that adequate building thermal insulation can save up to 65 percent of energy usage [166]. As mentioned in section 1.2, most insulating materials are made mostly of natural and man-made synthetic fibers, and various studies have investigated their thermo-physical properties. In order to save energy, an effective thermal insulator must minimize heat conduction between two surfaces of differing temperatures [167]. An optimization framework was presented after a comparative examination of building insulation materials features (thermal, hygroscopic, acoustic,

fire reaction, environmental, and cost) and their response in various climate conditions [168]. Thermal conductivity, *k* and volumetric heat capacity, C are two basic features of insulation materials. Insulation materials should have a lower thermal conductivity than frequently utilised construction materials, leading to less heat transfer via the building envelope [169]. The volumetric heat capacity is a measurement of a material's ability to store thermal energy. By anti-phasing with external temperature, high thermal mass material has the potential to curb and reduce indoor peak temperature, reducing the danger of summer overheating [170]. Thermal conductivity, thermal energy storage, acoustic, thermal mass, hygroscopic, fire reaction , and environmental were among the attributes of insulation materials studied in several review studies [171]. Other research looked at the outcomes in relation to dynamic behaviour , lifecycle cost , and operational CO_2 emissions [7].

Ultimately the overall thermal conductivities of ARFRPCs are based on the values of polymers and thermally conductive fillers [172]. Guo *et al* [173] stated that the commonly used concepts that are employed to explain thermal conduction mechanisms in polymer composites are thermal conduction course, thermal percolation, and thermoelastic coefficient theories. Out of all the three theories mentioned, the most commonly known is the thermal conduction mechanism which states that pathways are created by the interaction of thermally conductive fillers within the polymer matrix [174]. The heat flux can flow through thermally conductive particles or channels that have lower thermal resistance. Once the loading of thermally conductive fillers is reduced, they are separated from one another and isolated by a low-value polymer matrix, forming a "sea-island" system as shown in Figure 2-13(a) [173].

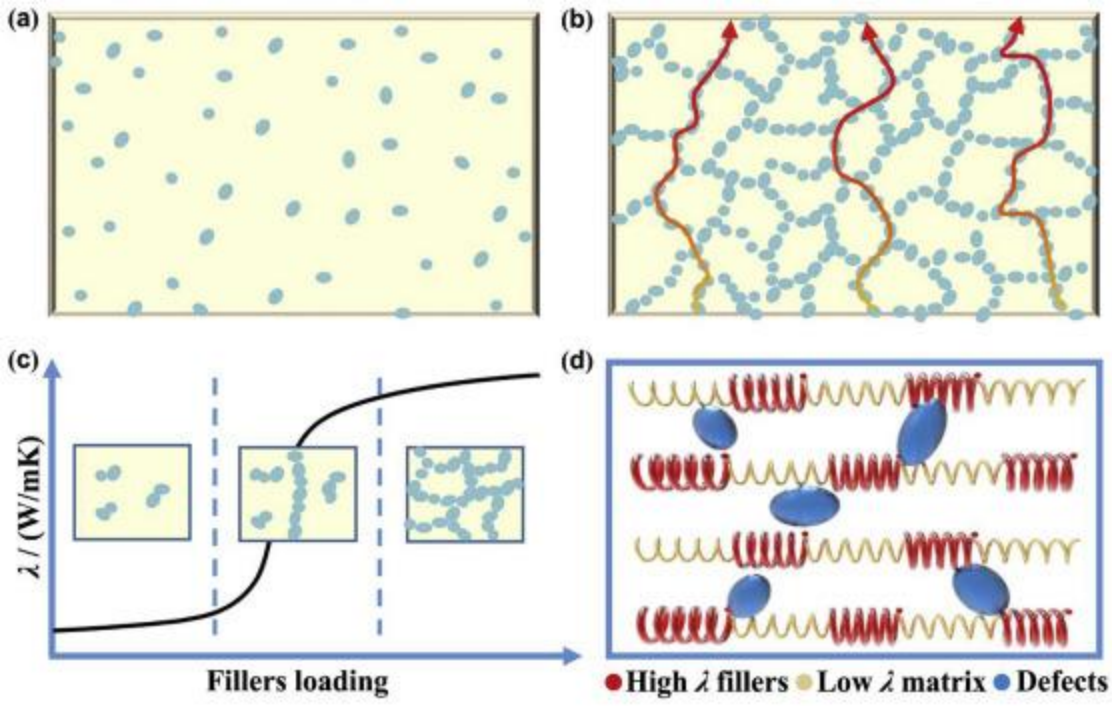

Figure 2-14: (a) "Sea-island" in low fillers loading; (b) Thermal conduction paths in high fillers loading; (c) Percolation phenomenon; (d) Thermoelastic coefficient theory [166].

2.7.2 Thermal Characterisation

The thermal conductivity *k* (W/(m.K)) for a steady state condition and thermal diffusivity (D) (m^2/s) for an unstable state are commonly used to analyse the primary characteristics of thermal insulation effectiveness [54]. Thermal characteristics for a wall thermal envelope can be calculated and stated using the U-value of thermal transmittance, as per ISO 6946 standard [175]. Dixit & Yadav [155] noted that given the same filler is used, that the final thermal conductivity of ARFRPCs with a greater thermal conductivity polymer matrix are substantially higher compared to polymer composites with a lesser thermal conductivity polymer matrix. Abu-Jdayil *et al.* [13], analysed the thermal conductivity of building insulation materials by categorizing them as traditional, state-of-the-art, renewable, or other construction materials . They discovered that the

most advanced insulators have the lowest thermal conductivity (under 25 x 10^{-3} W/m.K), whilst building material made from cement have the highest thermal conductivity of 200 mW/m.K Agave and wheat grass fiber were investigated as possible insulation materials and the thermal conductivity values obtained for each sample in the temperature vary from 10–60°C with the final overall value ranging from 0.04555–0.06835 W/m .K [176]. Without any polymer matrix bonding, loose fibers of agave and wheat straws have an overall thermal conductivity value of 0.043592 and 0.044678 W/m.K, respectively.

2.8 Conclusion

Although there are several reports in the literature which gives the idea of the mechanical behaviour of ARFs, very limited research has been done on mechanical behaviour of indigenous agriculture waste-based fibre reinforced polymer composites. Many investigators found and reported that the interfacial interlocking can be improved by the treatment of ARFs through alkali treatment and treatment, which as a result improves the ultimate performance of the fibre in their applications. In overall it was concluded from the literature that the alkali-treated fibre presented superior mechanical properties compared to untreated fibres. It is concluded that the suitable fibre surface modification, being hybrid treatments for this study, vastly contributes to enhancing the interfacial characteristics of the resulting natural fibre polymer composites.

CHAPTER 3: METHODOLOGY

3.1 Chapter Summary

In this study, ARFs from sorghum stalks were extracted, treated, and pulverised to reinforce recycled low-density polyethylene (rLDPE). rLDPE was selected for this study based on its consumption pattern, availability in refuge, recyclability and use potential. The thermoplastic used in this study originated from post-consumer recycling sources. In this study, the composite samples were made through compounding followed by compression moulding in a hot press. These composite panels were tested to quantify the material performance according to the standard methods outlined in American Standard Testing and Materials (ASTM). The Fourier transform infrared spectroscopy (FTIR), bending and tensile properties, surface morphology, crystallinity, thermal conductivity, and thermal gravimetric analysis (TGA) of the composites were investigated so as to identify which treatment produced the best composite.

3.2 Materials

3.2.1 Sorghum stalk Residues

The agricultural residue-based (AR) fibers used in the production of the ARFRPCs in this study were from sorghum plant. Sorghum (Sorghum bicolour L. Moench) residue stalk is one of the most abundant and resilient crops planted in Botswana [177]. Sorghum production output in Botswana varies greatly from year to year, and is almost totally dependent on rainfall, with annual production average 46,000 tonnes and varying between 8,200 and 175,000 tonnes [178]. Sorghum is a versatile crop that can be used for grain, forage, fodder, and, more recently as a bioenergy crop. Sorghum grain is consumed by humans or used as animal feed in Botswana, while the stalks are

used as animal feed or building material. The stem is oval or circular in cross-section and the diameter ranges from 5 to 30 millimetres [72]. A thick waxy covering covers the internodes, giving it a blue-white colour. The waxy layer decreases transpiration and improves the plants' resistance to drought. Figure 3-1 shows the dried and cut up sorghum plant residue stalk that were collected from Pandamatenga Farms, Botswana.

Figure 3-1: Sorghum Plant Stalks

Agriculture residue fibers (ARF) were extracted from sorghum stalk (Figure 3-1) obtained from a farm in Pandamatenga, Botswana. The sorghum stalks were physically treated by scrubbing them completely in a 2 percent soap solution at room temperature of 23-25°C followed by tap water, and then drying in the sun under normal temperatures for 72 hours until they reached no change in mass. Before characterization and chemical treatment for further processing, the fibers were gathered together and stored in the lab.

3.2.2 Recycled Low Density Polyethylene (rLDPE)

Study of literature which focuses on virgin and polymer matrix has revealed that the mission to reduce pollution caused by plastics has increased the use of recycled polymer in development of composites. When coupled with ARFs and treated in various ways, recycled plastics can have a wide range of grades, colours, and properties, resulting in an impressive performance comparable to virgin plastics. As a result, the performance of ARFRPCS made from recycled rLDPE and sorghum residue fibers was used in this study. Myplas of South Africa provided the recycled LDPE granules. Table 3-1 shows the properties and processing data of the rLDPE as obtained by the technical data sheet.

Table 3-1: Mechanical properties and processing data of Recycled LDPE – from the supplier's technical data sheet

Properties	Values
Density (g/cm^3)	0.91-0.93
Tensile strength (MPa	6.9-15.9
Tensile modulus (MPa)	4500
Thermal Conductivity (W/m.K)	0.4
Elongation (%)	>100
Melting point (°C)	98-115
Thermal Decomposition Temperature (°C)	450

3.3 Water Retting

Natural water retting was found to be the most promising approach at the moment. Dew retting and mechanical extraction of fibres, among other procedures, are not acceptable due to the poor quality of the produced fibres [80]. Natural water used had an initial pH of 7.54. Sorghum plant stems were chopped into pieces ranging in length from 300 mm. The stems were light brown in colour before water retting process. The stems were kept in water at room temperature (28°C) for 10 days as study of literature in section 2.2.1 demonstrated that water retting is efficient when conducted for a period of 10-15 days. The pectin of sorghum plant straw bundles was broken down by bacteria in the water, which loosened the individual fibers. The sorghum plant fibres were immersed in a water bath of over a period of 10 days at room temperature to make extraction easier as seen in Figure 3-2.

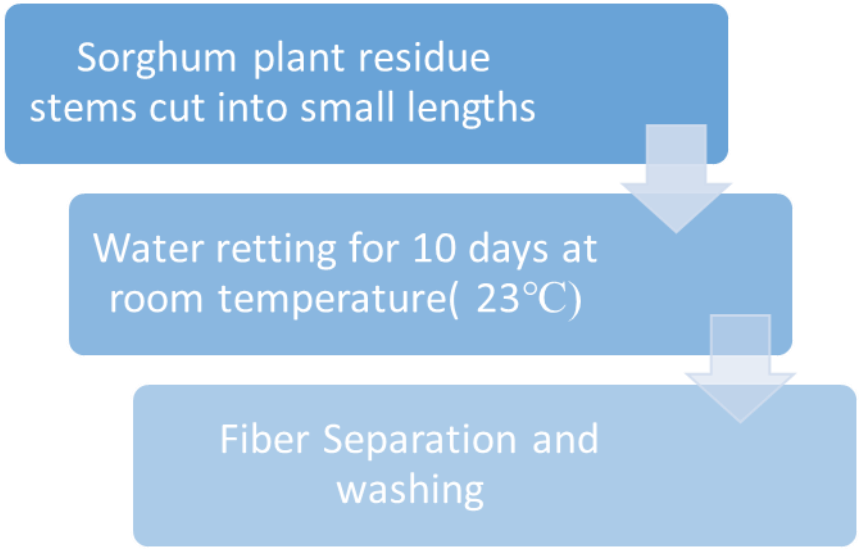

Figure 3-2: Process flow for extracting fibers from Sorghum plant fibers

Figure 3-3: Water Retting of sorghum stalks

The soaked sorghum plant stems were taken from the water and washed in distilled water to remove the stem residue and any waste particles and separate the individual fibers manually using hands after 10 days had elapsed. The leftover water was filtered after the fibers were extracted. Figures 3-2 shows a flow chart of different steps of the retting process. The extracted fibers were air dried for 3-5 days at room temperature.

3.4 Surface Modification of Natural Fibres

Hybrid treatments have been proven to modify the surface of ARF effectively compared to singular chemical or biological treatments. Therefore, for this study surface modification of the natural

fibres by means of economic thermo-alkali and thermo-laccase treatment was carried out to improve adhesion and interfacial interlocking strength, water absorption resistance, dimensional and thermal stability in the ARFRPCs.

3.4.1 Thermo-alkali treatment method

Sorghum residue fibers were soaked separately in a 1wt% NaOH solution in a water bath for 5 and 10 days at room temperature, resulting in a fiber: liquid ratio of 1:4 to completely cover the fibers (by weight). A 1wt% of NaOH is chosen for this study to use as little toxic chemical as possible. However, the duration is varied to compensate for the constant percentage concentration. The fibers were washed multiple times to remove any NaOH solution that had adhered to the surface.

Figure 3-4: Neutralising Kit

Every NaOH treated sample was then washed in diluted 0.9 wt% HCl. The pH of the samples was periodically checked using the neutralising kit seen in Figure 3-4. After neutralisation, , the fibers were then dried in an 80°C oven for 24 hours. Heat treatment of the ARFs under oxygen-deficient conditions leads to a physical modification method for modifying quality characteristics of the fibres such as surface roughness and strength. This process included degradation of lignin and hemicelluloses to extract cellulose fibers. Surface imperfections were clearly visible on the untreated ARFs surface. When compared to the untreated ARFs, the surface of the treated ARFs seemed rough after the treatment process.

3.4.2 Thermo-laccase Treatment

Laccase enzyme performed well in improving the surface quality of fibers by increasing surface roughness, eliminating hemicellulose and lignin as well as increasing fiber stiffness when compared to alternative ways of surface modification [179]. The method is also sustainable and environmentally friendly as it is extracted from termite mounds found in nature with few harmful chemicals.

Figure 3-5: (a) Termite Mounds (b) Laccase Enzyme

Laccase enzyme extracted from termite mounds shown in Figure 3-4 was used as the biological treatment. Laccase enzyme in solution state was added to distilled water in a 1:20 ratio, respectively. Extracted fibers were immersed in the enzyme solution for 5 and 10 days at room temperature conditions. After the treatment, the fibers were washed with distilled water and dried in an oven at 80℃ for 24 hours. Table 3-2 below summarises the types of treatment used in the fibers and duration of exposure to the treatment.

Table 3-2: Surface Modification Treatment Formulations

Sorghum Plant	Thermo-alkali (1% NaOH 80℃ / 24hrs)	Thermo-Laccase Enzyme
UR	-	-
L5	5 days	-
L10	10 days	-
A5	-	5 days
A10	-	10 days

3.5 Pulverisation of the Natural Fibre Samples

The modified solid fibres were pulverised into fine particles using a Fritsch Planetary Mill Pulverisette 5 classic line depicted in Figure 3-6. The Planetary Mill pulverisette 5 has 4 working stations, high-energy effect of the grinding balls and grinds hard, medium-hard, soft, brittle, tough and moist materials down to fineness.

Figure 3-6: Fritsch Planetary Mill Pulverisette 5

Sieving is a simple and convenient way of separating particles of various sizes; for the classification of the sorghum ARFs, sieves with various types of sizes were utilized as shown in Figure 3-7.

Figure 3-7:The round vibratory sieves (b) the sieves with Sorghum AR particle fiber

The treated and untreated particle fibers were separated using round sieves in a variety of sizes, 1.10 mm, 850 µm, 600 µm, 500 µm, 300 µm, and 100 µm mesh sizes. For this study, fibers the size of 100-300 µm were used to reinforce the rLDPE polymer composites.

3.6 Compounding

The thermoplastic based ARFRPCs are typically manufactured in two steps: (1) compounding the ARF filler and polymer granules, and (2) compression molding of the compounded material to fabricate a composite board product [84]. To make consistent composites, proper mixing of ARF filler and polymer is critical.

The sorghum stalk fiber particles and rLDPE granules were mechanically compounded using a compounding machine shown in Figure 3.8. The sorghum AR fibers and the rLDPE polymer granules were processed as shown in Figure 3-8(a), with the raw materials being crushed into small size particles.

Figure 3-8: (a) Compounding machine (b) Fiber particles and LDPE granules inside machine

Compounding is also necessary to thoroughly mix the two components together for 15 minutes into a homogenous mixture. Irrespective of treatment conditions, each compounding sample consisted of 40 wt. % LDPE and 60wt% ARFs. The specific ratio was suggested by an industry expert after consultation. Attempts to gather quantitative peer reviewed evidence in literature to support the suggestion did not bear fruit. Qualitative evidence supports the choice.

3.7 Compression Moulding

The compression moulding machine was switched on prior to pressing to allow the machine to reach a temperature of 150°C. Before the board was made, the compounded powder mixture was dried at 105°C for 1 h to remove the moisture gained as seen in Figure 3-9(a).

Figure 3-9: (a) Compression moulding mould (b) Removal of extra moisture

After drying the compounded pellets in the mould were pressed for 15 minutes (min) under a pressure of 1.0 mega-Pascal (MPa) and a temperature of 105°C. After this, the mould with the panel was moved to a cold press made up of a wet towel and weights to keep the board compressed whilst cooling as seen in Figure 3-10(b).

Figure 3-10: (a) Composite board after pressing (b) Cooling and compression of composite board.

The final composite board having dimensions of 165 × 152 × 5.0 (thickness) mm is shown in Figure 3-11.

Figure 3-11: Composite board

After cooling the composite was cut into pieces for the tests and analysis using a circular saw.

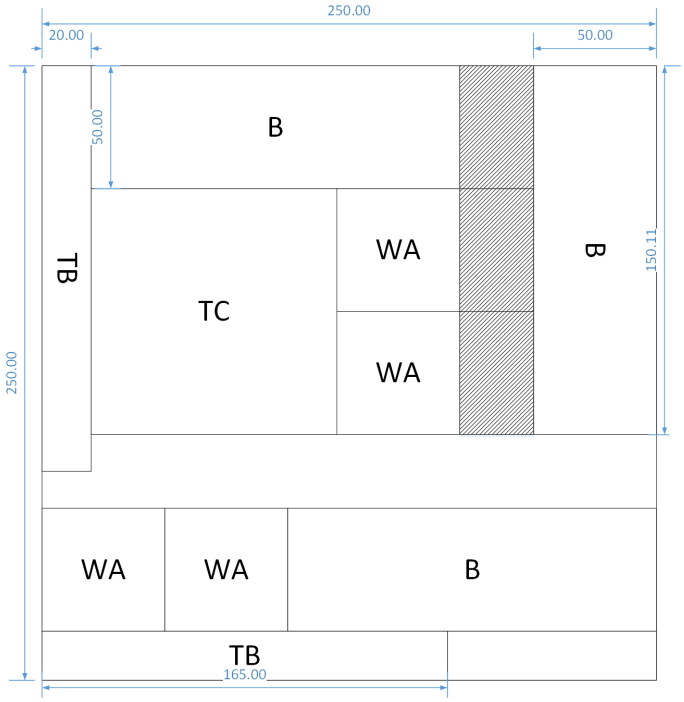

Figure 3-12: ARFRPCs Analysis Samples cutting scheme

Figure 3-12 displays the cutting scheme with dimensions in millimeters, used to cut the samples for analysis using ASTM D3039 and ASTM D790 where:

WA- Water absorption

B- Density, FTIR and XRD test samples

TA- Tensile Test

TC- Thermal Conductivity

Careful measures were taken to ensure that samples were cut from various points of the point to avoid bias when testing the samples and account for any lack of uniformity that may be present because of the fabrication process. After cutting, the pieces were stored in a conditioning room at a relative humidity of 60% and temperature of 24℃ for 10 days to allow the boards to reach a constant mass whilst awaiting analysis.

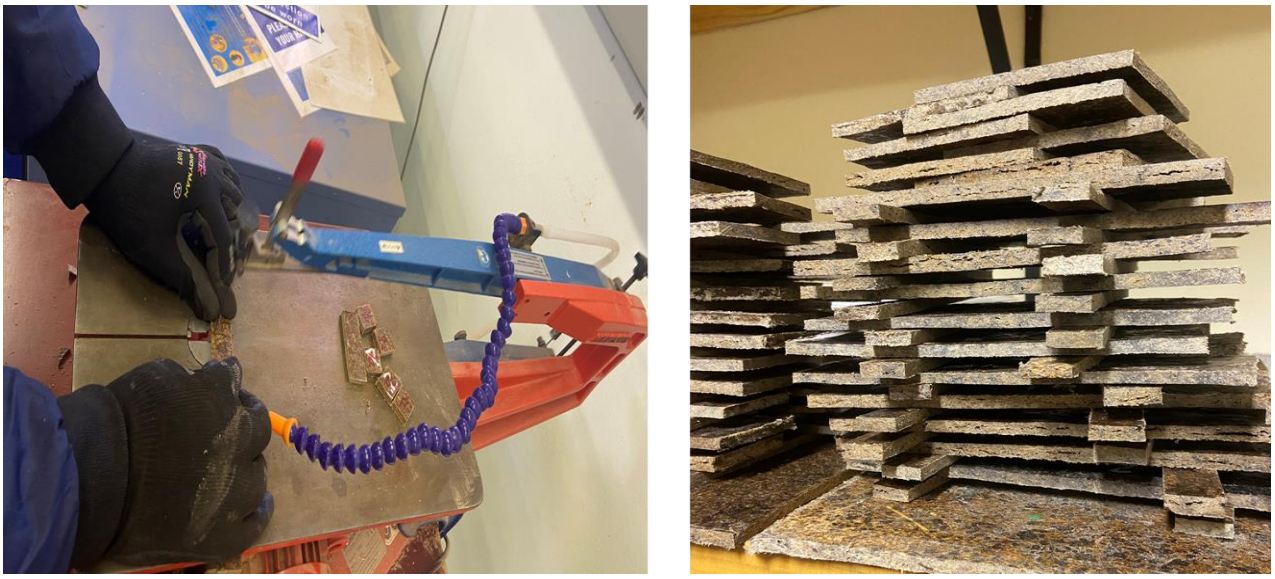

Figure 3-13: (a) Cutting boards into sample sizes (b) Boards stacked for conditioning.

3.8 Characterisation of composites

3.8.1 Mechanical Properties

3.8.1.1 Tensile testing

The tensile test was employed to determine tensile strength and elongation at break, which are all measurements of tensile characteristics of the ARFRPCs. The EN ISO 527- 4:1997 standard was used to conduct tensile testing on dog bone shaped specimens as shown in Figure 3-14. With a crosshead speed of 2 mm/min, an Instron universal testing machine (model: 3369) with a load cell of 50kN was employed for testing.

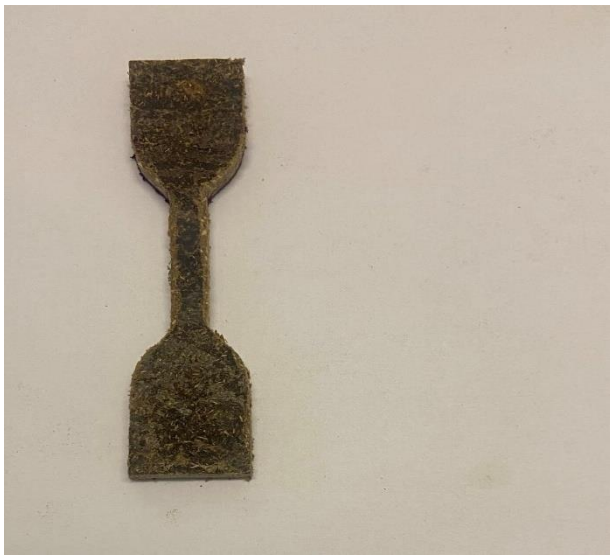

Figure 3-14: Tensile testing sample

Each treatment method was analysed using five specimens (150 mm x 25 mm). The raw data was analysed with Instron's Bluehill 2 Material Testing Software.

3.8.1.2 Scanning Electron Microscope (SEM) Analysis

ARFRPCs microscopic examinations were carried out using a Jeol JSM-7100F Thermal field emission Scanning Electron Microscope depicted in Figure 3-15. SEM characterization was used to examine the interfacial adhesion of the composites prepared form fibers subjected to different

treatments. SEM analysis was also used to observe the fracture surface of the ARFRPCs samples after tensile testing as well as interfacial bonding between the AR fibers and the polymer matrix caused by the varying surface modification treatments applied. SEM analysis was also used to observe partial removal of fiber surface contaminants in the composite samples such as hemicellulose, waxes and oils by the thermo-alkali and thermo-laccase treatments. The extent to which the respective treatment removed the above-mentioned contaminants was noted, as well as comparing the untreated and treated ARFRPCS.

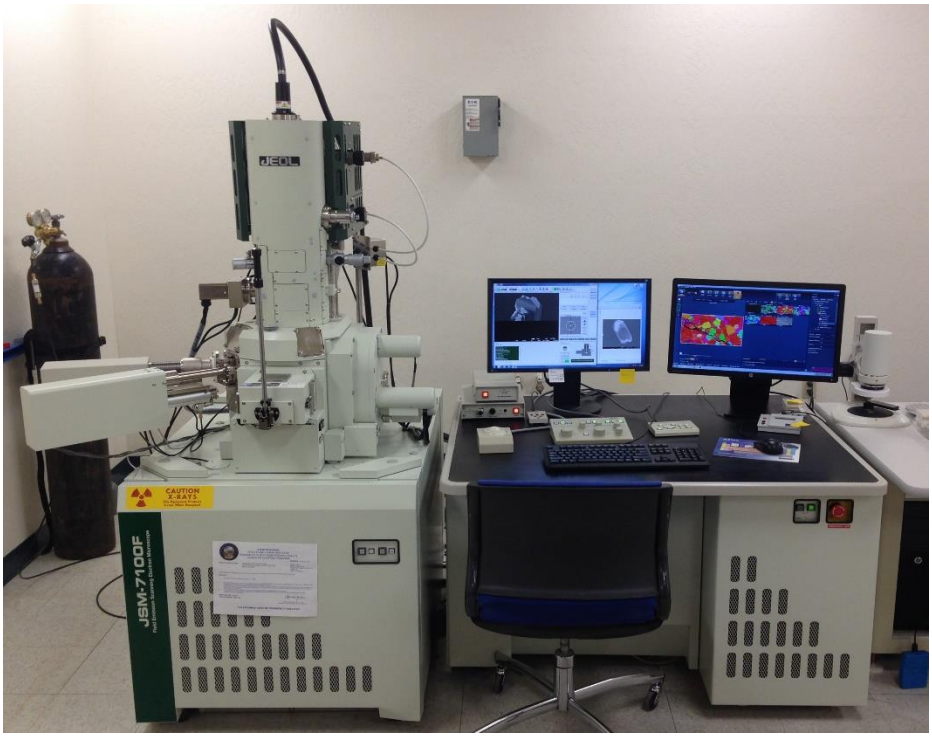

Figure 3-15 Jeol JSM-7100F Thermal field emission Scanning Electron Microscope

Samples from the five different treatment methods were examined to ascertain the observed phenomena. Samples were mounted on aluminium holders using a double sided electrically conducting adhesive tabs and coated with chromium using a Q150T Turbo-Pumped Sputter Coater/Carbon Coater prior to analysis.

Figure 3-16 Q 150T ES

3.8.2 Water absorption studies

The composites were tested for water absorption using the EN ISO 62:2008 standard. Each treatment method had five specimens submerged in distilled water for 28 days at room temperature. The specimens were removed from the water after 24 hours, dried, and weighed on a digital scale right away. This procedure was repeated for a total of 28 days. Water absorption was calculated using the following equation:

$$m_t(\%) = \frac{m_t - m_i}{m_i} * 100 \qquad \textit{Equation 3-1}$$

Where m_t is the weight of the sample at time t during water immersion and m_i is the weight of the dry sample at initial time.

3.8.3 Thermal Properties

3.8.3.1 Thermal Conductivity

Thermal conductivity refers to a material's ability to transport heat from one area to another. Thermal insulators possess a low thermal conductivity and allow little heat to pass though from one place to another. The thermal conductivity of composites is influenced by a number of parameters, such as the microstructure of the polymer matrix, the characteristics and morphology of ARFs, and the fiber-matrix interfacial adhesion. Thermo-alkali and thermo-laccase treatments were used to alter the microstructure of the composites; therefore, this test analyses the effect of the treatment on the thermal conductivity of the ARFRPCs. For this study a Linear heat conduction unit H112A and six thermocouples with an accuracy of 0.1 °C were used to measure thermal conductivity as seen in Figure 3-17.

Figure 3-17: Linear heat conduction unit H112A

Samples were cut into a circular shape of 5 cm diameter, and an average of 5 mm thickness measured using a vernier calliper as shown in Figure 3-18. The measurements were made under steady-state circumstances, which took roughly 5 to 6 hours from start to finish.

Figure 3-18: (a) Thermal conductivity sample (b) Measuring sample thickness using veneer callipers.

The process involved the samples being put between the cold and hot surface, and a temperature profile drawn to analyse the transfer of heat through the sample as shown in Figure 3-19.

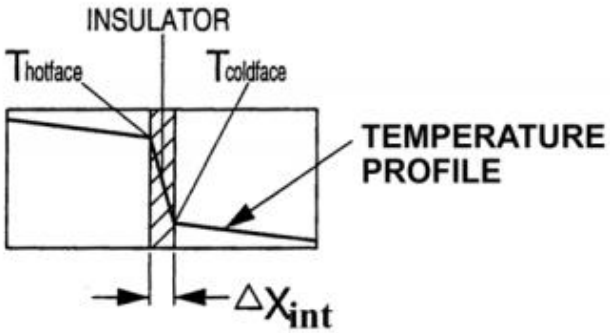

Figure 3-19: Thermal Conductivity Experimental setup

Thermal conductivity, k was calculated using:

$$k = \frac{Q\Delta x_{int}}{A_{int}(T_{hotface} - T_{coldface})} \qquad \textit{Equation 3-2}$$

where:

k = Thermal Conductivity

Δx_{int} = Composite sample thickness

A_{int} = Cross section are of composite sample

Q= Heat Transfer

$T_{hotface}$ = Temperature difference across hot surface

$T_{coldface}$ = Temperature difference across cold surface

3.8.3.2 Thermo-Gravimetric Analysis

Thermo gravimetric analysis (TGA) was carried out on 1 g ARFRPCs samples at a heating rate 10 °C/min in a nitrogen atmosphere using a LECO Corporation TGA 701 thermogravimetric Analyzer shown in the Figure 3.20.

Figure 3-20 TGA 701

The crushed composite samples were subjected to TGA in high purity nitrogen under a constant flow rate of 10 cm^3min^{-1}. Thermal decomposition of each sample transpires in a programmed temperature range of 30–950 °C. Continuous records of weight loss and temperature were determined and analysed with a view to determining the following TGA indices: onset temperature (T_{onset}/°C), peak temperature (T_p/°C), temperature at 50% mass degradation ($T_{50\%/°}$C), and degradation temperature range (°C). The point at which a material begins to disintegrate is known as the onset temperature, and it is a measure of that substance's thermal stability. When the largest component in the material decomposes, the temperature reaches its peak.

3.8.4 Physical and Chemical Characterisation

3.8.4.1 Density

The bulk density is the ratio between the dry weight of the samples and the volume in kg/m^3, and it was accurately calculated on the samples using EN 323 [54] and EN 1602 [55] standards; their length and thickness were determined using EN 822 [56] and EN 823 [57] recommendations. The

volume dimensions (Length *width*height) of the composite samples of were measured with a vernier calliper with a 0.5 mm accuracy to obtain the density. The mass was measured with an electronic scale with an accuracy of 0.1g.

The equation of density is:

$$\rho = \frac{L*W*H}{m} \qquad \textit{Equation 3-3}$$

For each treatment method, five specimens were used, and an average value of density calculated.

3.8.4.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

Fourier Transform Infra-Red Spectroscopy (FTIR) with a microscope was used to examine pulverised ARFRPCs at a molecular level. The FTIR equipment (Thermo-Scientific Nicolet iS10 smart iTR model) was used to obtain the chemical structural analysis of the ARFRPCs samples. The treated and untreated composite samples U, L5, L10, A5 and A10 crushed using a pulveriser as mentioned in section 3.4 for the infrared measurement. The FTIR spectra was collected with equipment in transmission mode, with a resolution of 4 cm^{-1} scans in the range of 4000–400 cm^{-1}. FTIR was used to observe the molecular vibration to examine the distribution of functional groups.

3.8.4.3 X-Ray Diffraction (XRD) Analysis

XRD was used to determine the effect of the treatments and crystallinity index (CI) and crystallite size (CS) of the treated and untreated ARFRPCs. The test was carried out using a D8 Advance Diffractometer by Bruker Technologies with the automatic data storing capability.

Figure 3-21: D8 Advance Diffractometer

An X-ray detector scanned between 10.0000° to 60.0106° (2Θ angle). The generator was functioning at 40kV and 40 mA at room temperature of 25°C. The method used to determine the crystalline index (C.I), equation (3.6) was the empirical method proposed by Segal *et al*. in 1959 (Chen, et al., 2018), which is:

$$C.I = \frac{I_{200} - I_{am}}{I_{200}}$$

Equation 3-4

where I_{200} is the maximum intensity of the (200) lattice diffraction; and I_{am} is the intensity diffraction of the amorphous band.

3.9 Conclusions

The research's experimental setup was carried out in accordance with the relevant standards and research guidelines. The sorghum agriculture residue fibers size range was determined using sieves, as described in this chapter. The water retting and treatment of the sorghum ARFs was

completed. 1wt% percent NaOH and laccase enzyme both followed by heat treatment. For varying treatment types and durations, the sorghum fibers reinforced rLDPE composites were manufactured under compression moulding. The physical, chemical, thermal, and mechanical property tests of composites were performed in accordance with the ASTM standards. The surface morphology, fiber breakage and interfacial adhesion of the composites analysis were studied by SEM.

CHAPTER 4: RESULTS AND DISCUSSION

4.1 Chapter Summary

Because composite materials used as building insulation materials are subjected to a variety of environmental conditions (moisture, temperature etc.) when in use, it's critical to assess the endurance of ARFRPCs after exposure to stresses such as moisture and weight. Five different experimental composites' physical properties, thermal properties, mechanical properties, and interfacial adhesion are examined in this chapter. The composites are made of fibers extracted from sorghum residues straw treated thermo-alkali and thermo-laccase, and the matrixes are made of rLDPE. The major goal of the research given here is to compare key attributes of composites in order to select the one that has the best overall performance for use as a building insulation material.

4.2 Density

The densities of the ARFRPCs are shown in Figure 4-1. Composites reinforced with treated fibers are characterised with greater density compared to composites incorporated with untreated fiber. When comparing the two treatments, alkali treated fiber produced more intact composites with a greater density by a factor of 0.01 g/cm^3. As the duration of treatment exposure is increased from 5 to 10 days, densities of both treatment options increased. Both thermo-alkali and laccase treatment causes non-cellulosic material found in the inner structure of ARFs to be removed from the fiber, resulting in a loss of hydrophilicity and smoothness, as well as improved adhesion and compatibility with polymers in the composite production process [180]. The degradation of low-density non-cellulosic materials leads to a reduction in fiber diameter, and the remaining high-

density cellulose subsequently leads to increasing density of ARFs. There are little differences between the densities of composite L5 and L10, as well as between A5 and A10.

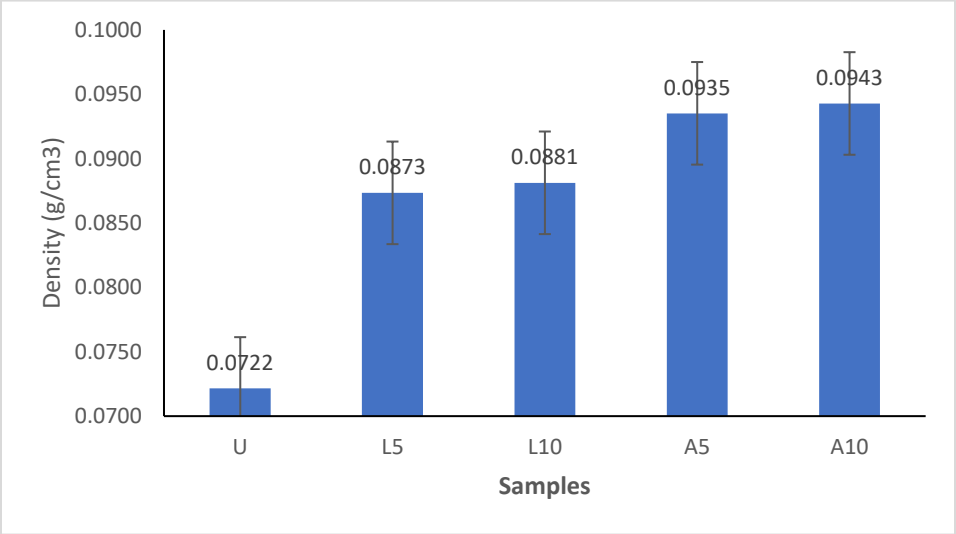

Figure 4-1: Composite Samples Density Graph

The results demonstrate that the composite made of the fiber treated thermo-alkali for 10 days had the best density of 0.0943 g/cm^3 signifying satisfactory compaction and adhesion between the fiber and matrix.

4.3 Moisture Absorption

Moisture is a problem for ARFs [27], and cellulose, the main constituent, has a lot of hydroxyl groups that are very hydrophilic. Under ambient temperature and humidity conditions, the water content of fiber reinforced composites is around 10-20 percent, reducing their mechanical characteristics significantly [95]. The absorbed moisture can cause the matrix to plasticize and modify the condition of stress, resulting in the production of cracks as a result of swelling [9]. The

rate of water absorption in composites is affected by factors such as fiber/matrix interface, reaction between water and the matrix and other variables [64]. Surface modification of fibers influence the rate of water absorption as it's intended to reduce the hydrophilic nature of fibers. Results of the composites processed using rLDPE and treated sorghum residue fibers are shown in Figure 4-2.

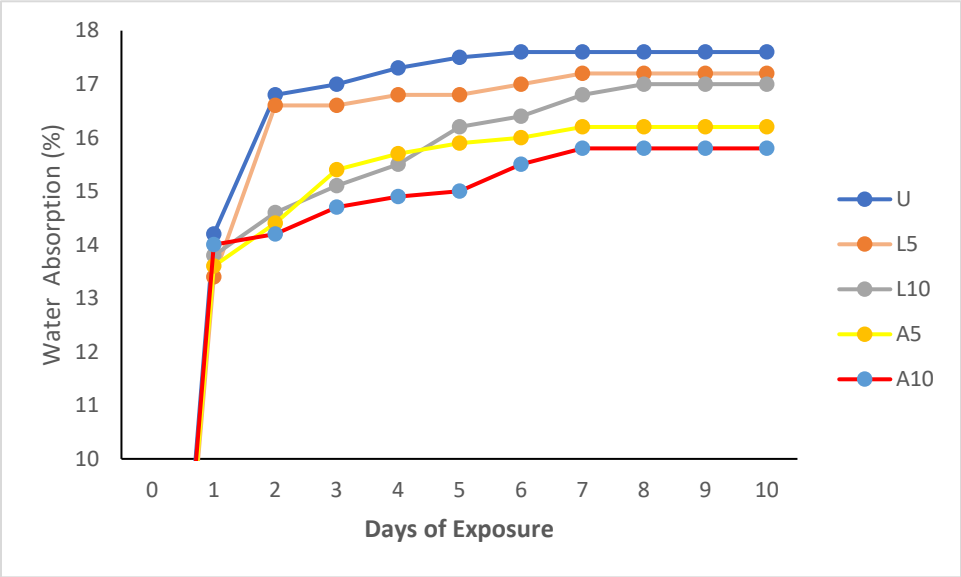

Figure 4-2: ARFRPCs Water Absorption Line Graph

The percentage of absorbed water for all sorghum residues fibers reinforced composites were determined by calculating the difference between the weight of water immersed and dry samples by the equation below, and results are given in Table 4-1.

$$\text{Water Absorption, \%} = \frac{m_t - m_i}{m_i} * 100\%$$

Where m_i is the weight of the dry sample at initial time and m_t is the weight of the sample at time t during water immersion.

Table 4-1: ARFRPCs Water Absorption

Sample Code	Water Absorption (%)
U	17.60
L5	17.20
L10	17.00
A5	16.20
A10	15.80

The ARFRPCs samples were immersed in distilled water for 10 days at 23 °C. The obtained results of the composite from untreated fibers were compared to the composite processed with treated fibers. The water uptake process is linear in the beginning and approaches saturation after a period of 5 days, as shown in Figure 4-2. Due to the high hydrophilicity of the untreated fibers, the composite with untreated fibers, U, has the highest water uptake of 17.60%. It's observed that as the treatment of fibers is introduced, and exposure duration increases, so does the water intake. Similar to density results, the thermo-alkali treatment yields an impressive rate of reduced water absorption in comparison to thermo-laccase and untreated reinforce composites due to removal of waxes, hemicelluloses that increases hydrophobic nature of the ARFs. Introduction of fiber surface modification reduced the water absorption rate by up to 2%. Composite A10 presented a water absorption rate of 15.80 whereas composite L10 had a water absorption rate of 17%. Following that composite L5 and A5 had a water uptake of 17.20 and 16.20% respectively.

4.4 Thermal Conductivity

The thermal conductivity of the five composites is measured using a surface probe on a Linear Heat Conduction unit. The measurements are done in pentaplicate on 0.025m diameter specimens at room temperature (20℃; 50% humidity).

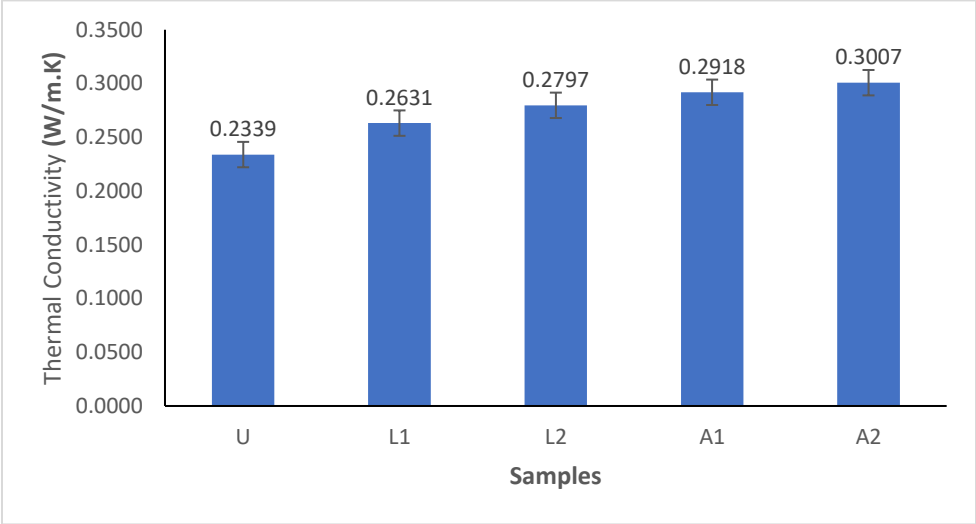

Figure 4-3: ARFRPCs Thermal Conductivity (W/m.K) Line graph

Table 4-2 shows the mean values obtained, as well as the values of density obtained from various treated composite materials.

Table 4-2: ARFRPCs Thermal Conductivity , k (W/m.K)

Sample	Density (g/cm$^{3)}$	K (W/m.K)
U	0.0722	0.233
L5	0.0873	0.263
L10	0.0881	0.279
A5	0.0935	0.291
A10	0.0943	0.300

Composites made from fibers treated have a greater thermal conductivity compared to untreated fiber composites. Between the two types of treatments, thermo-alkali treated fiber produced more intact composites with a greater thermal conductivity by a factor of 0.07 W/m.K As the duration of treatment exposure is increased from 5 to 10 days, thermal conductivities of both treatment options increased. There are no significant differences between the thermal conductivity values of composite L5 and L10, as well as between A5 and A10.

Irrespective of the treatment used, composites comprising treated ARFs have the highest thermal conductivity while those containing untreated fibers having the lowest. Fibers treated for 10 days, irrespective of the type, have the highest value (0.279 and 0.3 W/m.K) compared to composites treated for 5 days with 0.263 and 0.291 W/m.K Even though, the findings are higher than other commercially available insulation materials such as glass fiber blanket (0.039 W/m.K), cork board (0.043 W/m.K) and expanded polystyrene (0.029 W/m.K) to mention a few [181], they can be compared to those obtained from ARFs reinforced polymer boards. Studies from Chandramohan *et al*. [182] found the thermal conductivity of a aloe vera composite to be 0.206 W/m.K. Ramlee

et al. [17] also stated that composite with OPEFB fiber thermal conductivity reduces from 0.5 to 0.27 W/m.K after surface modification treatment using sodium hydroxide, according to the findings.

The high thermal conductivity as treatment is introduced and duration increased is explained by the fact that treating ARFs increases density, which plays a major role in thermal conductivity of a material. The first reason for improved thermal insulation is that ARFs have a natural tendency to limit heat transfer due to porosity in their microstructure [137]. These pores are filled with air, a good heat insulator (0.026 W/m.K) [183]. This implies that if the quantity of air content in external wall is expanded, the thermal insulation must also rise. Materials with lower density leads to lower thermal conductivity, therefore provide better thermal insulation as seen in where thermal conductivity of loose-fill sugarcane bagasse fibers insulation material with densities of 100 kg/m3 and 120 kg/m^3 was found to be 0.0468 and 0.0496 W/m.K respectively [184].

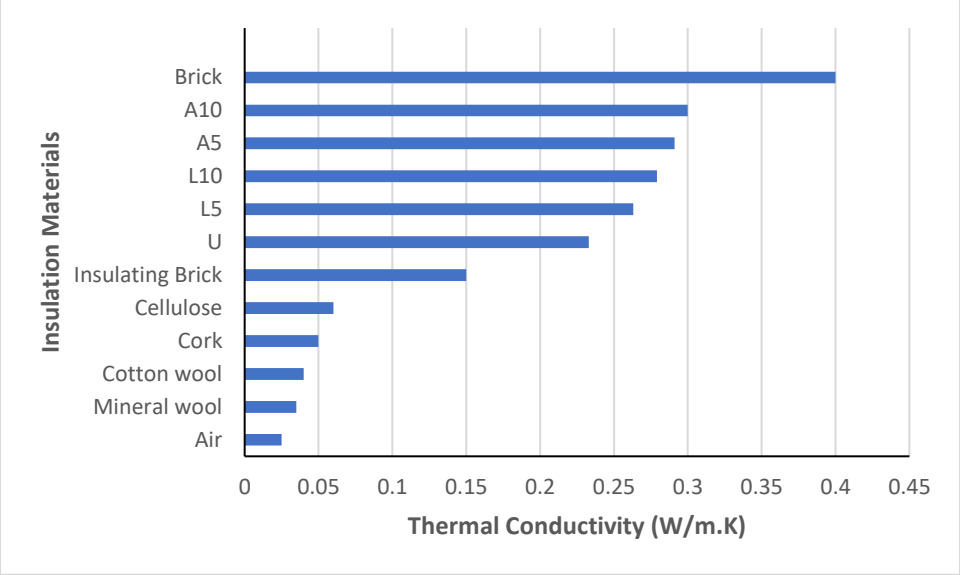

Figure 4-4: Comparison of Insulation Materials Thermal Conductivity Values

Looking at the data, the thermal conductivity of the composites have a thermal conductivity of 0.23-0.3 W/m.K This would mean that the conductivity of this ARFRPCs U, L5, L10, A5 and A10 is higher than conventional insulation materials like mineral wool has a conductivity of 0.035 W/m.K. and cork which has a thermal conductivity of 0.05 W/m.K. [185]. The composites, however, have lower thermal conductivities than which has a thermal conductivity of 0.4 W/m.K/ as depicted in Figure 4-5. These results prove that although the overall thermal conductivities of other composites are lower than concrete and dense brick, they are higher than conventional materials as stated. This is due to the fact that the fabrication process used, compression moulding produces dense material as opposed to the foamy state conventional materials are usually in. Another possible reason is the high polymer content as opposed to the fiber content ratio of 60:40. ARFs cellulose has a low thermal conductivity of 0.05-1.00 W/m.K and rLDPE possesses a thermal conductivity of 0.4 W/m.K as stated in section 3.2.2. Therefore, the polymer thermal conductivity value had a greater influence of the final ARFRPCs material thermal conductivity value.

4.5 Thermogravimetric Analysis

TGA was carried out at a temperature of 30 ℃ to 900 ℃ at a rate of 10 ℃/min. A mass of 1 g was utilized per sample in the experiment.

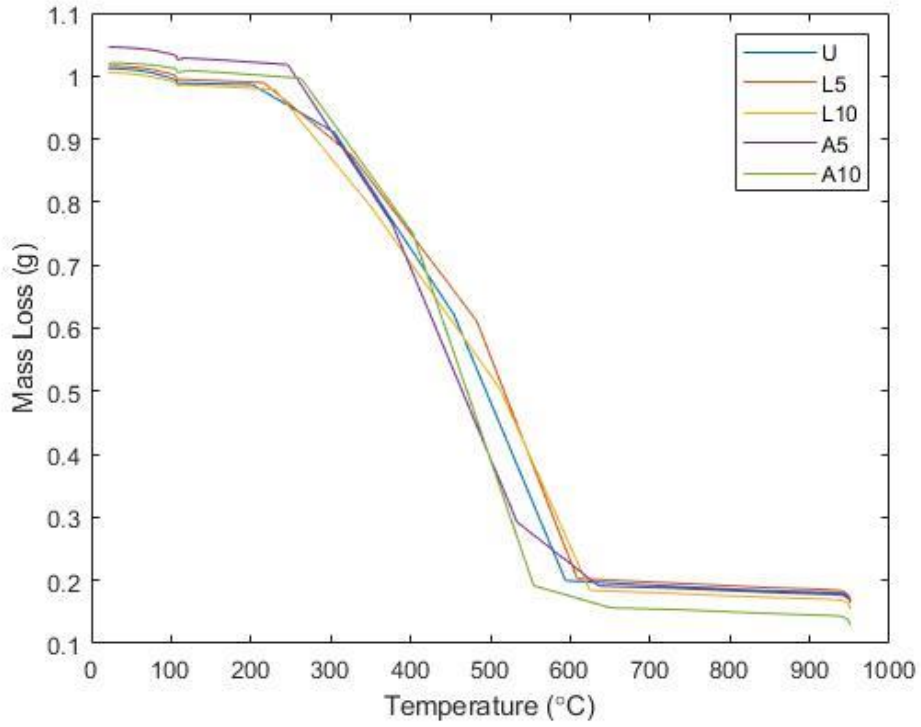

Figure 4-5: ARFRPCs Thermograms

Figure 4-6 shows the thermogram for thermo-alkali and thermo-laccase treated ARFRPCs with a temperature range of 100°C to 900°C. The degradation graph is derived from mass loss, which is directly proportional to the chemical composition of the composites [186]. As a result of moisture contained in the composites, which vaporized with a mass loss of 3%–10%, the degradation peak of the composites occurred at 100°C–120°. The second peak occurred at 210°C–260°C, with a mass loss of 12%–18%, revealing hemicellulose breakdown lignin found within the structure of the ARFs. The TGA graph shows that the hemicellulose deterioration continues until 350°C. Hemicellulose degradation made up to approximately 45-50% of the mass loss. Following that is lignin degradation from 400 to 600°C in the thermogram [187]. Lastly in the graph is the thermal degradation of residual materials such as the wax and char.

The specific degradation temperatures of composites U, L5, L10, A5 and A10 outcomes are shown in Table 4-3 so as to observe the effects of ARFs treatments on the composites. The onset temperature (T_{on}) is the temperature where loss of mass begins and is caused by loss of water in the materials. The maximum degradation temperature (T_{max}), which is considerably more significant, is attributed to cellulose thermal degradation, and could be attributable to lignin decomposition.

Table 4-3: ARFRPCs TGA Analysis Results

Composites	Onset Temperature (T_{on}), °C	Maximum Degradation Temperature (T_{max}), °C	Residual, %
U	139.3	275.1	5.88
L-5	226.9	300	6.61
L-10	255.2	410.1	6.81
A-5	283.5	441.5	7.28
A-10	350.3	442	8.44

Composite U breaks down, more quickly than treated composites, with an onset temperature of 139.3℃. The onset temperature is increased by introduction of fibers treatment as conveyed by

both thermo-alkali and thermo-laccase treated composites showing a greater and increasing T_{on} which proves that thermal stability can be improved by surface modification of ARFs which improves porosity and interfacial adhesion of the ARFs/rLDPE matrix. Furthermore, thermo-laccase treated composites, L5 and L10 have lower T_{on} and T_{max} than the thermo-alkali treated composites, A5 and A10. Consistent with the density and moisture absorption results, composite A10 displays the highest degradation temperature of 442℃ compared to L10 (410.1 ℃) and U (275.1 ℃).

4.6 Fourier transform infrared Analysis

The functional groups prevalent in the structure of composites reinforced with untreated and treated agricultural residue fibers are studied using FTIR spectral analysis. Figure 4-6 depicts the results. Peaks at (3329, 2888, 1424, 1018, 343cm^{-1}) are the most prevalent and common cellulose characteristic peaks seen in samples U, L5, L10, A5 and A10. The C-O stretching vibration of the cellulose backbone C-OH is ascribed to the emergence of peaks at 1018 cm^{-1} with higher intensities in the composite reinforced with an untreated ARFs in comparison to the treated ARFs. Furthermore, the bands at (3329 and 2888) cm^{-1} can be attributed to the stretching of hydrogen bonds of the ARFs, specifically the (O-H group). The CH_2 symmetric bending peaks at 1432 cm^{-1} are also exhibited by the spectra. The trend exhibited by the composite samples is the presence of the above-mentioned peaks, however, there is varying intensity depending on the lack or there of treatment.

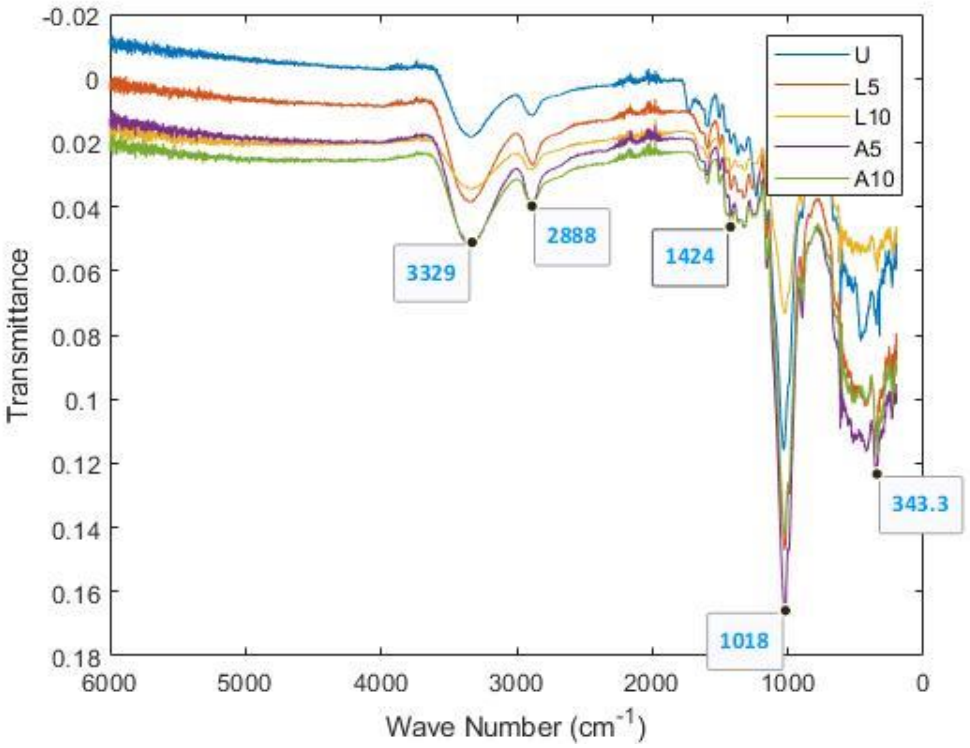

Figure 4-6: IR Spectra of Treated and Untreated ARFRPCs

The spectra for thermo-alkali treatments, show identical absorbance peaks to the thermo-laccase treatment samples with minor intensity differences. This reveals that the treatment caused the amorphous parts of the ARFs to dissolve even further. The absorbance intensity increases from 5 to 10 days after both thermo-alkali and thermo-laccase treatment, indicating a decrease in intermolecular/intramolecular hydrogen bonding between the hydroxyl groups of cellulose and hemicellulose in the ARFs [188]. The removal of the non-cellulosic components from the ARFs structure is helpful in the fiber-polymer interfacial adhesion necessary for good stress transfer in the composite material.

4.7 X-Ray Diffraction Analysis

All the composites studied had their crystallization size (CS) and crystallinity index (CI) calculated. Table 4-4 presents the results for the crystallization properties. Figure 4-8 shows the XRD spectra of the composites. Adding ARFs filler to a rLDPE matrix reduces the diffusion rate of the rLDPE chain and reduces the crystallinity of the polymer consequently affecting the crystallinity of ARFRPCs composite materials [55]. Treating fibers helps to reduce this by increasing crystallinity of fibers and ultimately increasing the crystallinity of the composites.

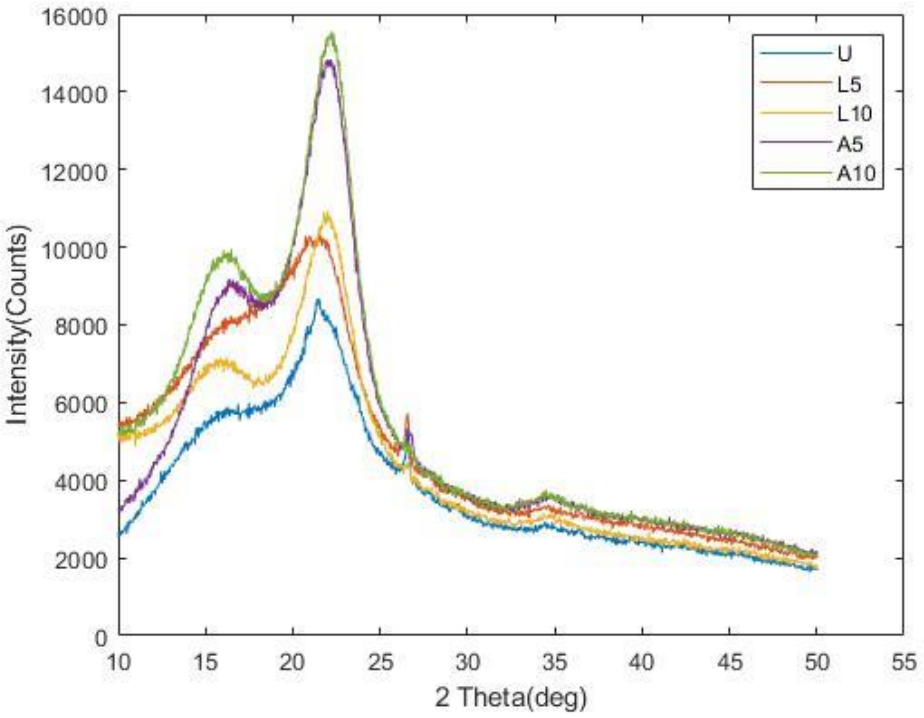

Figure 4-7: XRD Patterns of the treated and Untreated ARFRPCS

Figure 4-8 depicts the peak detected in XRD diffractograms of ARFRPCs at the (0 0 2) plane (2 = 22.5°), which is more acute and intense in composites reinforced with treated fibers, L5, L10, A5, A10, than in untreated fibers, U. A small peak can also be noticed at 35°, which corresponds to the fiber alignment and orientation [189]. The sharper peak for the (0 0 2) plane, which corresponds to an improved crystal lattice perfection [190], is more prominent in composites reinforced with thermo chemically treated and thermo biologically ARFs treated than in composite sample U.

Composite L5, L10 ,A5 and A10 depict (1 0 1) plane (2 Theta = 15.1° and 16.4°) peak that is significantly higher. With increasing treatment duration, the intensity and sharpness of the (002) plane (2 Theta= 22.5°) peak increases. Composites reinforced with fibers treated for 10 days had a more pronounced peak at the shoulder of the peak about 2 Theta= 20° than composites reinforced with fibres treated for 5 days. The increasing intensity of both peaks with exposure time, demonstrates that the quantity of tiny crystallites reduces during surface modification treatment, with superior results on sample A10 with stronger peaks.

Table 4-4: Crystallinity index (CI) and Crystalline size (CS) values of ARFRPCs.

Sample Name	Crystallinity Index (%)	Crystalline size
UM	29.73	8.34
L5	32.81	11.44
L10	36.33	13.23
A5	41.50	12.38
A10	48.62	15.34

The composites' CI values ranged from 29.73 percent to 48.62 percent, with the lowest being composite U (29.73) and the highest being composite A10(48.62), which was treated thermo-alkali for 10 days. When comparing the treatment methods, thermo-laccase produced a composite with 32.81 and 36.33 percent whilst thermo-alkali produced an outstanding 41.50 and 48.62 percent. The two treatments saw an increasing crystallinity with an increasing treatment duration from 5 to 10 days indicating a more organized cellulose structure. The treatments aid in producing a more

ordered cellulose structure by removing lignin and hemicellulose from the ARFs. Scherer's formula, as mentioned in section 3.8.4.3, was used to determine the CS of the composites. The composite samples' values ranged from 8.34 nm to 15.34 nm, with the lowest being attributed to the composite reinforced with the untreated ARFs and the highest value imparted into the composite reinforced with thermo-alkali treated ARF for 10 days. This therefore leads to a composite with a good interlocking and interfacial adhesion between the ARFs and rLDPE meaning an improvement of the composite material's mechanical properties [191].

4.8 Tensile Testing

Fiber-matrix adhesion, fibre aspect ratio and stress transfer at the interface are all elements that affect composite qualities. When it comes to optimising the mechanical properties of the composites, the fiber and matrix qualities are crucial. The interfacial adhesion between the fibre and the matrix determines the tensile strength, whereas the modulus is determined by the fiber characteristics and content. Tensile characteristics of composite specimens U, L5, L10, A5 and A10 are tested, and the results are reported in Figure 4-9 to 4-11 including elongation at break and tensile strength.

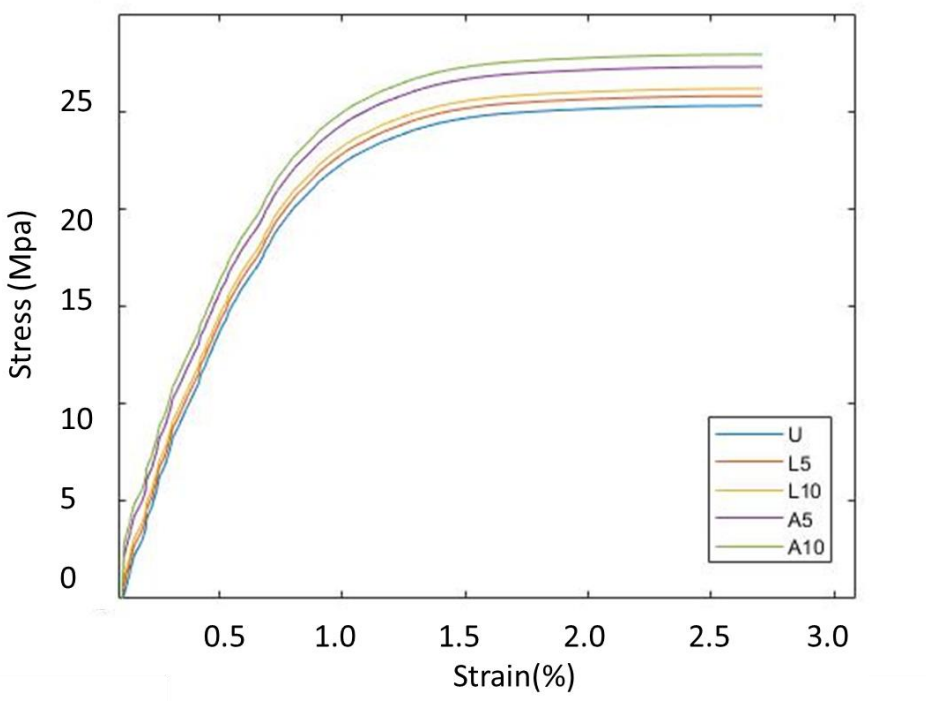

Figure 4-8: Stress-Strain Curves for ARFRPCs

The tensile stress of the ARFRPCs increased with the introduction of the combination surface modification treatment as proven by literature that hybrid treatments improve mechanical properties of ARFRPCs [188]. Furthermore, with the increase of treatment duration, this tensile strength improves as seen in the stress strain graph. The tensile strength trend observed in the graph is comparable to those measured by other researchers such as Sever *et al* [192] who reported that treatment of jute fibers improved the jute/HDPE composites' mechanical properties, despite the fact that the process of ARFs extraction and composite fabrication method differs. The ARFRPCs tensile strength is determined to be optimal at thermo-alkali treatment at 10 days compared to thermo-laccase treated composite for the same duration. The reason for this being that the surface modification treatment impacts the optimum surface roughness, removal of hemicelluloses and lignin from the ARFs after prolonged exposure to compensate for the low concertation of 1wt%.

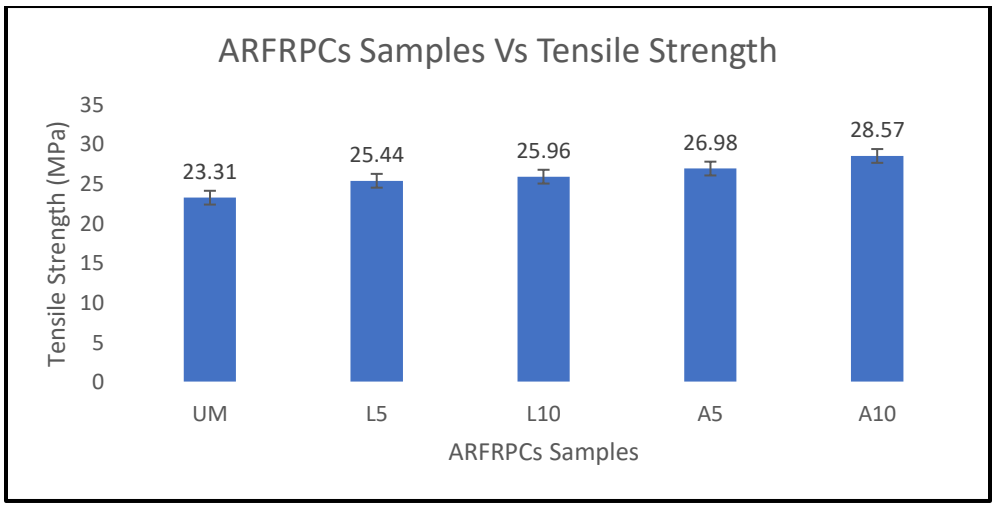

Figure 4-9: ARFRPCs Samples Tensile Strength

Untreated ARFRPCs have lower mechanical performance versus treated ARFRPCs, regardless of the treatment type. This implies that both thermo-alkali and thermo-laccase treatment improved the microstructural properties of the ARFs and consequently improving the mechanical properties of the resultant composites. As the duration of treatment exposure is increased from 5 to 10 days, the tensile characteristics of the composites improve, while their elongation at break decrease as shown in Figure 4-10. Surface roughness and removal of these substances results in greater interfacial interlock between the rLDPE matrix and ARFs. The sorghum residues fibers rLDPE polymer composites have comparable tensile strength to other ARFRPCs produced from similar fibers such as bagasse.

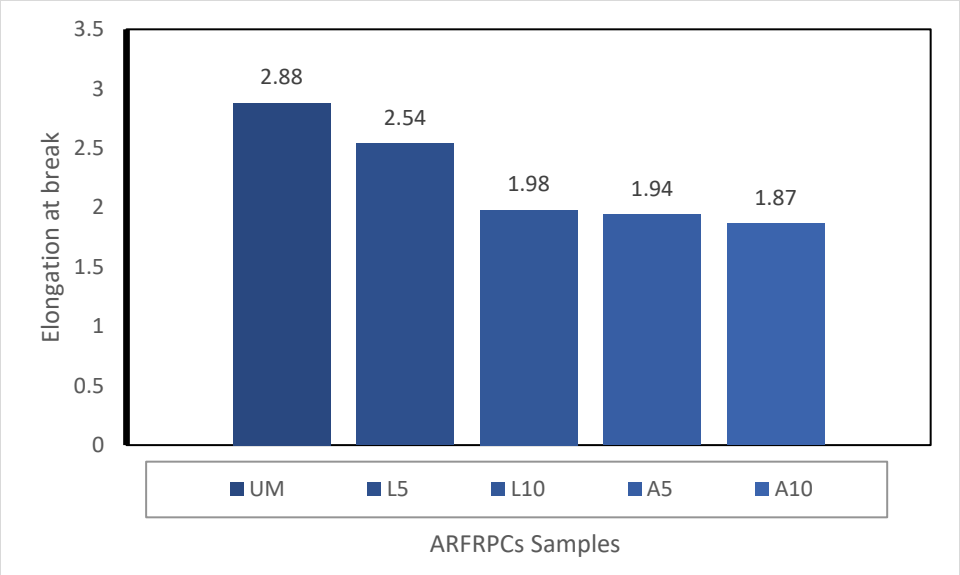

Figure 4-10: ARFRPCs Samples Elongation at break

When comparing the two treatments, the mechanical qualities provided by thermo-alkali treatment produced better results as opposed to thermo-laccase treatment. Composites A5 and A10 have lower elongation at break and higher tensile strength values compared to composites L5 and L10. The tensile strengths of the composite from this study that were subjected to thermo-alkali treatment for 10 days was 28.57 MPa. These values are considerably lower than the more popular fiber polymer composites but are comparable to other ARFRPCs such as corn/propylene composites which had a tensile strength of 30.41 MPa [193]. However, it is worth noting that other factors affect the tensile strength as reported by the reviewed literature such as fiber content and length, fabrication process such as injection moulding which produces stronger and evenly compacted composites compared to compression moulding.

4.9 Scanning Electron Microscope Analysis

The SEM micrographs of the ARFRPCs specimens used to assess the nature of inter-facial adhesion which determines its mechanical properties are as shown in Figure 4-11. As seen in Figure 4-11, tensile tension caused ARFs to rupture and the ARFs-rLDPE link to break. To investigate the morphological changes caused by the thermo-alkali and thermo-laccase treatment, untreated ARFRCPs Figure 11(a) was used as a reference sample.

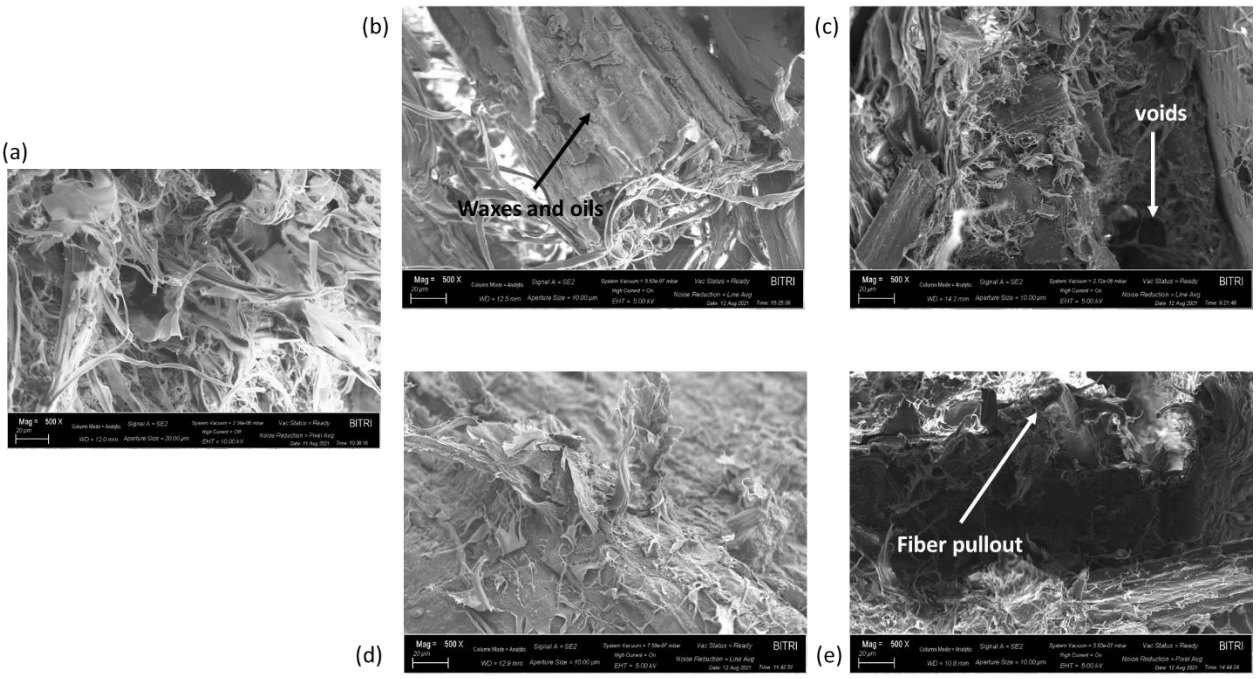

Figure 4-11: ARFRCPS SEM analysis (a) Untreated sample ,U (b) Thermo-laccase treatment for 5 days (c) Thermo-laccase treatment for 10 days (d) Thermo-alkali treatment for 5 days (e) Thermo-alkali treatment for 10 days

It is evident that the treated composites Figures 4-11 (b-e) composites have enhanced and better adhesion between fiber and matrix when compared to untreated composites with considerable numbers of voids (Figure 4-11a). The lower number of voids indicates higher-quality composites, as evidenced by the tensile strength results. In comparison to thermo-laccase treated ARFPCs seen

in Figure4-11 b & c, the thermo-alkali treatment eliminated undesired contaminants in the ARFs, as well as generated roughness in the ARFs, which improved the adhesion between fiber and matrix. Surface modification treatment have a significant impact on the morphology and consequently the fracture behaviour of ARFRPCs. González-López *et al*. [107] used a high magnification SEM to examine the cracked surfaces of cellulose-PLA composites and found fiber pull out and void patterns on the tensile-tested samples similar to Figure 4-11 (c). Untreated ARFs is seen to have a reduced binding property with the rLDPE, while composites treated with thermo-alkali and thermo-laccase depict a homogenous fiber/matrix formation. The incretion of treatment from 5 to 10 days resulted in improved fiber surface roughness and removal of waxes and oils leading to fewer deformations and a uniform dispersion of ARFs in the matrix. The characteristics are further improved by the 1wt% thermo-alkali treatment for 10 days compared to 5 days.

This is in line with the findings of Malenab *et al*. who treated waste abaca fibers with a 6 wt. % sodium hydroxide solution. The formation of a rough surface on the fibers as a result of pre-treatment makes them have a smaller surface area. As a result, the alkali-treated ARFs had a larger contact area and a stronger binding to the rLDPE matrix. The features of roughness to the interface between the fiber and the matrix seen in Figure 4-11 support this conclusion. In L5, L10, A5 and A10 composites with thermo-alkali and thermo-laccase treatment, cracks and fractures are visible in the fiber/polymer matrix near the fiber inclusion locations, indicating that plastic (viscous) fracture is prevalent. It's worth noting that the treated ARFs composite maintain their structure in the fracture zone, showing that they're stronger than the untreated ARF composite. Therefore, it is worth noting that the use of mixed thermo-alkali and thermo-laccase treated ARFs may be able to help produce even bigger improvements in ARFRPCs strength.

4.10 Conclusion

ARFs derived from sorghum residues stalks were used to reinforce rLDPE polymer composites. Thermo-alkali and thermo-laccase treatment at two different exposure time, 5 and 10 days were applied to improve mechanical and thermal properties in ARFRPCs in this study. With peaks at 3329, 2888, 1424, 1018 and 343 cm^{-1}, FTIR studies verified the high cellulose content in functional groups of the ARFRPCs. Mechanical studies showed that incorporating surface modification treatment into ARFRPCs improved the tensile strength of the composites. The treatments also improved the thermal stability of the composites with the thermo-alkali treatment being the best treatment at increasing the degradation temperature and thermal conductivity of the composites.

CONCLUSION AND RECOMMENDATIONS

5.1 Chapter Summary

The primary objective of this thesis was to produce a practical thermal insulation building material made from sorghum agriculture residue fibers and recycled polymers. This composite material has the advantage of being partially biodegradable and made from readily available renewable components. The effects of thermo-alkali and thermo biological treatment using alkali treatment and laccase enzyme respectively on the materials microstructure, physical, chemical, mechanical, and thermal properties were investigated using several experiments. It was observed that there are common results and recommendations to be made from all of these experiments.

5.2 Conclusions

The following conclusions from the research were reached:

1. ARFs provide a number of advantages to synthetic fibers, including lower cost, better environmental performance, and a higher specific modulus. ARFs, on the other hand, have limitations such as low shear interface strength, water absorption, biodegradation, and photodegradation, which limit the potential of ARFRPCs. Surface modification treatments of ARFs can help to mitigate some of the disadvantages.

2. The surface modification of ARFs promotes surface roughness, removes hemicelluloses, lignin, waxes, and oils promoting fiber- matrix adherence, which in turn improves the composite's mechanical properties. There exist various types of treatment namely chemical (alkaline, silane etc.), biological (enzyme, bacteria, fungi) and physical treatments (UV, heat, plasma etc.). Combining treatments was proven to produce better results by

improving mechanical and thermal treatments compared to one treatment thereby confirming the study of literature.

3. Treated sorghum stalk ARFs disperses well in the matrix producing a homogenous structure, improving the composite's water absorption, thermal conductivity, and tensile strength. The best combination of treatment among the tested composites is treating the fibers using thermo-alkali treatment for 10 days.

4. The thermal stability of rLDPE polymer composites reinforced with sorghum stalk ARFs was shown to be improved by surface modification treatments. The findings revealed that increasing treatment duration resulted in a significant increase in the values. The temperature of degradation was increased, emphasizing the removal of waxes and oils, surface roughness leading to increased fiber/matrix adhesion. Furthermore, the sorghum ARFs composites' thermal behaviour was enhanced by treatment, as evidenced by the three–step weight loss mechanism.
5. The use of thermo-alkali and thermo-laccase treatment on the ARFs to reinforce rLDPE polymer composites significantly improves the mechanical properties of the composites. With the increment of fiber treatment exposure time, the composite's mechanical properties such as tensile strength, microfibril angle and elongation at break improve as well. The optimum treatment method for the sorghum stalk ARFs was found to be thermal-alkali treatment for 10 days this investigation. The mechanical properties of fiber reinforced composites are determined by the interfacial region's stability. Alkali treatment reduced the ARFs surface contaminants leading to a good fiber-matrix interface, and decreased fiber pull-out, all of which improved tensile performances. The interaction between the untreated ARFs and the rLDPE matrix was poor, resulting in fiber debonding, fiber pull-out, matrix

fracture, and fiber fracture, according to SEM examination. The experimental tensile results for sorghum ARFs reinforced rLDPE composites were compared to other papers and were found to be comparable. In overall the material has a potential to be used as a building insulation material although a few properties such as density need further study to produce a material that is competitive with synthetic insulation materials.

5.3 Recommendations for Future Study

The composite was constructed using a compression moulding technique in the current study. For polymer matrix composites, however, there are various manufacturing methods. More fabrication methods could be tried and examined in order to arrive at a final judgment. The data presented in this thesis, on the other hand, can serve as a foundation for the use of this ARF.

Surface modification of the fiber with thermo-alkali and thermo-laccase greatly increases the mechanical performance of the composite, according to this research. Other ARFs surface modification techniques which are low in hazardous chemicals and low in operational costs, such as silane treatment, bacterial and fungi treatment may be put to the test, and a definitive conclusion reached as a result. Furthermore, only ARF particles of 100-300 μm were used in this study. This research can be expanded to include various particle sizes and lengths, so as to investigate the impact of particle size and type on composite properties.

It is very important to do research and test performance of engineering materials under various weather situations over prolonged periods. However, this study only looked at the ARFRPC material behaviours in short-term assessments. In the future, it is recommended that the long-term behaviour of sorghum ARFRPCs be investigated at different conditions such as temperatures, humidity, and stresses to predict the lifecycle behaviour of the composite materials.

Appendices

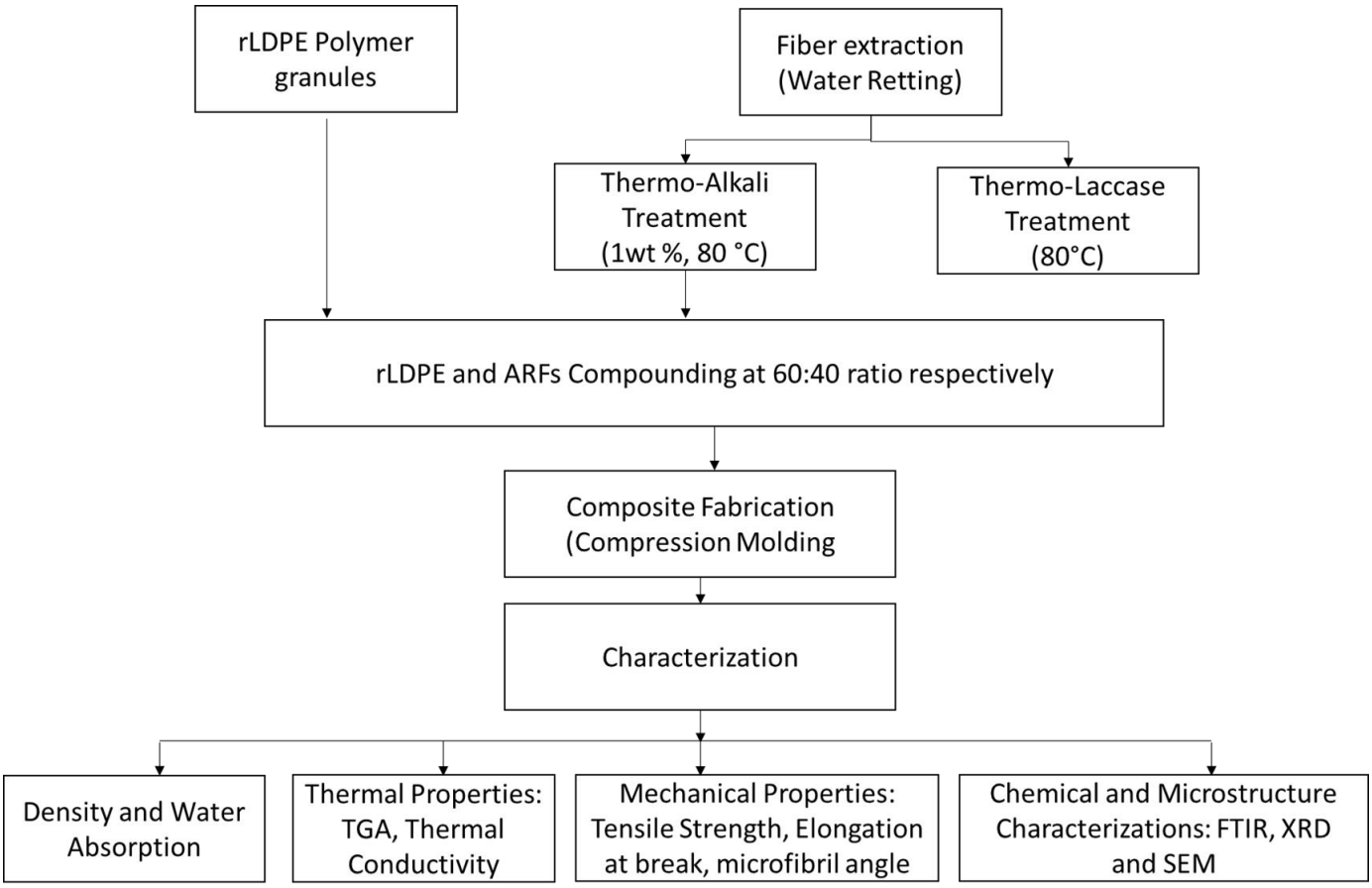

Figure 6-1: Methodology Workflow

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