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FABRICATION AND EXPERIMENTAL EVALUATION OF MOKOLWANE FIBRE-REINFORCED POLYESTER COMPOSITE FOR INDUSTRIAL APPLICATIONS

by

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I, **Keolebogile Seisa** declare that this dissertation/thesis is my original work and has not been submitted for any other degree or examination.

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The undersigned certifies that they have read and understood hereby recommend for acceptance by the Faculty of Engineering a thesis titled: ***Fabrication and Experimental Evaluation of Mokolwane Fibre-Reinforced Polyester Composite for Industrial Applications*** in fulfilment of the requirements for the degree of Master of Engineering in (Mechanical and Energy Engineering) of the BIUST.

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Date: 06/November/2024

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Date: 06/November/2024

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ABSTRACT

Natural fibres have captured the attention of researchers and industries alike, thanks to their renewability, widespread availability, biodegradability, and cost-effectiveness. However, despite these inherent advantages, natural fibre reinforced polymer composites encounter significant hurdles that hinder their utilization in semi-structural and non-structural applications, primarily due to their susceptibility to moisture absorption. In this study, we delve into the potential of Mokolwane fibre, an innovative indigenous fibre sourced from Botswana, as a reinforcement for polymers. The fibre undergoes comprehensive characterization, including tests for fibre diameter, moisture absorption, thermogravimetric analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscope (SEM) analysis. Subsequently, composite specimens are synthesized using Mokolwane fibre and Polyester resin, with variations in fibre loading and alkali treatment concentration to determine optimal conditions. Moisture absorption assessments reveal that higher fibre loading increases water uptake by the composites, while alkali treatments effectively reduce this absorption. Mechanical property evaluations demonstrate that composites fabricated with 50 wt.% fibre loading and 4% NaOH fibre treatment exhibit optimal performance, with tensile strength, flexural strength, and impact energy reaching 76.5 MPa, 124 MPa, and 3.83 J, respectively. The enhancements attributed to alkali treatment are substantiated by SEM and FTIR results, indicating improved fibre-matrix interfacial adhesion resulting from the removal of hydrophilic amorphous surface components. However, excessive alkali concentration proves detrimental to mechanical properties, as it damages the fibre surface. Furthermore, inadequate fibre loading leads to inadequate stress transfer, while excessive loading results in cracks, voids, and fibre agglomerations. Based on the study findings, the Mokolwane fibre and composite demonstrate suitability for integration into diverse applications spanning automotive, construction, and furniture industries. This research not only contributes to achieving the United Nations' Sustainable Development Goals but also underscores the utilization of readily available, indigenous natural resources in industrial applications, thereby promoting sustainability and resource efficiency.

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LIST OF EQUATIONS

NOMENCLATURE

°C	Degrees Celsius
MPa	Mega-Pascals
GPa	Giga-Pascals
cm	Centimetre
mm	Millimetre
µm	micrometre
h	hour
ρ	density
m	mass
V	volume
$W_A\%$	%Moisture absorbed
W_0	Initial weight of specimen
W_1	Final weight of specimen
T_s	Thickness swelling
E	Young's Modulus
σ_{max}	Flexural Strength
UPM	Untreated Mokolwane fibre reinforced polyester composite
UPM_40	40 fibre wt.% untreated composite
UPM_50	50 fibre wt.% untreated composite
UPM_60	60 fibre wt.% untreated composite
TPM_40_2SH	40 fibre wt.% treated composite by 2%NaOH
TPM_50_2SH	50 fibre wt.% treated composite by 2%NaOH
TPM_60_2SH	60 fibre wt.% treated composite by 2%NaOH

TPM_40_4SH	40 fibre wt.% treated composite by 4%NaOH
TPM_50_4SH	50 fibre wt.% treated composite by 4%NaOH
TPM_60_4SH	60 fibre wt.% treated composite by 4%NaOH
TPM_40_6SH	40 fibre wt.% treated composite by 6%NaOH
TPM_50_6SH	50 fibre wt.% treated composite by 6%NaOH
TPM_60_2SH	60 fibre wt.% treated composite by 6%NaOH

ABBREVIATIONS

PMCs	Polymer Matrix Composites
SDGs	Sustainable Development Goals
MEKP	Methyl Ethyl Ketone Peroxide
OH-	Hydroxyl
NaOH	Sodium Hydroxide
SEM	Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscope
ASTM	American Society for Testing and Materials
MAPP	Maleic anhydride grafted polypropylene
PP	Polypropylene
PE	Polyethylene
TGA	Thermogravimetric Analysis
DTG	Derivative Thermogravimetric
wt.%	Weight percentage
vol.%	Volume percentage
LCA	Life Cycle Analysis
w/v	Weight/Volume
v/v	Volume/Volume
UTM	Universal Testing Machine
RTM	Resin Transfer Moulding
VARTM	Vacuum-Assisted Resin Transfer Moulding
FRP	Fibre Reinforced Polymer
r-HDPE	Recycled High-density polyethylene

CHAPTER 1: INTRODUCTION

1.1 Overview

This chapter presents the background and the motivation behind initiating this research endeavour. The aim, objectives, and thesis structure of this thesis are also established.

1.2 Background of the Study

In recent years, fibre-reinforced composites have emerged as promising alternatives to conventional metals. They exhibit excellent resistance to corrosion and fatigue, along with high specific tensile and compressive strength. Additionally, they offer controllable electrical conductivity, a low coefficient of thermal expansion, and the versatility to be easily shaped into complex forms (Bhat et al., 2019). Due to their clean ecological effect, natural fibre-reinforced composites have piqued the interest of engineers over synthetic-based ones due to their availability, renewability, sustainability, and biodegradability. This has led to a proliferation of their use and integration into various industries like aviation, automotive, and manufacturing. Many researchers have explored how several natural fibre-reinforced polymer composites perform physically, chemically, and morphologically. Researchers and industries have explored and utilized natural fibres like sugar palm, kenaf, sisal, jute, and flax in fabricating composites (Akil et al., 2011; Asyraf et al., 2022; Saxena et al., 2011; H. Singh et al., 2018; Yan et al., 2014). This study would break new ground for a novel fibre to be explored and utilized.

Among the different natural fibre reinforcing materials, Mokolwane fibre offers a new opportunity. Mokolwane tree (*Hyphaene Petersiana*), shown in Figure 1.1 is plentiful and indigenous to Botswana with research on its usage as a fibre reinforced polymer composite material unexplored. The leaflets from this plant are currently utilized by locals to make various textile products and handmade artifacts like baskets, wallets, and handbags. In various regions across Africa, this plant serves as a vital source of sustenance and livelihood. Its versatile contributions include yielding palm wine extracted from its sap, providing nutrition through its nuts and shoots, and supplying materials for construction derived from leaves and stems (Foote et al.,2003; Tselaesele et al.,2023). Although the plant is heavily exploited, its robust regenerative capabilities ensure its resilience, and it remains abundant in nature. Research on the efficacy of Mokolwane fibre is limited, yet Machaka and Basha (2014) have investigated its ability to strengthen cement for concrete production. In a separate study, Moumakwa et al. (2022) delved into the impact of alkali and thermal treatment on the fibre's crystallinity, thermal stability, tensile strength, and morphological characteristics. However, no prior research within the author's scope has examined the effectiveness of the fibres as a polymer composite reinforcement.

Figure 1.1: Mokolwane tree (captured in Nata, Botswana)

1.2.1 Polymer Matrix Composites

A composite combines two or more materials wherein one would be in a matrix phase while the other in a reinforcement phase forms a material with better chemical and physical properties than its individual constituents (Kumar et al., 2021). Composite materials are further classified into polymer, ceramic, and metal-matrix composites. Polymer Matrix Composites (PMCs) are highly attractive for engineering and structural applications due to their relatively simple processing, which enables users to customize them according to their needs(Raju & Shanmugaraja,2020). PMCs have higher strength and stiffness compared to other composites. They can be modified to achieve a high strength-to-weight ratio and corrosion resistance, making them capable of withstanding severe environmental conditions. However, the usage of synthetic fibre reinforcements has been hindered by their negative ecological effects.

PMCs are made from a continuous phase of polymer matrix and a reinforcement in the dispersed phase (Singh et al., 2020). The interphase of a polymer matrix composite is the region between the matrix and the reinforcement that allows load-transfer between the two. The matrix of a composite is responsible for holding the composite in shape whilst also transferring the load between the reinforced fibres. It shields the reinforcement from damage, such as wear and chemical attack (Loos, 2015; Sajan & Philip Selvaraj, 2021). The discontinuous phase or reinforcement may be in the form of particles, fibres, and flakes. In fibre-reinforced polymer materials this reinforcement is in the form of a fibre e.g., glass, carbon, organic, sisal, etc. Reinforcement of a composite should not chemically react with the matrix. Its role is to add stiffness and strength to the composite. Additionally, factors like reinforcement constituents, fibre type, fibre volume fraction, composition, geometry, and orientation influence the composite's overall properties (Abilash & Sivapragash, 2016).

1.2.2 Applications of Polymer Matrix Composites

Automotive

Because of their low density and impressive mechanical properties, PMCs are widely used in vehicle interiors. Their ability to reduce vehicle weight contributes to maintaining optimal fuel efficiency and reducing emissions without compromising strength requirements. Additionally, PMCs exhibit favourable acoustic properties. The automotive industry has increasingly utilized plastics for fabricating complex components, leading to a decrease in the overall percentage of metals in vehicles. From 6% in 1970, the mass percentage of plastics in vehicles rose to 16% in 2010 and was projected to reach 18% in 2020, as depicted in Figure 1.2 (Bouzouita, 2016). However, cost-effective manufacturing techniques are crucial for the mass production of PMC parts.

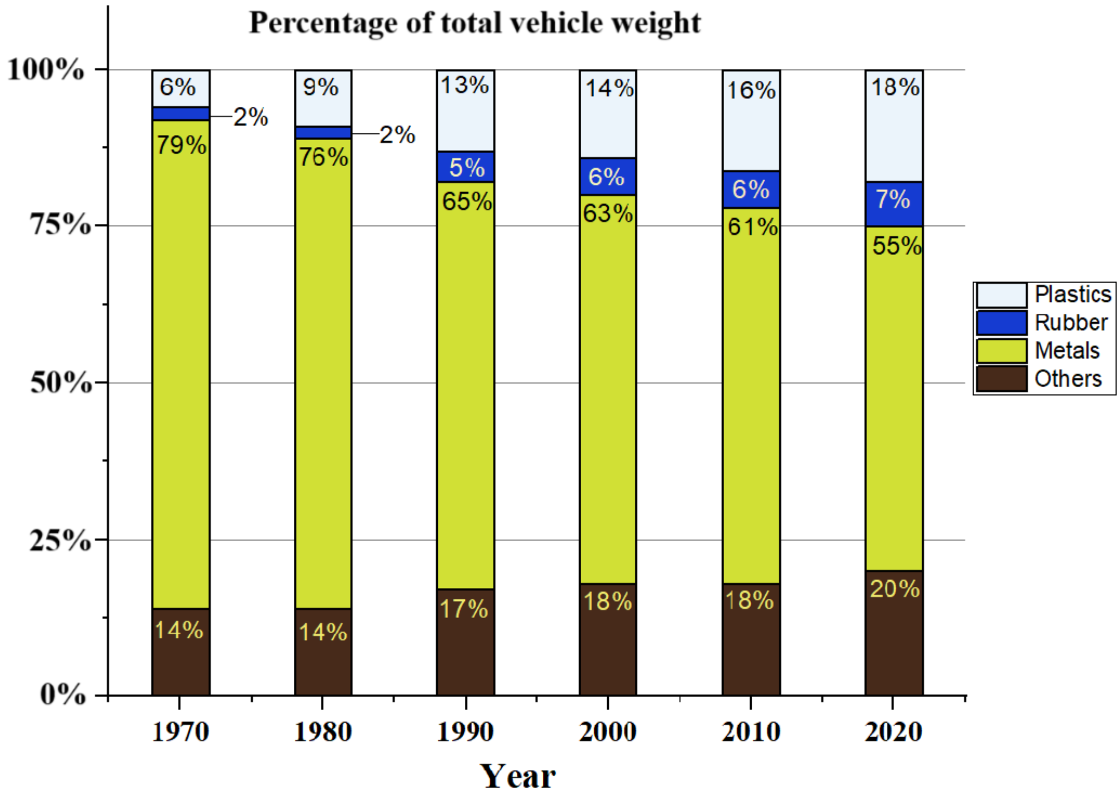

Figure 1.2: Evolution of the use of plastics in vehicles (adapted from Bouzouita, (2016))

Currently, fibre reinforced composites are employed for both structural and non-structural automobile parts such as bumpers and car seats (Todor et al., 2017). Carbon-polymer composites present new design opportunities for engineer in crafting vehicle structural components. For instance, over 95% of the McLaren F1's chassis was constructed using high-performance advanced carbon(graphite) epoxy composite material (Davies, 2012). Although carbon fibre dominates the industry, its high cost has spurred research into alternative materials like natural fibres. Incorporating natural fibres could further reduce vehicle weight due their lower density compared to synthetic counterparts, while also offering cleaner ecological effects. Government and environmental agencies' regulations aimed at reducing carbon emissions have incentivized vehicle manufacturers to explore natural fibres. For example, European regulations since 2015 mandate that end-of-life vehicles be 85% reusable/recyclable, with the remainder designated for energy recovery or landfill (Balla et al., 2019; Paul et al., 2015).

European car manufacturers have embraced thermoset and thermoplastic-based natural fibre composites for various automotive components such as door panels, seat backs, interior parts, and dashboards(Holbery & Houston, 2006). Notably, Audi^{TM} has incorporated hemp-reinforced epoxy composites into the manufacture of side panels, replacing Acrylonitrile Styrene(ABS) material (Chaudhary & Ahmad, 2020). In 2019, Porsche, the sports car manufacturer, introduced a groundbreaking development with the 718 Cayman GT4 Clubsport racer, featuring exterior components crafted from composites reinforced with natural fibres derived from hemp and flax (Porsche, 2019).

Aerospace

The aerospace sector stands as a pioneering realm in engineering, having embraced composites early on due to their exceptional strength and stiffness while maintaining low weight. This attribute not only reduces fuel costs but also becomes paramount for achieving high speeds, longer travel distances, and accommodating increased payload. Consequently, the utilization of composites leads to a noteworthy reduction in carbon emissions from aircraft.

Traditionally, most aircraft bodies have been constructed using aluminium alloys, owing to their commendable mechanical properties, lightweight nature, and corrosion resistance. However, aircraft manufacturers are actively pursuing ways to mitigate direct operating costs by integrating advanced composite materials to decrease weight and subsequently lower fuel consumption (Kaufmann et al., 2010). Furthermore, the fabrication of airplane components with fibre-reinforced composites enhances their fatigue performance, making them ideal for crafting mechanical structures like wings, fuselages, and undercarriages (Chen et al., 2023).

Fibre-reinforced polymer composites are widely used in various aircraft models, including the Boeing 757, 767, and 777, as well as the Airbus A310, A320, A330, and A340 airliners (Quilter, n.d.). Carbon fibre has been instrumental in reducing weight when used as a substitute for aluminium alloy materials. For instance, Bell helicopters achieved a weight reduction of 20% by transitioning from metallic airframes to fibre-reinforced composites (Asim et al., 2018).

The unique demands of aircraft design, such as the V22 Osprey tilt rotor, necessitate materials with low density to ensure optimal stiffness and critical design parameters (Bell & Osprey, n.d.). The Boeing 787 stands out for its extensive use of composite materials, constituting up to 50% of its primary structure, surpassing any other commercial aircraft, as shown in Figure 1.3 (Singh & Kumar, 2022). This results in an average weight reduction of 20% and enhanced impact resistance compared to traditional metal-aluminium design aircraft.

The benefits of composite materials in aviation are manifold, including high strength-to-weight ratio, excellent fatigue resistance, improved corrosion resistance, and subsequently lower maintenance costs (Mansor, Nurfaizey, et al., 2019). The ability to tailor carbon fibre-reinforced polymer composites to desired shapes and properties further enables the construction of aircraft with efficient aerodynamic configurations.

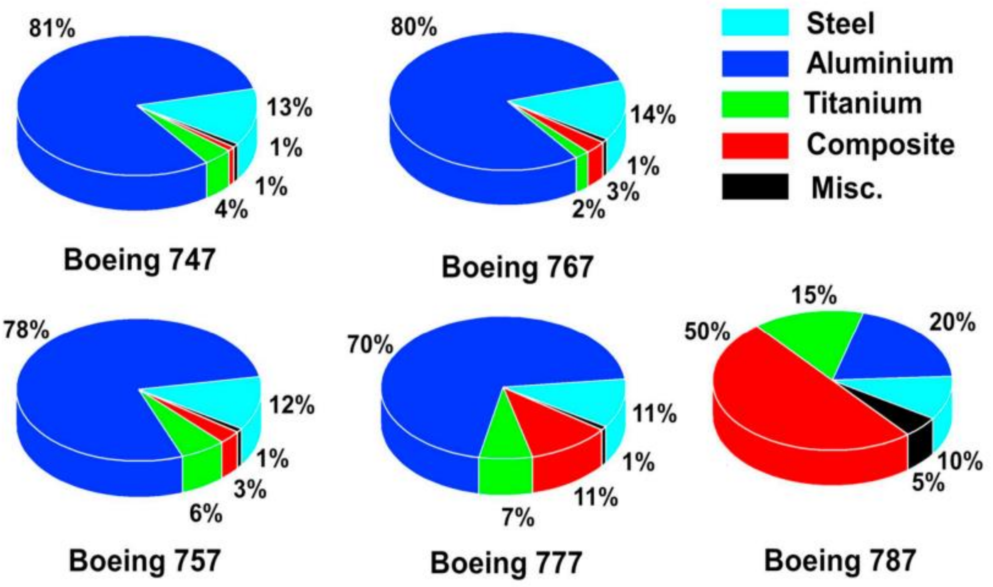

Figure 1.3: Increase in the use of composite materials in Aircrafts (Singh & Kumar, 2022)

Marine

The adoption of fibre-reinforced composites as substitutes for traditional materials in this sector has been propelled primarily by weight reduction. This has resulted in higher cargo capacity, improved fuel efficiency, reduced inertia, better acceleration, as well as heightened ship stability and buoyancy. Utilizing fibre-reinforced composites enhances corrosion resistance, thereby decreasing maintenance requirements (Rubino et al., 2020). Furthermore, the attractiveness of fibre-reinforced composites stems from their inherent flexibility. The ability to customize their mechanical properties by adjusting the type, quantity, and orientation of reinforcing materials positions them as optimal candidates for high-pressure conditions (Shamsuddoha et al., 2013).

Naval engineers have integrated glass and carbon fibre polymer composites into propeller blades to aid in acoustic damping and cost reduction. Fibre-reinforced polymer composites, incorporating carbon, aramid, or glass fibres, emerge as superior alternatives to steel for skin materials, boasting notable corrosion resistance, reduced maintenance demands, and enhances design flexibility. The utilization of glass fibre-reinforced polymer composites in tidal turbine blades strikes a balance between structural integrity and affordability, while carbon fibre-reinforced polymer composites offer high performance at reduced weight (Grogan et al., 2013; Jo et al., 2013).

Carbon fibre composites have found practical application in superstructures of larger vessels, such as the Russian Navy's Admiral Gorshkov class stealth frigate, and the United Staes Navy's *Zumwalt* class DDG 1000 destroyer (illustrated in Figure 1.4) where they are employed in the upper-section deckhouse, helicopter hangar, and ballistic screen (LeGault, 2010). Swedish Visby class corvettes incorporate vinyl ester/carbon sandwich hulls with a polyvinyl chloride core to enhance resilience (Lowde et al., 2022). Nonetheless, the production of composite sandwich structures proves to be more costly and labour-intensive compared to conventional materials. Additionally, the manufacturing process can lead to the presence of voids in fibre-reinforced composites, rendering the parts susceptible to moisture absorption.

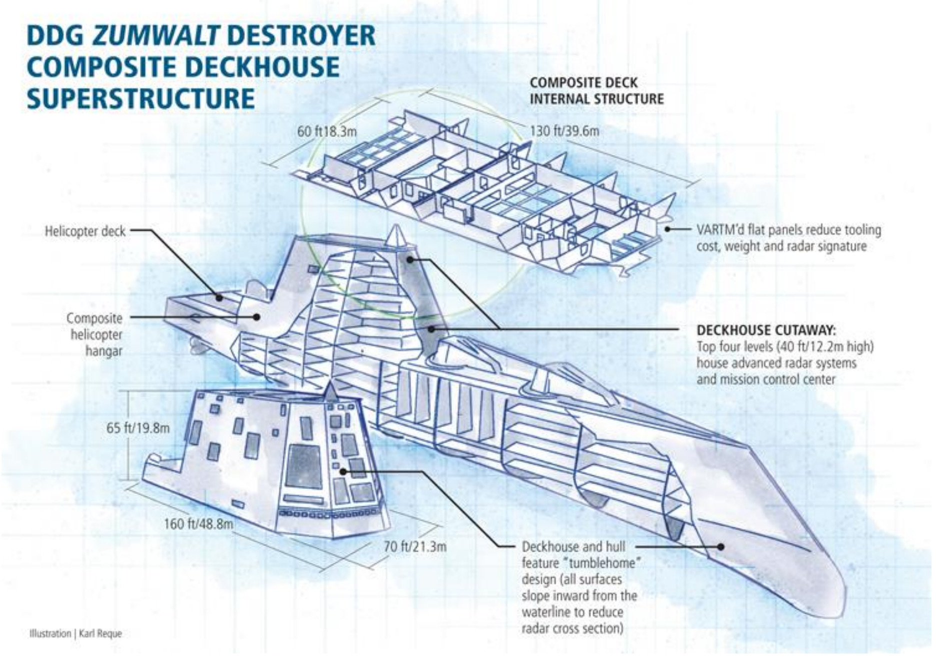

Figure 1.4: DDG *Zumwalt* Destroyer 1000 (LeGault, 2010)

Natural fibre composites have demonstrated their utility in marine applications, exemplified by the TariTari sailboat crafted from a combination of Jute and glass composites, which successfully voyaged 14,000 miles from Bangladesh to France (El Hawary et al., 2023). Advancing the integration of natural fibres into high-performance marine vessels could yield more economical, lighter, and environmentally sustainable substitutes for synthetic fibre reinforcements in various non-structural components.

1.3 Problem Statement

The utilization of natural fibres in composite materials presents numerous advantages over their synthetic counterparts, such as lightweight, affordability, renewability, flexibility, eco-friendliness, and biodegradability. Despite their individual applications across various sectors, natural fibres like kenaf, hemp, jute, coir, banana, and cotton exhibit drawbacks when employed as reinforcing materials in polymer composites. These caveats encompass high moisture absorption, reduced durability, inadequate fire resistance, inferior mechanical properties, quality variability, limited thermal resistance, and susceptibility to microbial degradation. The primary cause of these shortcomings lies in the inherent high polarity of natural fibres, attributed to their substantial polysaccharide content, notably hemicelluloses and celluloses, resulting in heightened hydrophilicity (Abdullah & Ahmad, 2013). To address these challenges, various fibre treatment techniques, including alkali treatment, silane treatment, and acetylation, are employed to mitigate water absorption and enhance interfacial bonding.

While extensive research exists on various natural fibres, the potential of Mokolwane fibre remains largely untapped. Indigenous to Botswana, this fibre offers a sustainable solution due to its excellent regenerative properties when extracted from the leaflets. A composite can be synthesized, experimentally evaluated for its properties, and assessed for suitability in industrial applications. Recognizing the importance of exploring this locally available fibre for industrial applications, it is compelling to investigate its potential. Whilst undertaking a study on this fibre, some expected problems would be finding the most optimum fibre loading and alkali treatment condition to yield a composite with the best performance. Important factors to assess include, but are not limited to, moisture absorption, tensile strength, flexural strength, and impact strength of the composite. Additionally, there is scarcity of literature on characterization of this indigenous fibre. This research involves characterizing the fibre's properties, fabricating a composite, and subsequently conducting comprehensive tests. Additionally, a comparative analysis is conducted to assess the impact of alkali fibre treatment, and fibre volume fraction on the properties of the composite.

1.4 General and Specific Objectives of the Study

General objective: To fabricate and experimentally evaluate mokolwane fibre reinforced polyester composite for industrial applications.

This objective was achieved by the following specific objectives.

i. To characterize the properties of Mokolwane fibre through fibre diameter distribution, moisture absorption rates, thermogravimetric properties, and fibre morphology.
ii. To investigate the influence alkali treatment and fibre composition on the water absorption properties of the Mokolwane fibre-reinforced polyester composite.
iii. To examine the effects of alkali treatment and fibre composition on the mechanical morphological properties of Mokolwane fibre-reinforced polyester composite.

1.5 Scope and Limitations of the Study

This study focused on conducting specific characterization tests aimed at gaining insight into the properties of the novel fibre under investigation. These tests include analysis of fibre diameter distribution, moisture absorption rates, thermogravimetric properties, and fibre morphology. Additionally, the study explores the application of this fibre as a reinforcement to a polyester composite. Supplementary tests were conducted to evaluate the performance of the resulting composites and to investigate the impact of pre-treating the fibres prior to composite fabrication. These tests encompass tensile, flexural, and impact strength evaluations, as well as assessments of density, water absorption, and thickness swelling. Furthermore, scanning electron microscopy is employed to examine the morphology of the composites in detail.

The study is limited to fibres extracted from leaflets of Mokolwane tree and their viability as reinforcement materials. The fibres from other parts of the tree are outside the scope of this work.

1.6 Significance of the Study

In 2015, representatives from 193 nations convened to address the formidable challenges ahead. This historic gathering led to the creation of the United Nations' Sustainable Development Goals (SDGs), a blueprint for global progress. Among these goals, SDG 12 advocates for "*Responsible Consumption and Production*," SDG 13 focuses on "*Climate Action,*" and SDG 14 aims to safeguard "*Life Below Water*" (Shulla & Filho, 2023.) The research conducted for this thesis endeavours to advance eco-friendliness through the utilization of natural fibres in composite materials, thereby contributing to the realization of SDG 12. As discussed in preceding sections, the integration of natural fibre-reinforced composites in the automotive industry has facilitated the production of lighter vehicles, resulting in reduced fuel consumption and emissions, thereby aligning with SDG 13. Contrastingly, the use of synthetic fibres has been linked to the proliferation of microplastic pollution in the oceans. By leveraging natural fibre-reinforced composites, it becomes feasible to develop materials that rival their synthetic counterparts in terms of mechanical properties, all while adhering to environmentally sustainable practices. Consequently, this approach not only satisfies SDG 14 by safeguarding marine ecosystems but also contributes to a holistic advancement towards a greener, more sustainable future.

Developing countries are rich in natural resources, yet they still rely on developed countries to lead industrial innovations. This investigation seeks to emphasize the importance of studying and integrating indigenous, eco-friendly materials that are abundant in these countries into engineering industries. The underlying theme of this work emphasizes the value of natural, indigenous resources. The outcomes of this research will enable future researchers to design improved composite processing methods and assess how alkali treatment influences composite properties.

1.7 Thesis Outline

This dissertation aims to explore and assess the potential of Mokolwane fibre as a reinforcement in polyester composites for industrial applications. The introductory chapter provides an overview of fibre-reinforced polymers, their contemporary industrial uses, and associated challenges. Specific objectives, scope, limitations, and the significance of the study are also outlined in this chapter.

In the second chapter, natural fibres are examined in detail, including their classification, composition, and compatibility with polymer matrices. Natural fibre reinforced polymer composites manufacturing techniques are evaluated. A review of relevant literature evaluates the impact of factors such as fibre loading and surface treatment on the performance natural fibre reinforced polymer composites. This chapter specifically reviews how surface treatment techniques have been used to alter mechanical, moisture absorption, chemical, morphological, and thermal properties, of natural fibre-based composites. Furthermore, the chapter assesses the sustainable development with natural fibre composites in industrial settings.

The third chapter outlines the methodological framework employed in this research. It details the processes involved in fibre extraction, processing, and experimental evaluation of Mokolwane fibres. A description of fabrication process of Mokolwane fibre reinforced polyester composite is outlined. The subsequent experiments carried out on the fibre reinforced composite samples to achieve thesis aim (iii) are outlined in this chapter. Additionally, the chapter provides insights into the equipment utilized and justifies the selection of specific tests conducted.

The fourth chapter delineates the outcomes from the experiments carried out in this study. The findings of the fibre characterization and composite test are presented graphically, and the results interpreted by comparing them to existing literature.

The discussions & conclusions of this study are presented in the penultimate chapter of this dissertation. This entails a summarized presentation of research outcomes, suggestions on the industrial applications of the composite, and the manufacturing considerations when incorporating the composite in industrial applications. The chapter also outlines the contributions of this research and the gaps that future works could explore when investigating this fibre reinforced composite.

The last chapter conveys the research sources cited during the write up of this document.

1.8 Chapter Summary

This chapter gives a background to the study of natural fibre reinforced composites and why I have taken such an interest in this field. The advantages of using composites over conventional materials are discussed. Natural fibres have been adopted as an eco-friendly alternative to their synthetic counterparts, but their shortcomings limit their applications. Polymer matrix composites are introduced as well as their current applications in different engineering sectors. The chapter presents the statement of problem of this thesis work, the aim and objectives of the study, the research questions and lastly the justification for this work.

CHAPTER 2: LITERATURE REVIEW

2.1 Introduction & Overview

Fundamentally, a composite is a material composed of two distinct materials. The birth of fibre composites in the 1930s came about when scientists discovered plastics, although natural resins were already in use as adhesives. The 20th century saw the discovery of synthetic plastics like vinyl, polyester, and polystyrene. The inherent low strength in plastics inhibited their ability to meet the load-transferring requirements in automobile, aircraft, and sport equipment parts, therefore, reinforcements were introduced to address this. The need for lightweight materials during the second world war propelled the evolution of fibre-reinforced composites (Vigneshwaran et al., 2020). After the world war, the further discovery of resins and synthetic fibres transformed how conventional materials were used. The mechanical performance of synthetic fibres like glass and carbon in polymer composites impelled their integration into various applications (Gay et al., 2002). Despite their pros, the nonbiodegradability of synthetic fibres means they have a negative ecological effect. The substitution of synthetic fibres with natural fibres in composites has been on the rise due to environmental reasons.

Natural fibre are substituting synthetic fibre in fibre reinforced composites because they are light-weight, non-abrasive, possess less health hazards, are eco-friendly, are process friendly, have high impact resistance, and are cheaper (Kerni et al., 2020). The automotive industry has extensively incorporated the usage of natural fibre-based composites in their applications. They are primarily used in the manufacture of interior components of cars such as door panels, dashboards, and seat backs. Their inherent light weight means that vehicles would consume less fuel and emit less harmful gases. Sporting goods (like tennis rackets and bicycles) and electronics (like laptop cases) are other areas where the natural fibre reinforced composites have found use (Jaafar et al., 2019).

This chapter evaluates the available literature on natural fibre reinforced composites. It looks at natural fibres, their classification and microstructure, composite manufacturing techniques, the influence of surface treatment on composite properties as well as sustainable development of natural fibre composites.

2.2 Natural Fibre Reinforced Composites

2.2.1 Natural Fibres

Fibres are continuous or discrete hair-like materials that are like pieces of thread. These materials have been an important part of composites for years. Fibres are classified as either natural or synthetic. Natural fibres are further classified according to their origin and grouped into plant, animal, and mineral fibres. One significant contrast between animal and plant fibres lies in their primary composition: animal fibres primarily comprise protein, whereas plant fibres are predominantly composed of cellulose. Animal fibres include sheep's wool, goat hair, horsehair, and cashmere (Lotfi et al., 2021). Mineral fibres such as asbestos are well-known for their outstanding thermal stability and impressive tensile strength (Nayak, 2016). Of the three, plant fibres, or lignocellulosic fibres, have caught the attention of engineers and researchers. They are classified into five groups illustrated in Figure 2.1. Seed fibres are extracted from the plant seeds. Examples of these are cotton and kapok. Leaf fibres like sisal are extracted from the leaves. Flax, hemp, and ramie are examples of bast fibres. These are fibres are extracted from the skin around the plant stems (Markova, 2019). Fruit fibres like coir are collected from plant fruits. The fifth class of plant fibres are stalk fibres extracted from the stalks of the plant. An example of this is bamboo fibre.

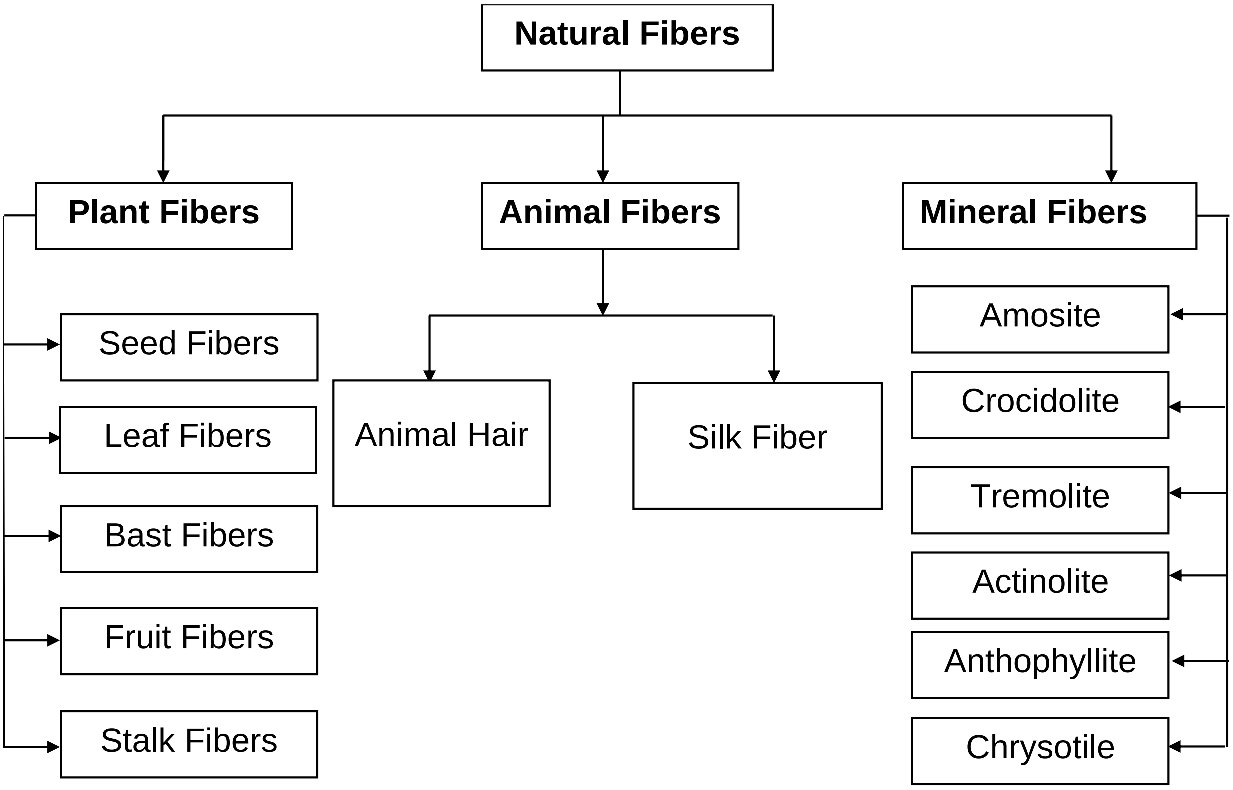

Figure 2.1: Classification of Natural Fibres (Adapted from Lotfi et al., (2021))

Contemporarily, natural fibres have been found to be a worthy substitute to their synthetic counterparts in reinforcing polymer- based composites because they are lighter, cheaper, and biodegradable. Natural fibres like Jute, Sisal and Kenaf are produced world-wide to primarily produce composites (Anandjiwala & John, 2010). Besides reinforcing polymers, natural fibres are used to reinforce cement-based matrices to enhance composite ductility, post-cracking toughness and to improve impact and fatigue properties. By curbing the propagation of cracks and enhancing durability of reinforced concrete, fibres have found use in construction engineering (Castoldi et al., 2022). Table 2.1 shows tensile properties of some of the common natural fibres.

Table 2.1: Density and tensile properties of natural Fibres

Fibre	Density	Tensile Strength (MPa)	Young's Modulus (GPa)	Elongation (%)	Refs
Kenaf	1.2	101	23	17.3	Sharba et al., (2015)
Coir	1.2	286	2.74	20.8	Yan et al., (2015)
Jute	1.3-1.46	393-800	10-30	1.5-1.8	Rajak et al., (2019)
Flax	1.4-1.5	345-1500	27.6-80	1.2-3.2	Rajak et al., (2019)
Ramie	1.20~1.49	30~40	-	1.6~2.0	Tamta and (Kalita, 2020)
Sisal	1.33-1.5	400-700	9-38	2-14	Rajak et al., (2019)
Caraua	-	700 – 1100	26 - 46	4.2	Pandey et al., (2010)
Cotton	1.5-1.6	287-800	5.5-13	3.0-10	Abdollahiparsa et al., (2023)
Hemp	1.5	550-1110	27-80	1.6	Abdollahiparsa et al., (2023)
Synthetic Fibres					
Glass	2.5	2400	50	3.0	Sharba et al., (2015)
Carbon	1.8	3500-5000	260	2.5	Pandita et al., (2014)
Aramid	1.45	2700-4500	130	3.3-3.7	Pandita et al., (2014)

2.2.2 Natural Fibre Composition

The cell wall morphology of plant fibres is comprised of several, concentric layers of cellulosic microfibrils with varying thicknesses as shown by the Figure 2.2. Lignocellulosic fibres have an appearance of microscopic tubes, that is, cell walls that surround the central lumen. Water uptake of plants occur through the lumen. The fibre is made up of several cell walls, that are composed of oriented microfibrils embedded in hemicellulose-lignin matrix (Thomas et al., 2011). Cell walls are composed of two sections, the primary cell wall, that has closely packed cellulose microfibrils, and the secondary wall, comprising of three separate layers. Natural fibres mainly constitute of cellulose, hemicellulose, lignin, wax, pectin, and water-soluble compounds. Cellulose is the major chemical constituent of the lignocellulose fibre that provides tensile strength, stability and stiffness to the cell walls and the fibre(Komuraiah et al., 2014).

Cellulose is comprised of D-glucopyranose units joined together by β-(1-4)-glucosidic bonds. The abundant hydroxyl groups found in cellulose contribute to the poor dimensional stability of plant fibres. However, cellulose demonstrates a positive correlation with strength, elasticity, and overall physical properties of fibres (Amiandamhen et al., 2020). Hemicellulose polymers are amorphous, highly hydrophilic components of the plant cells that enclose cellulose fibrils. They consist of polysaccharides and helps with providing necessary stiffness to the fibres.

Lignin is a highly complex, aromatic hydrocarbon polymer that acts as an adhesive between fibre cells and the fibrils that make up the cell wall, and in so doing adds rigidity to the plants. Lignin is a fully amorphous and hydrophobic constituent of plant cells made up of phenyl propane units. Waxes and pectin are the other constituents of a lignocellulosic cell, the latter of which imparts flexibility to plants (Manral & Bajpai, 2018).

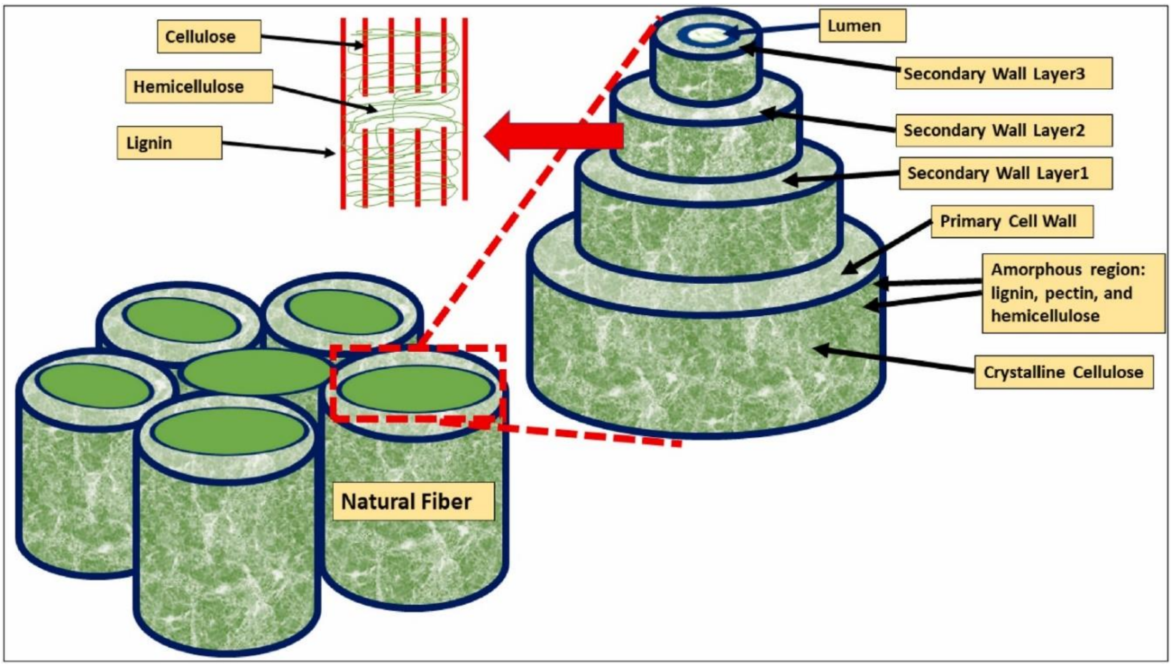

Figure 2.2: Natural fibre composition (Mohammed et al., 2023)

2.2.3 Matrix

The matrix of a fibre reinforced composite is a resin system that binds the fibre reinforcements together to allow for stress transfers. The matrix protects the fibre reinforcements from mechanical and environmental abrasion and gives the composite its solid form.

Polymer matrices that are used in natural composites are classified into thermoplastics and thermosetting. A thermoplastic is a malleable material above a certain temperature that solidifies when cooled (Jaafar et al., 2019). Thermoplastics in matrix applications that are frequently employed are Polypropylene (PE), Polyethylene (PP), Polystyrene (PS), Polytetrafluoroethylene (PTFE), Polylactic acid (PLA), and Polyvinyl chloride (PVC). Thermoplastic composites are known for their lightweight and recyclability. However, they possess high melt viscosity. This is rectified by manufacturing thermoplastic materials in fibre forms (Al-darkazali et al., 2018). Thermoset, on the other hand, is a type of plastic that has undergone an irreversible cooling process from a soft solid or viscous liquid prepolymer or resin. Curing transforms the resin into an infusible and insoluble polymer network through the application of heat or radiation, often under high pressure, or by combining it with a catalyst. The most frequently used thermosets are epoxy, polyester, and phenolic. Of the two polymer types, thermoset polymers are more commonly used due to their higher structural strength than the thermoplastics.

Polyester Resin

The appeal of polyester resins, most specifically the "unsaturated" types, is their inherent ability to cure from liquid to solid under various conditions. The most employed catalyst for the polyester resin is the Methyl Ethyl Ketone Peroxide (MEKP) at about 1-2%. Presence of the polyester resin breaks down the catalyst generating free radicals. The free radicals would then initiate polymerization process by reacting with unsaturated groups such as carbon-carbon double bonds (C=C) (Bagherpour, 2012). The competitive advantage of polyester resin over other thermosets is their low cost, inherent low viscosity, and shorter curing time. However, polyesters generally have lower weathering resistance and mechanical properties than other resins like epoxy (Maxineasa & Taranu, 2018). Polyester resins are still popular because they give the best value economically, and for their structural value. Some important utilitarian products that are manufactured with the use of unsaturated polyester resin are pearl buttons, knife and umbrella handles, bathroom fixtures as well as to enclose electronics assemblies.

The use of polyester in fibre reinforced composites has found important practical applications in construction, automotive, and biomedical applications (Zaghloul et al., 2021). Research has shown that composites with polyester matrix showed good mechanical strength, rheology, heat stability and chemical stability, making them good candidates for various industrial applications (Gorrasi et al., 2018). Mechanical properties of some of the natural fibre reinforced polyester composites from various researchers are highlighted in Table 2.2.

Table 2.2: Mechanical properties of natural fibre reinforced polyester composites.

Material	Fibre loading	Ultimate Tensile Strength (MPa)	Tensile Modulus (GPa)	Ultimate flexural Strength (MPa)	Flexural Modulus (GPa)	Reference
Polyester + Acro shell	9wt.%	38	-	-	-	Abass et al., n.d.
Polyester + Sansevieria Triafasciata Fibres	30wt.%	32.53	2.15	61.42	2.42	Premkumar et al., (2022)
Polyester + NaOH treated	30wt.%	48.47	2.72	69.17	3.33	Premkumar et al.,

Sansevieria Triafasciata Fibres						(2022)
Polyester + Hemp Fibre	20wt.%	28.87	0.38	60.06	1.86	Neves et al., (2019)
Polyester + Hemp Fibre	30wt.%	31.46	0.51	49.09	1.22	Neves et al., (2019)
Polyester + Bagasse	5wt.%	-	3.44	23.1	-	Naguib et al., (2015)
Polyester + NaOH treated Bagasse	5wt.%	-	4.42	26.4	-	Naguib et al., (2015)
Polyester + Grewia Serrulata	15wt.%	32.98	2.87	57.62	2.25	Mahesha et al., (2018)
Polyester + acetylated Grewia Serrulata	15wt.%	49.18	3.11	50.18	2.55	Mahesha et al., (2018)
Polyester + NaOH treated Jute Fibre	18 wt.%	9.23	0.81	44.71	1.91	Gopinath et al., (2014)
Polyester + Mallow fabric	40wt.%	110	11	-	-	De Moraes et al., (2018)

2.3 Manufacturing Techniques of Fibre Reinforced Composites

Different techniques are employed in the fabrication of natural fibre polymer composites. These include hand lay-up, compression moulding, injection moulding, Resin Transfer Moulding (RTM), and Vacuum Assisted Resin Transfer Moulding (VARTM). Choosing an appropriate manufacturing method is crucial for shaping the structure and ensuring the desired composite properties without minimal flaws. The initial evaluation to determine the most suitable manufacturing process entails assessing various key criteria, such as the shape, dimensions, and desired characteristics of the composites, alongside factors like production expenses, speed, and the qualities of the raw materials. The pros and cons of these techniques are outlined in Table 2.3 shown.

Table 2.3: Pros and Cons of different manufacturing techniques.

Method	Advantages	Disadvantages
Hand lay-up	• Simplicity • Low tooling costs • Complex parts can be manufactured	• Skilled labour is required for process accuracy. • Results in high void content • Higher production time • Lower mechanical properties
Compression Moulding	• Excellent part reproducibility • Low labour costs • High production rates	• Process can distort complex features. • The process produces waste
Injection Moulding	• High consistency and dependability • Compatible with various materials and colours	• Requires bespoke moulds. • Not recommended to create durable parts. • Costly to incorporate design changes
Resin Transfer Moulding (RTM)	• Design flexibility. • Low start-up cost. • Good surface finish • Faster production • Lower temperature requirements	• Expensive tooling to create complex moulds. • Reinforcement materials are constrained by the flow and resin saturation of the fibres.
Vacuum Assisted Resin Transfer Moulding	• Ability to manufacture complex and large parts of good quality. • Low tooling costs	• Requires a complex set-up. • The cosmetic finish on the surface doesn't match the quality achieved through the

(VARTM)		open mould process

2.3.1 Hand Layup

Hand layup is a commonly employed manufacturing technique in which the fiber is manually impregnated with resin, followed by consolidation using a roller (Elseify et al., 2020). The schematic for hand layup is illustrated in Figure 2.3 below. Typically, this procedure comprises of four stages: mould preparation, application of gel coating, lay-up, and curing. Curing involves solidifying the fibre-reinforced resin composite without external heat. Initially, a pigmented gel coat is administered to the mould surface to achieve a superior product finish (Jamir et al., 2018). The method produces highly complex parts, adaptable to design changes and is economical. However, this manual process means that production rate is low or leads to high cost of labour (Elkington et al., 2015). The fabrication of marine and aerospace structure mainly employs this technique(Jamir et al., 2018).

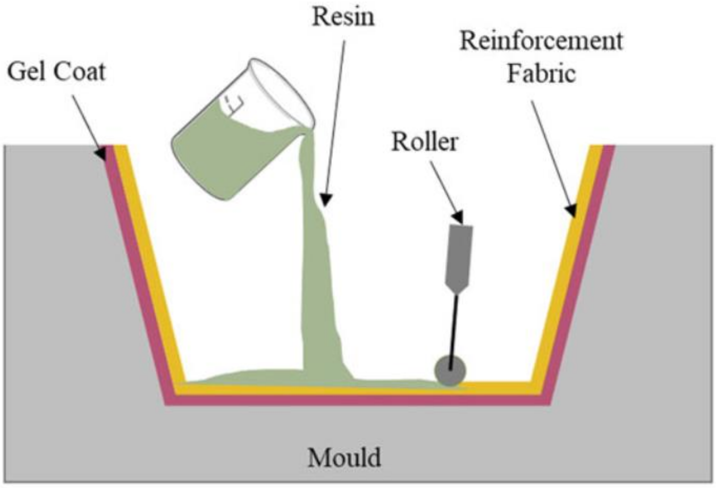

Figure 2.3: Hand layup schematic(Elseify et al., 2020)

2.3.2 Compression Moulding

This is a composite fabrication method that a composite is moulded under heat and high pressures to form complex shaped parts (Howell et al., n.d.). Figure 2.4 shows the schematic of fabricating a component using this method. It leads to low fibre attrition and is quick to undertake. Studies have reported that the method produces composites with high strengths and impact resistance (Aji et al., 2009.; Nyior and Mgbeahuru, 2018). This method is beneficial due to its ability to fabricate complex shapes that are difficult to undertake with traditional metallic fabrication processes.

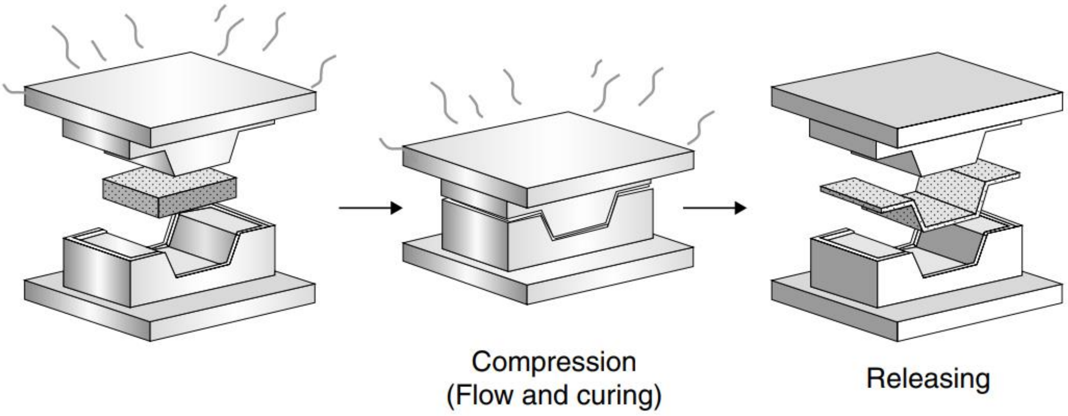

Figure 2.4: Compression moulding process (Park & Lee, 2012)

This technique is employed in fabrication of semi-structural automotive parts such as bumper beams tailgates and exterior body panels, and in home appliances like refrigerator doors and tabletops (Park & Lee, 2012). Compression moulding is extensively found in literature, mostly for fabricating thermoset composites, possibly due to their easier processing compared to thermoplastics.

2.3.3 Injection Moulding

In the injection moulding process, pellets of fibre composites are introduced through a hopper, and subsequently transported by a heated barrel via a screw mechanism, as illustrated in the schematic diagram depicted in Figure 2.5**.** After the required material amount has been melted, it passes through the nozzle into the mould. The polymer undergoes solidification, and the mould is securely fastened. Once the composite has completed curing, the mould is then detached (Balasubramanian et al., 2018). This method is renowned for its ability to mould many identical composites parts of high precision at very low cycle times (Rajak et al., 2019). Injection moulding is used to manufacture lightweight materials in aerospace, electronics, sports and construction (Rajak et al., 2021).

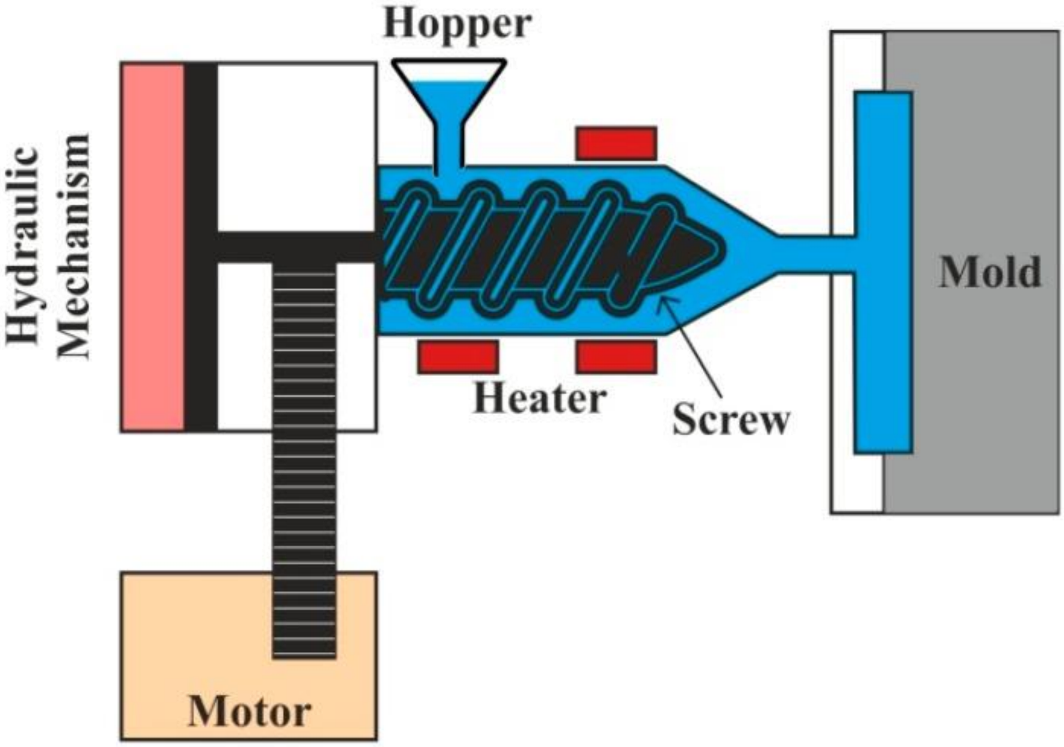

Figure 2.5: Injection moulding schematic(Rajak et al., 2019)

2.3.4 Resin Transfer Moulding (RTM)

This manufacturing technique is whereby a resin is injected into a sealed cavity mould that contains fibre reinforcement (called a ‘preform’). In RTM, a dry fibre reinforcement preform is positioned within a mould's cavity. The mould is then sealed, resin is injected under pressure into the preform, and subsequently cured to produce a composite part. The schematic of this technique is outlined in Figure 2.6 shown. This technique is used in the fabrication of bath and shower enclosures, aircraft parts, and submarine sonar domes (Bhatt et al., 2018). RTM method was found to exhibit higher flexural strength values than the traditional hand layup method by Davallo and Pasdar, (2009) in their study on glass-polyester composites. RTM composites are found to have lower void content resulting in improved mechanical properties over hand layup composites.

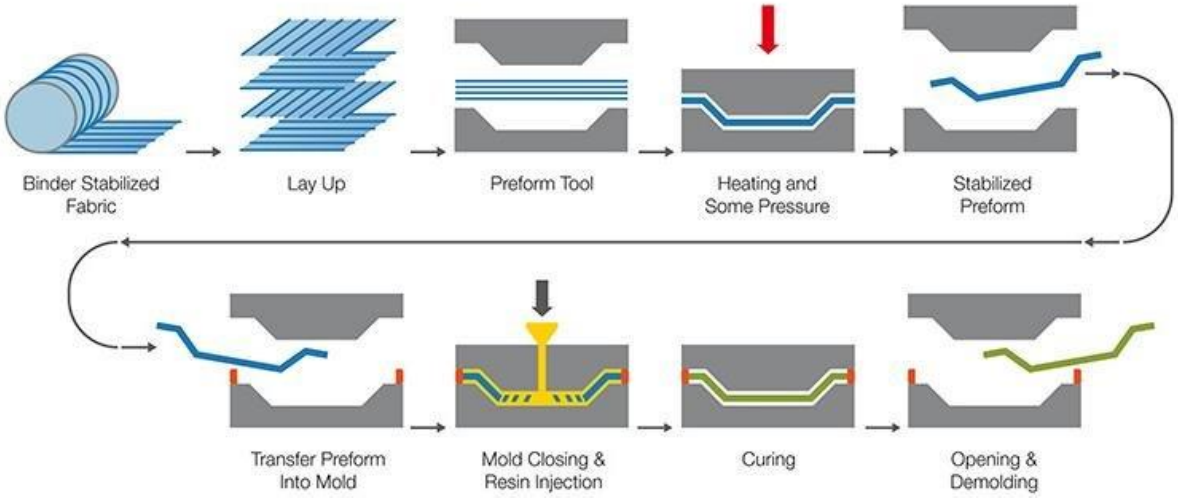

Figure 2.6: Resin Transfer Moulding Schematic(Ahmadova, 2018)

2.3.5 Vacuum-Assisted Resin Transfer Moulding (VARTM)

VARTM, a widely used technique, builds on the RTM method by utilizing the pressure differential between vacuum pressure and environmental pressure to press the preform against the mould and to infuse resin into it (Hsiao & Heider, 2012). Due to the high quality, repeatability, affordability and part complexity of VARTM-manufactured components, this technique has found use in marine, infrastructure building and aerospace industries.

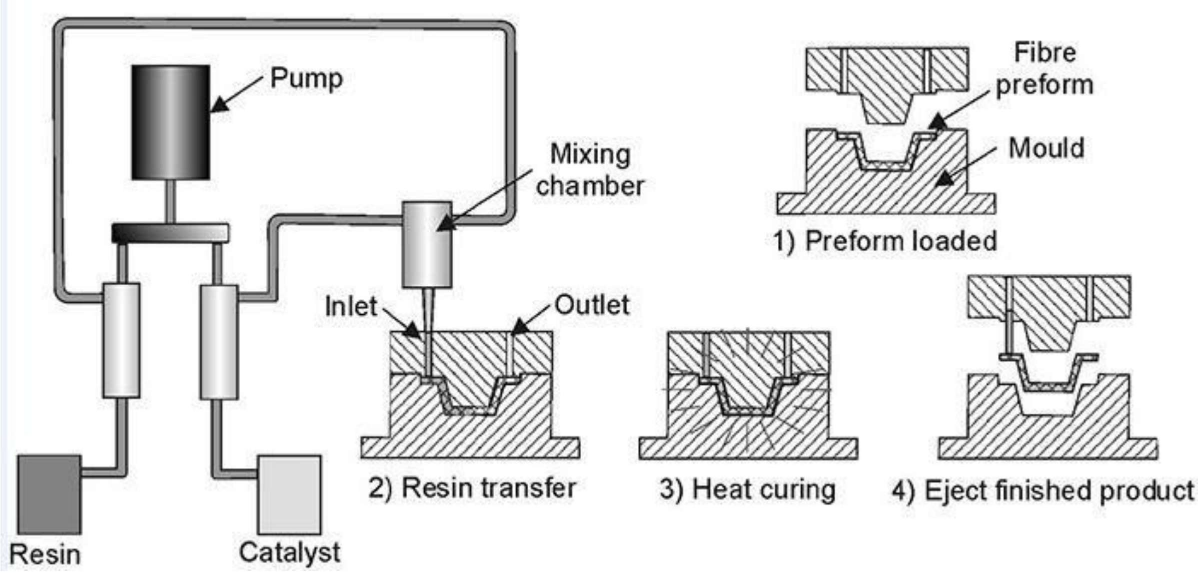

Figure 2.7: Vacuum-Assisted Resin Transfer Moulding schematic (Ahmadova, 2018)

Mohamed et al. (2020) reported that prestressing VARTM- fabricated glass fibre epoxy composites using a hydraulic tensile machine led to an increase in tensile, compressive, and flexural strength over non-prestressed samples. The authors posited that the method reduces fibre waviness in composites, which usually results in structural defects.

Despite the promising performance of VARTM, manufacture large and complex composite structures, void formation remains an issue. Elevated-temperature vacuum-assisted resin transfer moulding process was developed by Menta et al. (2013) to fabricate high performance aerospace composites by modified single vacuum bagging and double vacuum bagging infusion processes. The researchers were able to obtain composites with void content less than 1% and fibre volume fraction of around 60 wt.%. Infusion pressure control to reduce void formation originating from trapped air and resin evaporation was found to be effective by Chen et al. (2015) .

2.4 Influence of Fibre Loading on Composite Properties

Determining the optimal fibre loading in natural fibre reinforced polymer composites is a pivotal aspect of research. Gheith et al. (2019) investigated the ideal fibre loading for date palm fibre reinforced epoxy composites. The study revealed a 50 wt.% fibre loading significantly enhanced the flexural strength and modulus of neat epoxy by 24.8% and 45.1% respectively, outperforming both 40 wt.% and 60 wt.% loadings. They attributed the diminishing properties at higher loadings to inadequate polymer matrix coverage, hindering effective fibre wetting. Similarly, Nyior et al. (2018) noted weakened interfacial adhesion, fibre agglomeration and increased voids at higher fibre loadings, resulting in inferior mechanical properties.

El-Shekeil et al. (2014) observed an 11.5% and 38.5% increase in flexural strength with fibre loadings of 30 wt.% and 40 wt.%, respectively, in cocoa pod husk fibres reinforced thermoplastic polyurethane composites, from an initial 20 wt.% fibre loading. They ascribed this enhancement to robust interfacial bonding capable of withstanding bending stresses, corroborated by SEM images demonstrating excellent fibre matrix adherence without pullouts or gaps.

Incorporating 10 wt.% Bagasse fibre by Subramonian et al. (2016) improved the flexural strength of polypropylene, albeit at the expense of ductility due to poor homogenous mixing leading to fibre fractures and pore formation, impairing stress transfer efficiency. Das et al. (2018) determined the optimal jute fibre loading in propylene to be 50 wt.%, yielding maximum tensile strength, bending stress, and impact strength. However, further fibre addition resulted in diminishing properties, attributed to inadequate homogeneity and fibre orientation. Additionally, the authors reported that lower fibre content in composites contributes to low load transfer as result of highly localized stress points in the polypropylene matrix.

Ibrahim et al. (2014) observed an increase in voids with higher fibre loading in date palm composites, necessitating additional matrix to cover all fibres. Hybridizing fibres with flax also increased void fraction, albeit enhancing flexural properties. These findings align with Asim et al. (2018), who demonstrated superior mechanical properties in pineapple leaf and kenaf phenolic composites at 50 wt.% fibre loading to 40 wt.% and 60 wt.% loadings, emphasizing the importance of optimal fibre content in composite materials.

De Moraes et al. (2018) investigated the mechanical behaviour of mallow fabric reinforced polyester composites. The increase in fibre volume fraction from 0vol.% all the way to 40vol.% gave an exponential increase in the tensile strength, a linear elastic modulus increase, as well as power law increase in resilience of the composites. However, cracks were observed to propagate at 30vol.% throughout the composite, resulting in fibre-matrix decohesion and inhibiting the tensile properties. A positive correlation of Tasar silk fibre loading in epoxy with tensile, flexural and impact strength was established by Ranakoti et al. (2022). Their study was able to show that optimizing fibre loading and fibre treatment enhanced mechanical properties, deducing that higher fibre fraction imparted high stiffness and restricted epoxy resin mobility.

2.5 Influence of Surface Treatment on Composite Properties

The potential usage of natural fibres as substitutes for synthetic ones is hindered mainly by the hydrophilic nature of natural fibres. This propensity to absorb moisture results in poor mechanical properties and dimensional stability in natural fibre-reinforced composites (Hashim et al., 2017).

Various methods have been employed to improve the properties of natural fibre reinforced polymer composites. Abundant research has been conducted on evaluating how these techniques impact the performance as well as the resulting properties of the composites. These methods are focused on altering the fibres, the matrix and/or the interphase.

Several surface treatment techniques have been employed to alter the fibre morphology and are broadly categorized as physical, chemical, and biological treatment. Chemical treatments are the most commonly utilized modification techniques owing to their effectiveness in altering the fibre surface and improving interfacial bonding (Nurazzi et al., 2021). These include alkali, acetylation, benzoylation, and silane treatment. Surface treatments refer to techniques that alters the surface of fibres to enhance their ability to bond with the matrix. This subsequently leads to improved properties in the composites made from these fibres.

2.5.1 Alkali Treatment

The poor wettability and weak interfacial adhesion of the natural fibres to the hydrophobic matrices is a result of hydroxyl groups that are present on the fibre surface. Alkali treatment or mercerization is the most common surface treatment to counter the hydrophilicity of the natural fibres. It is the most common method due to the low cost in comparison to the other techniques. This surface treatment refers to immersing the natural fibres in a Sodium Hydroxide (NaOH) solution of a known concentration for a certain duration and at a certain temperature. The alkali reacts with the hydroxyl group (OH-) that is found in the fibre hemicelluloses as shown by the formula:

$$\text{Fibre-OH} + \text{NaOH} \rightarrow \text{Fibre-O}^{-}\text{-Na}^{+} + H_2O + \text{impurities}$$

Equation 2.1

This technique leads to defibrillation, whereby the untreated fibre bundles are broken down into single fibrils by dissolving the hemicellulose (Nurazzi et al., 2019). This facilitates the unidirectional arrangement of the fibrils in the tensile deformation direction resulting in improved mechanical properties. The hemicellulose, lignin, wax, and oils present in the fibre surface are removed by NaOH leaving the surface with a rough texture and a reduced fibre diameter. Incidentally, the effective surface area of the lignocellulosic fibres that adheres to matrix is increased. This action improves the matrix-fibre interfacial bonding because it exposes fibre cellulose content while allowing for better fibre wetting (Chandrasekar et al., 2017). The process increases the percentage composition of the cellulose in the fibres that is instrumental in fibre-matrix adhesion. The bulk of research that has incorporated alkali treatment has comparatively analysed the mechanical, moisture absorption, thermal, chemical, fracture and morphological properties of treated and untreated fibre-based composites and studied the underlying mechanisms for the changes. Alkali-treated natural fibre reinforced composites have better fibre-matrix adhesion, therefore improved properties. Alkali solution concentration, immersion duration and temperature are important variables during this treatment that influence the performance results (Fiore et al., 2015; Hashim et al., 2017; Mohd Nurazzi et al., 2019)

2.5.2 Effects of Fibre Treatment on Mechanical Properties

Lassoued et al. (2018) studied the outcomes of alkali treatment on the Tunisian palm fibres' mechanical properties. The fibres were treated with different NaOH concentrations of 0.5%, 0.75%, 1%, 2% and 5% and over different durations of 2, 5, 7 and 24 hours. They were then rinsed with distilled water to remove residual NaOH before being dried at room temperature and atmospheric pressure. The 2 hour- 2%NaOH treated fibres gave the most optimum result with a tensile strength of 242.6 MPa and Young's modulus of 7750.3 MPa. Scanning Electron Microscopy (SEM) showed untreated fibre surface to be irregularly shaped with presence of impurities whilst the treated fibre surface is smoother and clean. The treatment removes the impurities on the fibres allowing them to adhere better in composites. Naguib et al. (2015) reported that alkaline treatment removed the network structure surrounding the fibre surface, and therefore enhanced the free hydroxyl groups to interact with the matrix leading to strong interfacial adhesion.

Manalo et al. (2015) studied the influence of testing the mechanical properties of alkali-treated bamboo-fibre reinforced polyester composites at elevated temperatures, varying the NaOH concentration from 4% to 8% by weight. An improvement of flexural, tensile, and compressive strength of the composites by 7, 10 and 81% respectively, by treating the bamboo fibres with 6% NaOH was observed. The authors credited better adhesion of the fibres to the NaOH as the driving factor for this. Significant delignification and fibre degradation was associated with the observed decrement in the mechanical properties of the 8% NaOH treated composites. The damage, due to high alkali concentration, rendered the fibres unable to efficiently transfer loads to the matrix, thereby compromising the composite's performance.

Although alkali treatment is the most utilized surface treatment, techniques like acetylation have been found effective by researchers. Acetylation is an esterification method that introduces an acetyl functional group (CH_3COO^-) to an organic compound. Senthilraja et al., (2020) varied the acetyl chloride concentration (from 2% to 10% in increments of 2%) and soaking time for palm fibre that reinforced vinyl ester polymer resin to evaluate the most optimum treatment. The highest tensile strength of about 35 MPa was found for the composite made of vinyl fibre soaked for 98 hours in 6% acetyl chloride solution. The highest impact stress of about 42 MPa came from 24-hour fibre soaking time at 6% concentration.

A comparative investigation of the effects of chemical treatments on mechanical properties of *Grewia serrulata* bast and polyester composites was done by Mahesha et al. (2018). The fibres underwent an initial alkali pre-treatment with 5% NaOH, followed by additional chemical treatments including acetylation, permanganate, and Silane coupling treatment. Composite samples with a fibre composition of 15 wt.% were manufactured by hand lay-up method. Results showed that the tensile modulus of the composites increased by 27% and 8% with 1-hour-1% silane and 5% acetylation treatments, respectively. The tensile strength of the acetylated fibre-reinforced composite was enhanced by 53%, while the silane treatment led to a 10% increase in tensile strength. Furthermore, flexural strength improvements of 73% and 24% were observed for composites treated with silane and permanganate, respectively.

The influence of benzoylation and alkaline treatment on Sugar palm fibre’s tensile, and morphological properties was investigated by Izwan et al., (2020). Initially, the fibres underwent NaOH pre-treatment at a concentration of 18% for 30 minutes, followed by treatment with benzoyl chloride. Benzoyl chloride was mixed with 10% NaOH, and the immersion duration varied between 10, 15, and 20 minutes. SEM analysis revealed a reduction in fibre diameter after they were treated, attributing this to the hydrolysis of waxy surface residues. The highest tensile strength and modulus were achieved at 173.99 MPa and 6.64 GPa, respectively, with a 15-minute immersion treatment. Prolonged immersion for 20 minutes led to a degradation of the fibre's tensile properties due to excessive chemical reactions.

2.5.3 Effect of Fibre Treatment on Moisture Absorption Properties

The hydrophilic natural fibres and hydrophobic polymeric matrix incompatibility is caused by the affinity of the fibres to absorb moisture leading to weak interfacial adhesion. Water absorption is reported to occur through cavities and holes, deteriorates mechanical structure, and leads to a decline in the performance of composites (Mohammed et al., 2023). The absorption of moisture or water is considered a significant concern in composites due to the presence of chemical constituents in lignocellulosic fibres, such as cellulose and hemicellulose. These components are sensitive to weather conditions, particularly humidity and moisture, when composites are exposed to diverse outdoor environments (Chen et al., 2018).

Sanjeevi et al. (2021) has found water absorption to diminish the mechanical performance of natural fibre reinforced composites by observing that composites, of various fibre loadings, immersed in distilled water for 10 days, had lower tensile, flexural and impact strengths than their dry counterparts. Due to this, fibre treatment is employed to enhance interfacial adhesion and hence eliminate gaps between the fibre and the matrix. Fibre treatments are implemented to improve this issue due to their ability to remove the polysaccharide material on the fibre surface, that is hydrophilic. Moisture absorption of Luffa fibre reinforced epoxy composites were found by Bera et al. (2019) to increase with fibre loading when the tests were performed in saltwater, distilled water and sub-zero temperature conditions. This is postulated to be caused by the increase of free O-H groups that form hydrogen bonds with water molecules (Ismail & Ishak, 2018). Alkali treatment, by NaOH, was employed in the work by Ranakoti et al. (2022) to improve water absorption of tasar silk fibre waste- epoxy composites. The authors posited that, due to the elimination of the O-H groups, water entrapment by the composites was hindered.

The thickness swelling of the hybrid of sugar palm-glass fibre reinforcement in thermoplastic polyurethane resin was investigated by Atiqah et al. (2019). The authors were primarily interested in how different treatments of 6% alkali, 2% silane, and a mixture of both in sugar palm fibre affected the mentioned property. The combined alkaline-silane treatment substantially improved the thickness swelling performance of the composite by 21% thickness reduction. The work also reports a reduced water absorption value following treatments. Alkaline treatment removed the hemicellulose and the amorphous impurities that absorb water from the sugar palm fibres, and undergoing silane treatment further enhanced the fibre surface topology and hydrophobicity. The decrease in water absorption after treatment by alkali and silane was also reported by Abdullah & Ahmad, (2013) in their work on coconut-fibre reinforced polyester composites. The surface modified fibres strongly adhere to the resin and as a result there is lower water uptake due to the hydrophobic surface.

2.5.4 Effect of Fibre Treatment on Chemical and Morphological Properties

The studies focused on optimizing fibre treatment to improve wettability and enhance matrix adhesion involves exploring both chemical and physical modifications. Utilizing various analysis techniques, like Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FTIR), researchers investigate the alterations in the microstructure of fibres to observe the resulting changes facilitating improved wettability and matrix adhesion.

Research conducted by Ikramullah et al. (2018) reveals that prolonging the immersion of fibres in NaOH during alkali treatment proves ineffective, as it leads to damage to the vital microstructure components of the fibre. FTIR spectra analysis derived from Typha fibre subjected to alkali treatment indicates a significant disappearance of cellulose, hemicellulose, and lignin after just one hour of immersion. Conversely, a two-hour alkali treatment is deemed excessive, resulting in detrimental effects on all fibre constituents, particularly cellulose, thus impeding the fibre's suitability for use as a composite reinforcement. The findings suggest that a controlled exposure interval of fibres to alkali is crucial for enhancing mechanical performance. This controlled exposure leads to partial dissolution of hemicellulose, which in turn, increases the cellulose content and promotes the re-orientation of microfibrils in the axial direction(Castoldi et al., 2022).

Hashim et al. (2017) explored how various conditions of alkali treatment affected the kenaf fibre physical properties. The examined conditions were alkali concentration (2,6, and 10 w/v%), immersion temperature (27, 60 and 100°C) as well as duration of fibre immersion (30, 240 and 480 minutes). The results of the experiments showed an increase of kenaf weight loss with increase of alkali concentration and immersion time. Increase of immersion time, temperature as well concentration led to a decrease of fibre diameter and as a result a decrease in the cross-sectional area as well. A study by Roy et al. (2012) validates this experiment by positing that alkali treatment leaches out the non-cellulosic materials from fibres.

The concentration of NaOH used in fibre treatment is a crucial parameter that significantly impacts the microstructure of the fibre surface. An investigation performed by Vardhini et al. (2019) revealed through FTIR analysis that treating banana fibres with NaOH up to a concentration of 10% resulted in an improvement in cellulose content, lignin removal, and crystallinity. However, beyond this concentration, no further improvement was observed. Similarly, micrographs obtained through SEM analysis by Muhammad et al. (2016) demonstrated that increasing the concentration from 2% to 8% led to a softening of the Kenaf fibre surface. Nevertheless, at the 8% concentration level, damage to the fibre was observed. This is illustrated in Figure 2.8 from their study.

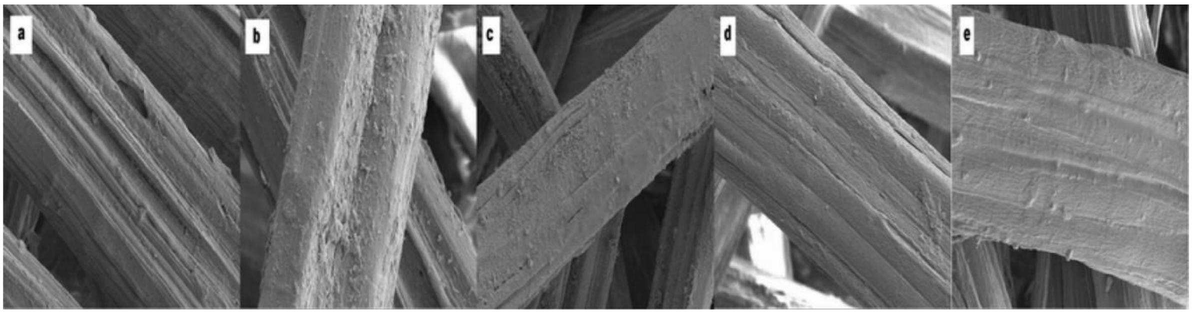

Figure 2.8: SEM images of Kenaf fibre surface (a) untreated, (b) 2% NaOH,(c) 4% NaOH, (d) 6% NaOH, (e) 8% NaOH (Muhammad et al. 2016)

In an investigation by Chen et al. (2021) of the alkali treatment effect on bamboo fibre and parenchyma cell, chemical composition analysis by Fourier Transform Infrared spectra showed that an evident absorbance band at about 1730 cm^{-1} in untreated fibres and its disappearance after alkali treatment. This band is attributed to the presence of C=O stretching band that exists in hemicellulose and lignin. Figure 2.9 shows this effect. Alkali treatment is postulated to result in partial dissolution of this hemicellulose and lignin removal.

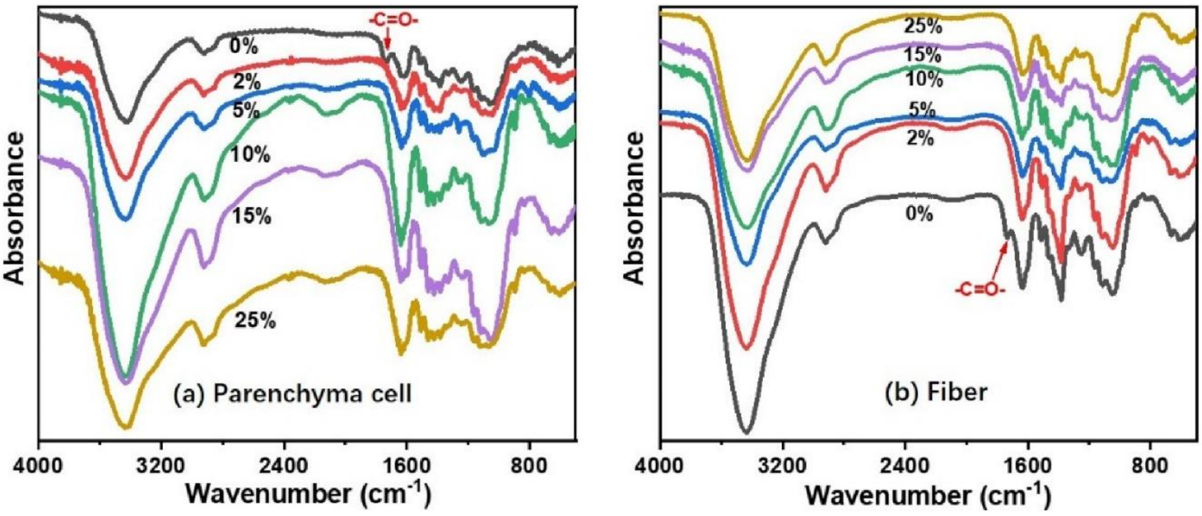

Figure 2.9: FTIR Spectra of (a) parenchyma cells and (b) fibres with alkali treatments (Chen et al., 2021)

Youbi et al. (2022) assessed the impact of alkali and silane treatments on the mechanical performance of Raphia Vinifera fibres. Silane treatment is attractive because it is seen as a promising and a versatile treatment technique. The fibres underwent silane treatment at concentrations of 1% and 5% for durations of 30 and 60 minutes, as well as alkali treatment with NaOH concentrations of 1%, 5%, and 10% for durations of 30 and 90 minutes. FTIR analysis, conducted in accordance with ASTM E1252 – 2013, examined the functional groups present in both treated and untreated fibres within the 400-4000 cm^{-1} region, with the spectra depicted in Figure 2.10. Notably, a broad absorption band in the 3600-3000 cm^{-1} range, with a peak at 3337 cm^{-1}, indicated the presence of OH- functional groups inherent in cellulose. Alkali treatment resulted in the degradation of hemicellulose and lignin, leading to a reduction in hydroxyl presence. SEM analysis of the fibre microstructure revealed detached microfibrils and traces of cork on the surface of untreated fibres. Alkalisation led to the disappearance of microfibrils, although prolonged exposure at higher concentrations induced surface degradation and the formation of cracks. Silane treatment enhanced surface roughness without the presence of cracks.

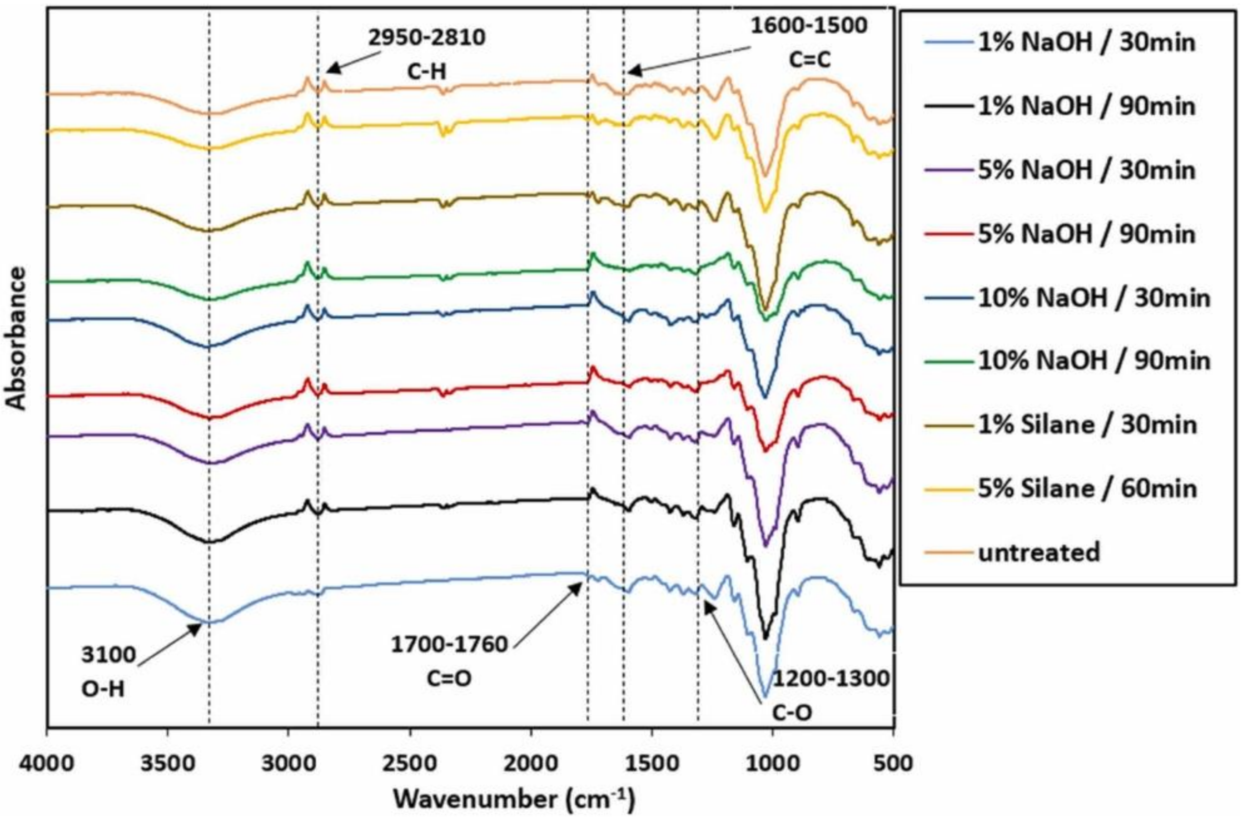

Figure 2.10: FTIR Spectra of Raphia Vinifera fibres(Youbi et al., 2022)

The impact of silane treatment extends beyond the surface of fibres, as demonstrated in the morphological analyses of composites comprising wood and palmyra fibre with recycled

Acrylonitrile-Butadiene Styrene(ABS) as a matrix (Chauhan et al., 2021). The study revealed that untreated fibre reinforced composites exhibited numerous cavities and voids, pullout fibres, and fibre agglomerations, contributing to the incompatibility between the fibres and polymer matrices. Treatment with 10% and 20% silane resulted in enhanced and uniform morphology in the composites, leading to improved tensile performance. However, a further increase in silane concentration correlated with a decrease in the tensile properties as it damages fibre surfaces.

Research has also investigated silane treatment method and its synergistic effect when coupled with alkali treatment. In their study, Asim et al. (2016) investigated the effects of alkali and silane treatments on Kenaf and pineapple leaf fibres. FTIR analysis revealed a partial leaching of lignin and removal of hemicellulose in pineapple leaf fibres, with alkali treatment demonstrating a stronger effect on hemicellulose removal than on lignin. SEM observations indicated the presence of impurities on the surfaces of untreated fibres, whereas treated fibres exhibited cleaner and smoother surfaces, attributable to the impurity-removing properties of silane. Diameter measurements revealed that untreated fibres had larger diameters compared to their treated counterparts. The combined treatment of 2% silane and 6% NaOH yielded the most optimal interfacial shear strength, measuring 4.5 MPa for pineapple leaf fibres and 2.3 MPa for kenaf fibres.

In another study, Orue et al. (2016) conducted a comparative analysis involving alkali treatment, silane treatment, and a combination of both techniques. Sisal fibres underwent alkali treatment with 2 wt.% NaOH at room temperature and a solution ratio of 1:20, followed by reflux at 100°C for 90 minutes. Subsequently, the fibres were rinsed to achieve a pH of 6 and then dried. Silane treatment involved the use of a 2% v/v aqueous silane solution with continuous stirring for 3 hours. Composites were fabricated using a PLA matrix, and subsequent physical, morphological, and tensile tests were conducted. The improved tensile properties were attributed to enhanced fibre-matrix bonding resulting from both NaOH and silane treatments. SEM micrographs revealed voids in the fibre structure of fibres treated with silane and NaOH, which were subsequently filled with PLA, facilitating better interlocking within the composite.

The employment of Poly(methyl methacrylate) (PMMA) film via admicellar polymerization has been identified as a means to enhance the interfacial adhesion between alkali-treated sisal fibres and unsaturated polyester, as demonstrated by Sangthong et al. (2009). The authors achieved optimal results with a 30% volume fraction of fibres, 30mm fibre length, and a PMMA concentration of 0.075% v/v. Admicellar-treated fibres exhibited robust bonding under SEM examination, while untreated fibres exhibited fracturing, leading to interfacial debonding and fibre pullouts within the composites.

In their study on hemp fibre reinforced polypropylene composites, Sullins et al. (2017) demonstrated that incorporating 5 wt.% of maleic anhydride grafted polypropylene (MAPP) into the polypropylene (PP) matrix effectively replaces the need for NaOH treatment on the fibres, while still enhancing the composite mechanical properties. Moreover, their investigation revealed that composites with a 30 wt.% fibre loading exhibited superior tensile and flexural properties compared to both 15 wt.% composites and pure polypropylene. Pre-treating the hemp fibres with NaOH prior to composite fabrication did not confer significant advantages over using MAPP alone. This coupling agent facilitates strong interfacial adhesion between the nonpolar PP matrix and the polar natural fibres, thereby enhancing the mechanical properties of the composite(Holbery & Houston, 2006).

The combination of treated fibre morphology and FTIR Spectroscopy has yielded valuable insights into the mechanisms governing interfacial adhesion between fibres and polymers. SEM micrographs from the study conducted by Mazzanti et al. (2019), which investigated the impact of fibre morphology on the reinforcement of poly-(lactic acid)/hemp composite, revealed a reduction in the presence of thick fibre bundles following alkali treatment. This treatment effectively removed lignin, hemicellulose, waxes, and oils from the fibre surface, resulting in increased roughness. This roughness plays a crucial role in exposing cellulosic hydroxyl groups, thereby promoting enhanced interfacial adhesion and, consequently, improved mechanical properties.

2.5.5 Effect of Fibre Treatment on Thermal Properties

Thermal properties are evaluated to investigate the thermal stability of material as well as their viscoelastic behaviour in high temperature applications. Thermal stability is defined as the maximum temperature at which a fibre decomposes (Neto et al., 2021). The practical implications for these parameters are to establish the temperature range that the composites can be utilized to the point where they degrade. The thermal properties of fibres as well as of the composites are key research areas in understanding them. A thermogravimetric analysis (TGA) equipment is used to study the thermal stability for the fibres and the composites. The TGA curves of lignocellulosic fibres show similar thermal decomposition pattern because of their comparable properties. The TGA curves are usually analysed in conjunction with Derivative Thermogravimetric(DTG) curves to establish the decomposition of fibre constituents at specific temperatures when the fibres are heated (Azwa et al., 2013). Figure 2.11 shows the typical weight loss curve(TG) and derivative(DTG) curve in the decomposition of a natural fibre across a temperature range (Mohammed et al., 2023; Sadrmanesh & Chen, 2019).

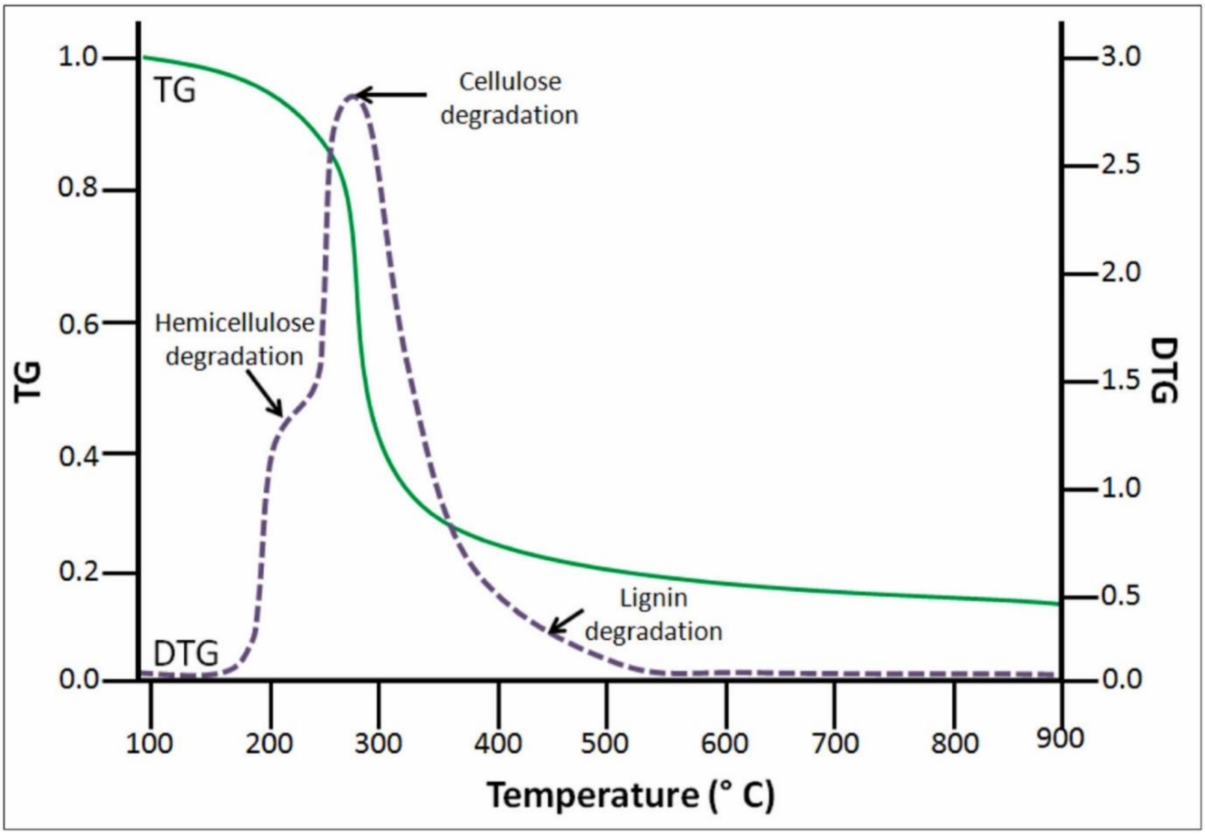

Figure 2.11: Typical TGA and DTG curves for a natural fibre (M. Mohammed et al., 2023; Sadrmanesh & Chen, 2019)

It has been reported that fibre treatment enhance the dynamic mechanical properties of sugar palm fibre-based Phenolic Composites by Mohammed et al. (2017), who treated sugar palm fibres by a combination of seawater for 30 days and 0.5% alkali treatment for 4 days. The storage modulus (E’) which measures the energy required to distort a sample, or in other words, the stiffness of a solid material, was highest for alkali treated composite, followed by the saline treated composite, and being the lowest for untreated fibre composite. The chemical effect of alkali treatment to dissolve hemicellulose and lignin, as well as the physical effect of the seawater to remove the external fibre surface, are credited for this. It has been reported by Jawaid et al. (2013) that stiffness is inherently influenced by the effective interfacial stress transfer, which is a product of stronger fibre-matrix adherence due to alkalisation.

The work into how alkali treatment coupled with hybridization affects sugar palm yarn/glass fibre reinforced unsaturated polyester composite thermal properties was performed by Nurazzi et al. (2019), by analysing the dynamic mechanical analysis and thermal properties. The loss modulus, which quantifies the energy released when the material turns viscous, was observed to have an increasing peak value with the increase in fibre loading, and after alkali treatment of 1% NaOH. The 40 wt.% fibre alkali treated composite with sugar palm to glass fibre ratio of 1:1 had the highest loss modulus of around 700 MPa and 1184 MPa at about the glassy area and the glass transition area, respectively. The authors posit that due to alkali treatment, which improves interfacial adhesion, and a reduced movement of the unsaturated polyester chains, is the reason why the glass transition temperature (T_g) from the loss modulus was shifted to a higher temperature.

A work by Barreto et al. (2011) paired alkali treatment with the bleaching technique to improve the thermal stability of sisal fibre reinforced phenolic composites. The authors employed NaOH of 5 and 10%, with Sodium Hypochlorite ($NaClO/H_2O$) (1:1). The thermogravimetric analysis results showed that the first stage of decomposition occurs at 341°C for the untreated fibre composite and was shifted to 344 °C for 5% and 10% treated fibre composite. The degradation temperatures of 438 °C, 440 °C, and 444°C were obtained for raw sisal, 5% NaOH sisal, and 10% NaOH sisal composites respectively in the second stage. The substantial increase in degradation temperature was reported in the last stage from 527°C with raw sisal fibre composites to 535°C and 542°C for 5% NaOH treated and 10% NaOH treated sisal fibre composites respectively. This shows an improvement of 10°C by treating fibres with 10% NaOH. The authors deduced that the results were an attribute to the improved diffusion of the polymeric matrix in the alkali treated sisal fibres.

Rafiqah et al. (2023) reported slight increase in thermal stability of Miswak fibre-reinforced polylactic acid (PLA) by pre-treating the fibres with 1%, 2% and 3% of NaOH. The authors posited that the increase in thermal degradation temperatures point towards a more stable interfacial bond in the alkali treated fibres and the PLA. This has been corroborated in the work by Sakuri et al. (2020) through interfacial shear strength tests, which showed that alkali treatment increased the interfacial bonding of Cantala fibre/microcrystalline cellulose composites by 47.4%.

2.6 Sustainable Development of Natural Fibre Reinforced Composites

2.6.1 Life Cycle Assessment Studies

The use of water, energy, chemicals, greenhouse gases emissions, waste materials throughout the supply chain of natural fibre reinforced composite materials are important factors in terms of sustainability. Consequently, the environmental ramifications of the composites have materializable alongside various other sectors. The production of natural fibre polymer composites has exacerbated existing environmental issues such as shortages in landfill space, plastic pollution, increased energy consumption, and depletion of petroleum resources (Malviya et al., 2020). Escalating environmental consciousness, burgeoning waste management challenges, and soaring crude oil costs have engendered significant apprehensions from legislative and consumer standpoints (Muthu, 2019; Wellbrock et al., 2020). In 2015, the United Nations adopted the Sustainable Development Goals (SDGs), which include objectives related to promoting responsible consumption and production of natural resources, as well as reducing carbon dioxide and greenhouse gas emissions (Suárez et al., 2021). Automotive manufacturers are under pressure to comply with both political guidelines and internal specifications, as well as with constantly changing individual customer wishes. Mercedes Benz have shown their commitment to sustainability with their climate protection goals for their new vehicle fleet to be CO_2-neutral by 2039 (Daimler, 2019).

Assessing the sustainability of natural fibre reinforced composites involves employing life cycle assessment (LCA) analysis. This method scrutinizes the potential environmental impacts of products, spanning from the extraction of raw materials to processing, manufacturing, product utilization, and eventual disposal (Malviya et al., 2020; Mansor, Mastura, et al., 2019). This aspect of product development helps engineers and decision makers in the process of selecting materials. The **cradle-to-grave** concept, as applied to LCA analysis, involves assessing and analysing the inputs, outputs, and environmental impacts of a product system from its inception to its disposal. The **cradle-to-gate** method focuses on assessing environmental impacts from raw material extraction through manufacturing, excluding considerations for transportation, sales, usage, and end-of-life stages (Gkoloni & Kostopoulos, 2021; Gonzalez et al., 2023). Additional approaches are the **Gate-to-Gate** and the **Gate-to-Grave** that consider just the production stage, and the usage up to the end-of-life stages, respectively. These approaches are delineated in Figure 2.12 and Table 2.4.

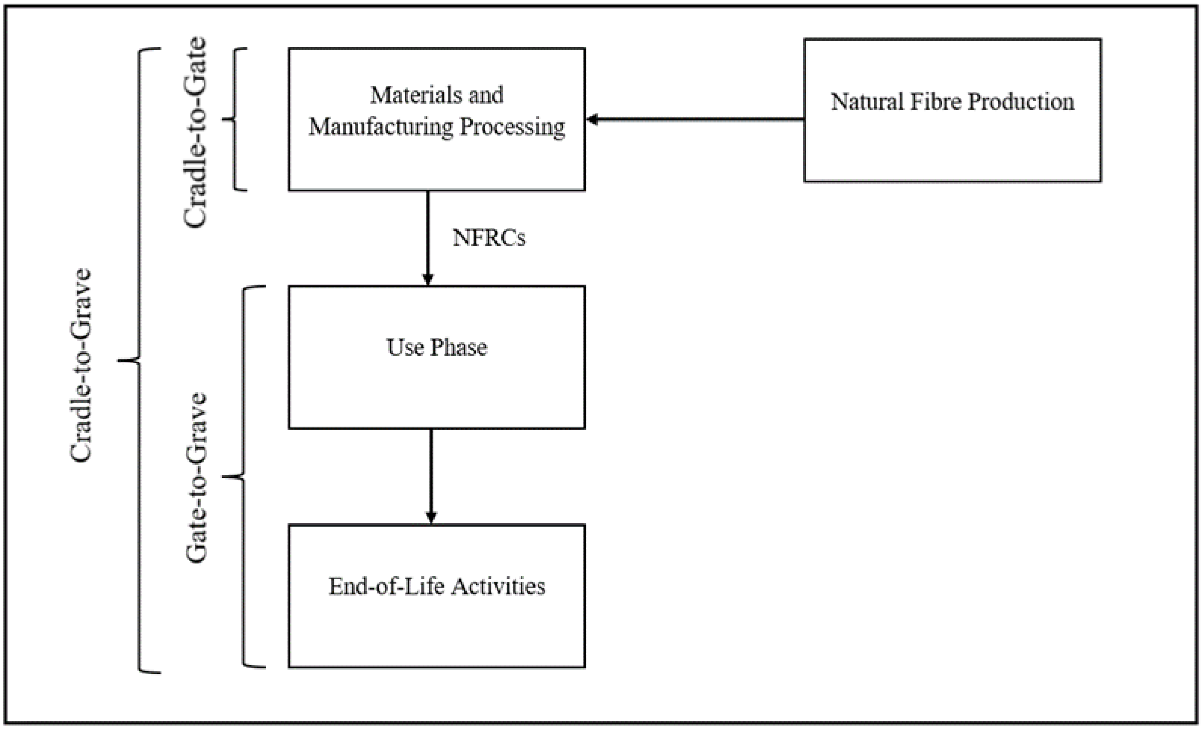

Figure 2.12: Life Cycle Analysis phases of a natural fibre composite (Adapted from (Hermansson et al., 2019)

Table 2.4: Life Cycle Analysis methods

LCA	Boundaries
Cradle-to-Gate	-Raw material extraction -Manufacturing
Gate-to-Gate	-Production
Gate-to-Grave	-Usage -End-of-life
Cradle-to-Gradle	-Raw materials extraction -End-of-life

Synthetic composites impose significant environmental harm due to their substantial demand for resources, generation of waste, and intensive materials processing. Furthermore, they lack biodegradability and cannot be recycled at their end-of-life (Ead et al., 2021). Natural fibre composites are purported to provide environmental benefits, including diminished reliance on non-renewable energy and material sources, decreased pollutant emissions, reduced greenhouse gas emissions, improved energy recovery, and biodegradability at the end of their life cycle (Joshi et al., 2004). However, there are environmental concerns in the life cycle of natural fibre composites, particularly the emission of nitrogen and phosphorus during cultivation, large arable-land requirements, and ecosystem quality. Luz et al., (2010) reported that the environmental impacts of sugarcane bagasse-PP composites include abiotic depletion, acidification, eutrophication, and greenhouse gas emissions.

In a study by Cabrera et al., (2023) the incorporation of banana rachis into recycled high-density polyethylene (r-HDPE) was found to have an inverse relationship with environmental impacts. Specifically, the composite with a 20 wt.% fibre loading exhibited reduced freshwater, marine, and human carcinogenic toxicity compared to 100% r-HDPE. This finding is supported by a comparative life cycle assessment (LCA) conducted by Rodríguez et al., (2020) on coffee jar lids made from banana fibre, polylactic acid (PLA), and HDPE. The lid fabricated with 40 wt.% banana fibre, along with equal amounts of HDPE and PLA, demonstrated notably lower impacts on climate change, photochemical oxidant formation, and fossil depletion than the one manufactured solely from 100% HDPE. Furthermore, this lid exhibited superior performance compared to the counterpart made entirely from 100% PLA across 18 impact categories.

Harvesting and cultivation, as well as ethylene production were found to be two of the most impactful stages in producing Bagasse fibre reinforced polyethylene(PE) composites (Ita-Nagy et al., 2020). The graphical representation of the system boundaries in the production of the bio-composite is shown in Figure 2.13 to identify the inputs, process steps and outputs during the production. The use of agrochemicals during cultivation and harvesting of biomass is correlated with ozone formation, terrestrial acidification, freshwater eutrophication, and fossil resource scarcity. Sugarcane based PE composites have been found to pose a higher acidification and eutrophication impacts than their fossil-based counterparts (Liptow & Tillman, 2012).

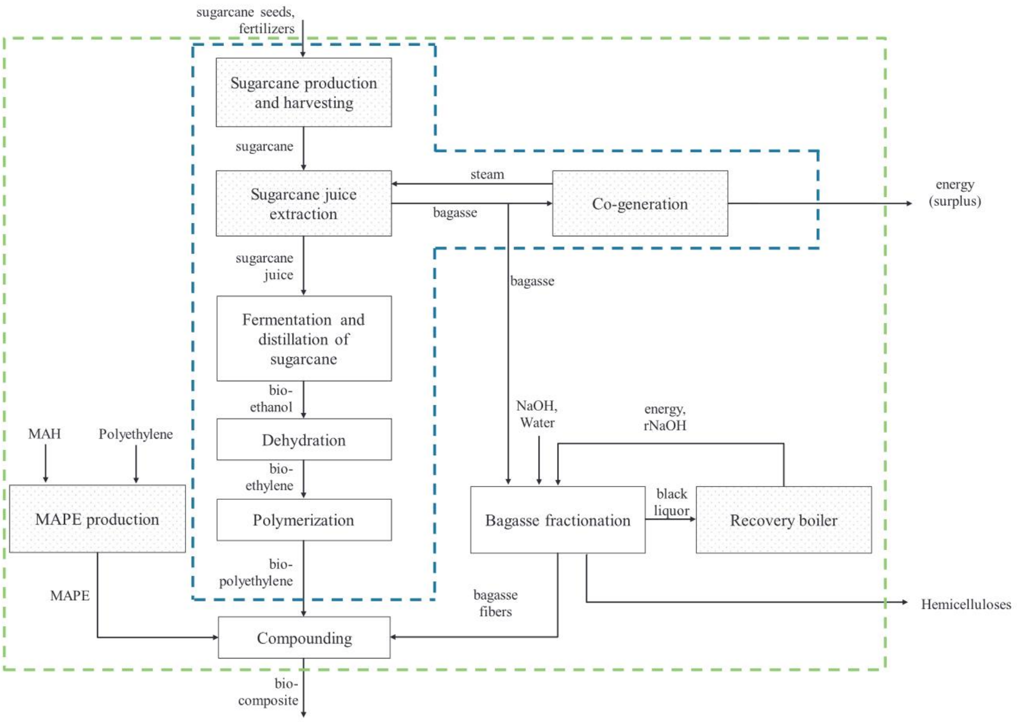

Figure 2.13: System boundaries of production of Bagasse fibre reinforced polyethylene composites (Ita-Nagy et al., 2020)

A research team in nova institute in 2019 measured the carbon footprint of flax, hemp, jute, and kenaf fibres in production for the automotive and insulation industry. The cultivation and harvest of natural fibres entails pesticide and fertilizer application, cutting down plants and fibre processing. The highest emissions were noted to be due to fertilizers, especially mineral fertilizers. The carbon uptake of the fibres was found to be between 1.3 and 14 kg CO_2 per kg of fibres, which will consequently be released at the end of life of the product (de Beus & Barth, 2019). The results from their study based on the carbon footprint due to field operations, seeds, fertilization, pesticides use, fibre processing, and transportation are presented in Table 2.5.

Table 2.5: Carbon footprints of natural fibres from Nova Institute study

Natural Fibre	Carbon footprint of one tonne natural (in CO_2-eq/tonne fibre)	
	Mass allocation	Economic allocation
Hemp (mineral fertilizer)	410	860
Hemp (organic fertilizer)	360	760
Hemp (dual use)	370	530
Flax	360	900
Jute	550	970
Kenaf	420	980

2.6.2 End-of-Life Considerations

Since natural fibre composites are purported to be renewable, their end-of-life phase of natural fibre composites entails the recycling, which can be achieved mechanically, chemically, or biologically. The integration of a circular economy into bio composites offers notable benefits to the environment, economy, and society alike.

In mechanical recycling, material is reduced to small fragments in the range of 20 and 200 mm through crushing, grinding, or milling (Gopalraj, 2009). The product can then be reprocessed as powders, fibres, or flakes to produce new products (Molina et al., 2021). This method is economical and effective. Mechanical recycled flax fibre reinforced-Maleic Anhydride grafted Polypropylene (MAPP) composite has been found to possess comparable tensile properties to reference flax-MAPP by Bensadoun et al., (2016). Mechanical recycling has however been found to result to a deterioration of products properties. Lopez et al., (2012) found that successive reprocessing of biodegradable polymer matrices diminished their tensile, flexural, and impact properties.

Hydrolysis, gasification, pyrolysis, and hydrocracking represent various chemical recycling methods aimed at depolymerizing polymer chains to recover monomers, which can then be reused as raw materials in both the fuel and polymer industries. In hydrolysis, water is utilized to dismantle large molecules into smaller units. Gasification involves the breakdown of carbon-containing polymers into carbon monoxide, hydrogen, and other gases, typically facilitated by air or oxygen. Pyrolysis takes place in an environment with limited oxygen, causing the decomposition of polymers. Hydrocracking, on the other hand, applies high partial pressure to transform high-boiling chemicals into lower-boiling ones (Elseify et al., 2020).

The biological recycling of natural fibre composites involves the degradation of biopolymers by microorganisms, air, and water. This approach is particularly useful for recycling natural polymers like cellulose and polylactic acid (Elseify et al., 2020). While the recycling of polymeric materials is well-established, the recycling of natural fibre reinforced composites is still undergoing research and development. Governing bodies such as the European Union have established guidelines and regulations stipulating that a substantial percentage of vehicles must be recycled or reused.

Another aspect to consider at the end of a product's life is energy recovery via incineration or combustion, a method frequently utilized. While this approach yields substantial energy from polymers, it also produces harmful emissions posing health risks (Shanmugam et al., 2021).

2.7 Chapter Summary & Conclusion

In this chapter, a comprehensive review of natural fibre reinforced composites was presented. The examination encompassed the classification of natural fibres, their structures, and the polymer matrices. Furthermore, the influence of fibre loading and surface treatments on water absorption, mechanical, thermal, and morphological properties of natural fibre reinforced composites were assessed. Lastly, the chapter reviewed the sustainability of bio-composites by assessing their life cycle assessments and end-of-life scenarios.

As per reviewed literature, selecting the optimal fibre loading has emerged as a crucial factor in maximizing the performance of natural fibre-reinforced composites. Low fibre loadings tend to impede stress transfer efficiency, while excessively high loadings can lead to issues such as agglomeration, cracking, and the formation of voids within the composites. Moreover, the use of various fibre surface treatments, such as alkali, acetylation, and benzoylation has been found to improve fibre-matrix adhesion by various researchers. Alkali treatment has emerged as the most preferred technique due to its effectiveness and simplicity. Most studies evaluated concentrations ranging from 1 to 10wt% NaOH, with the most effective treatment often identified at median concentrations. Increasing the concentration of alkali treatment has been found to be beneficial, but high concentrations of alkali have been shown to compromise the mechanical properties of natural fibers and their effectiveness as polymer reinforcements.

CHAPTER 3: METHODOLOGY

3.1 Introduction & Overview

This chapter introduces the materials and outlines the procedural steps employed to achieve the research aim and objectives. The reason for performing the tests and any relevant information is discussed as well as the apparatus and methods used in the experimental work.

The specific objectives of this research are to review the present literature on the effect of fibre loading and surface treatment on natural fibre reinforced polymer composites, characterize Mokolwane fibre, fabricate mokolwane fibre reinforced composite, and investigate the influence of alkali treatment and fibre volume fraction on the properties of the composite. The previous chapter reviewed literature and found that alkali treatment is commonly employed to rectify lignocellulosic fibres' propensity to absorb moisture. Alkali treatment has been noted for its effectiveness, simplicity and being economical over other chemical treatments. The previous chapter also reviewed the influence of fibre loading on the performance of the composite, underscoring the significance of finding the optimum treatment and loading on the overall properties.

This chapter outlines the materials, and the experimental procedures carried out to achieve the objectives of this research work. This is done by providing a description of the extraction and characterization of mokolwane fibres, fabrication of mokolwane fibre reinforced composites and experimental evaluation of mechanical, water absorption, and morphological properties of mokolwane fibre reinforced composites. Mokolwane fibres are characterized by water absorption test, fibre diameter test, thermogravimetric analysis (TGA), Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM). The fibre reinforced composites were fabricated by varying the fibre weight percentage (40wt%, 50wt% and 60wt%) of mokolwane fibres in the composite as well as varying the Sodium Hydroxide (NaOH) concentration (untreated, 2%, 4% and 6%) used to treat the fibres. The composites were then cut to standard dimensions before they underwent subsequent experiments. The composites samples are then experimentally evaluated through density, water absorption, thickness swelling, tensile, flexural, impact tests and Scanning Electron Microscopy (SEM).

These quantitative and qualitative techniques collectively characterise and establish understanding of the influence of alkali treatment and the fibre loading on the overall properties of the fibres and the composites. The methodological framework of this study is shown in Figure 3.1.

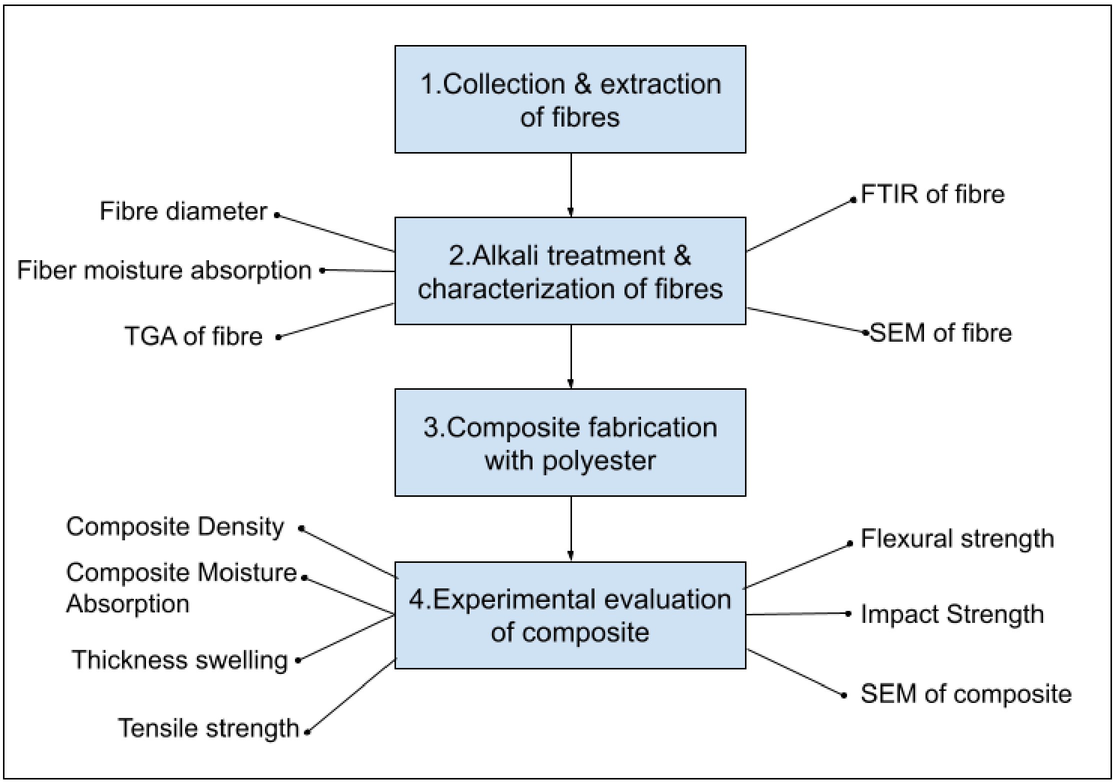

Figure 3.1: Methodological Framework

3.2 Materials and Sample Preparation

3.2.1 Extraction of Mokolwane fibre

The leaflets of the Mokolwane tree were selected as the optimal source for fibre extraction due the ease of extraction compared to fibres from the stem. The schematic of the process of extracting the fibres is given in Figure 3.2. The leaflets were harvested from Nata village, Central District, Botswana. The leaflets underwent a 10–14-day soaking period in water, a technique known as water retting. This process served a dual purpose: facilitating easier extraction from the leaflets and breaking down waxy epidermal tissues, adhesive pectin, and hemicellulose that bind fibre bundles together (Cao et al., 2006). Moreover, it aided in the removal of dirt and colouring matter from the fibres. To ensure the extraction of clean fibres, the water was replaced every 3 days. Following the water retting process, the fibres were manually stripped from the leaflets, washed with tap water, and left to dry in the sunlight.

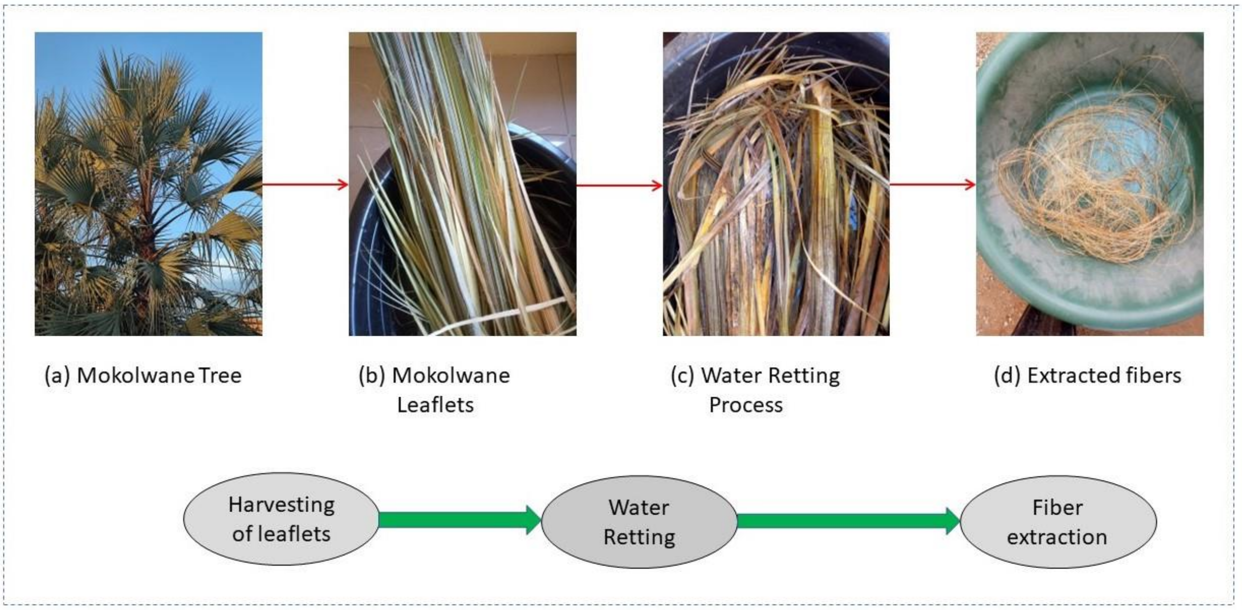

Figure 3.2: Fibre Extraction Schematic

3.2.2 Alkali treatment of Mokolwane Fibre

Prior to alkali treatment, the stripped Mokolwane fibres were sun-dried for 8 hours to remove moisture. The sodium hydroxide pellets used to prepare the treatment solutions were supplied by the university Chemistry department. They were used to make solutions of 2,4 and 6w/v% NaOH in the beakers {Studies have demonstrated that alkaline concentrations exceeding 6% significantly damage the microstructure of natural fibers, leading to poor mechanical properties. As a result, concentrations above 6% have been empirically excluded from this work. These studies include (Cai et al., 2016; Khan et al., 2019; Mittal & Sinha, 2017; Owen et al., 2017)}. Following that, the fibres were immersed and soaked in the prepared alkali solutions, as depicted in the beakers shown in Figure 3.3, for a duration of 24 hours. After soaking, the fibres underwent rinsing with tap water to eliminate any residual alkali before being subjected to drying for 10 hours at 80°C in the oven. Subsequently, they were stored in desiccators until further processing.

Figure 3.3: Alkali treatment of Mokolwane Fibres

3.2.3 Preparation of Composite

Pre-accelerated unsaturated polyester resin, under the commercial name NCS 901 PA was purchased from NCS resins, Durban, South Africa along with the hardener, Methyl Ethyl Ketone Peroxide (MEKP). NCS 901 PA is a moderately curing ortho phthalic unsaturated polyester resin known for its rigidity and thixotropic properties. The properties of the polyester resin as given by the supplier are outlined in Table 3.1. NCS 901 PA was supplied along with the hardener, Methyl Ethyl Ketone Peroxide (MEKP), under the commercial name Butanox M-50.

Figure 3.4: NCS 901 PA and Butanox M-50

Table 3.1: Properties of NCS PA

Typical Properties of NCS 901 PA (unfilled castings) Prepared, post-cured and tested in accordance with SABS 713-1974, as amended	
Temperature of deflection – under load (1. 80 MPa),	90
Water Absorption:	
a) Increase in mass after 28 days immersion, mg	100
b) Loss in mass after drying, mg	45
Barcol (GYZJ 934-1) hardness	45
Tensible strength, MPa	76
Flexural strength, MPa	84
Flexural modulus, MPa	3930
Compressive Strength, MPa	152

The fibres were cut to 3mm to make a discontinuous fibre reinforced composite. The 30 x 30 x 5 mm^3 mould was fabricated to be used to make the fibre reinforced composites. Aluminium foil was laid in the mould to allow easier removal of the composites by minimising the chances of the resin sticking to the inner surfaces. This procedure has been performed by Mbeche et al. (2020) to fabricate Sisal/Cattail hybrid polyester composite. The fabrication of the composite was performed at room temperature and conditions. Polyester resin was cured by mixing it with 1.5 per hundred resin(phr) MEKP for use as a polymer matrix. A mould of cured polyester resin was made to act as a control in addition to the fibre reinforced composite moulds of varying fibre loading and alkali treatment. The mixtures were stirred to completely mix the fibres with the resin uniformly before being poured into the moulds. This was done to prevent agglomeration of fibres, which results in deteriorated properties of the composites. The resulting mixtures were left to cure for 24 hours at room temperature. Composite configurations with fiber loadings of 40 wt.%, 50 wt.%, and 60 wt.% were fabricated. As discussed in Subsection 2.4, the optimal fiber loading for various composites is found to be around 50 wt.%. Table 3.2 showing the nomenclature of the specimen given their fibre loading and the type of treatment is given. Various specimens were cut from the moulds in accordance with different standards to prepare for experiments as shown in Figure 3.5(d).

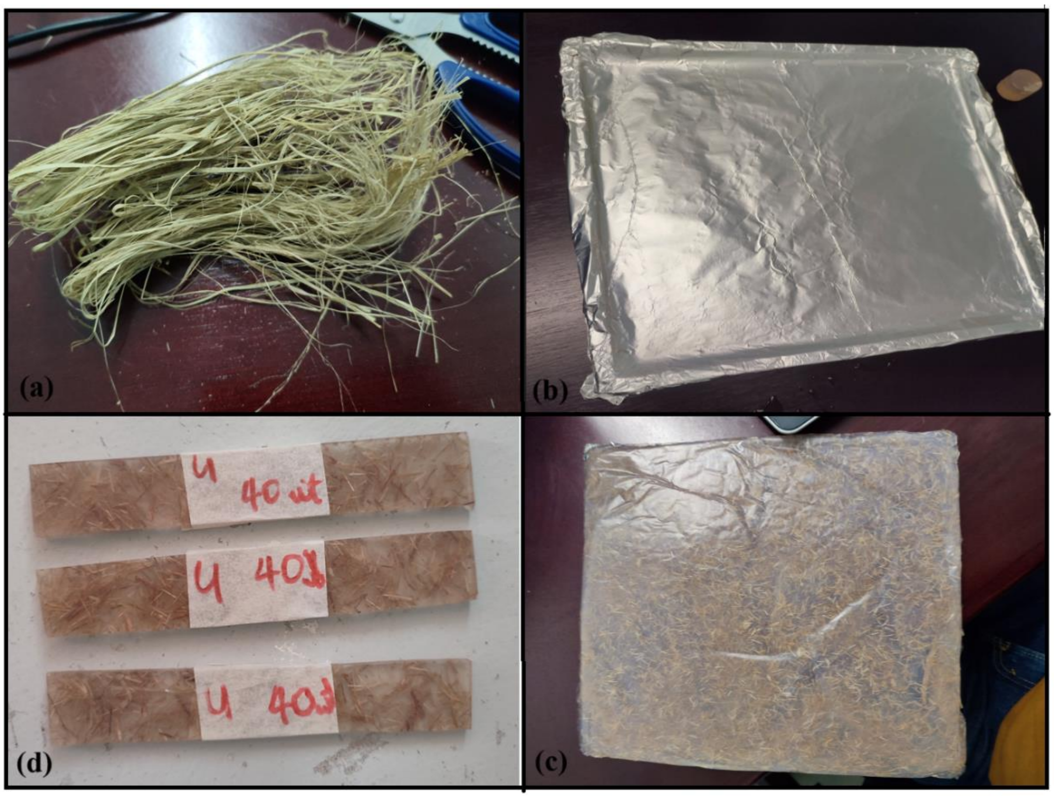

Figure 3.5: (a) Extracted Mokolwane fibre (b) Mould wrapped in Aluminium foil (c)Fabricated composite and (d) Prepared specimens for testing

Table 3.2: Nomenclature of specimens.

Designation	Polyester (wt.%)	Fibre (wt%)	NaOH (w/v)
Neat	100	0	-
UPM_40	60	40	-
UPM_50	50	50	-
UPM_60	40	60	-
TPM_40_2SH	60	40	2
TPM_50_2SH	50	50	2
TPM_60_2SH	40	60	2
TPM_40_4SH	60	40	4
TPM_50_4SH	50	50	4
TPM_60_4SH	40	60	4
TPM_40_6SH	60	40	6
TPM_50_6SH	50	50	6
TPM_60_6SH	40	60	6

3.3 Characterization of Mokolwane Fibre

3.3.1 Fibre Diameter

The diameters of untreated and treated mokolwane fibres were evaluated using Leica DM2700M Optical Microscope. Firstly, 50 single fibres were glued to a black paper before being placed for observation under the optical microscope as shown in Figure 3.6. The black background allows for easy observation of the fibres under the microscope. This microscope uses light and lenses to generate magnified images of the specimens under focus. The software allowed for the measurement of the diameters to be performed. Diameter measurement accuracy is difficult to obtain given the variation of the diameter along the fibre length, as well as the irregular fibre cross section. Therefore, average length of each fibre was evaluated using measurements of 3 locations along the fibre length.

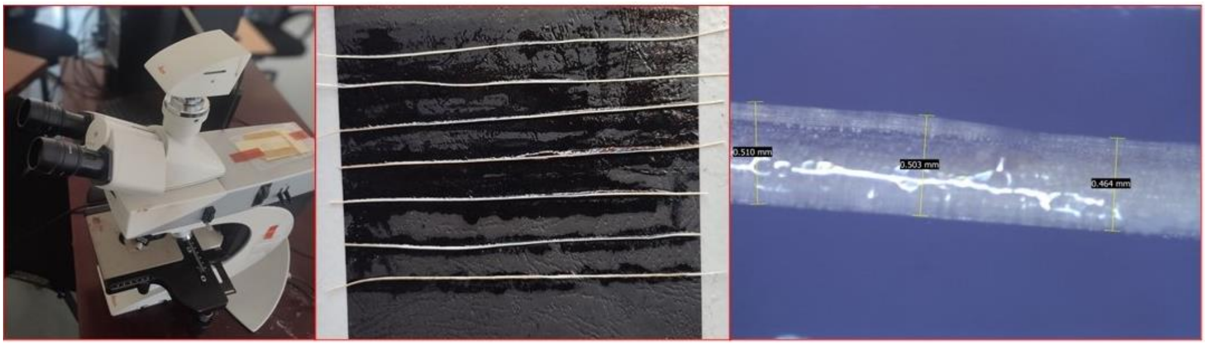

Figure 3.6: (a) DM 2700M Optical Microscope (b) Fibre layout (c) Optical Microscope image

3.3.2 Moisture Absorption of Fibres

The moisture absorption of mokolwane fibres was evaluated by the ASTM standard 570 – 98. The specimens were oven-dried at 50 ± 3 °C for 24 hours, cooled in a desiccator and then immediately weighed. The fibres were then immersed into boiling distilled water for 2 hours. After this the fibres were removed from the water and cooled in distilled water at room temperature for 15 minutes. The fibres were then removed and dried by a dry cloth to eliminate any surface water and then immediately weighed. The percentage increase in weight during immersion is calculated by Equation 3.1 as follows:

$$increase\ in\ weight(\%) = \frac{wet\ weight - conditioned\ weight}{conditioned\ weight} * 100$$

Equation 3.1

This experiment was repeated to evaluate the moisture absorption of alkali treated Mokolwane fibres. For each configuration, the test was performed 3 times and the average values calculated. The schematic diagram for the process is illustrated in Figure 3.7. Figure 3.8 shows the oven and the fibres during weighing and boiling process.

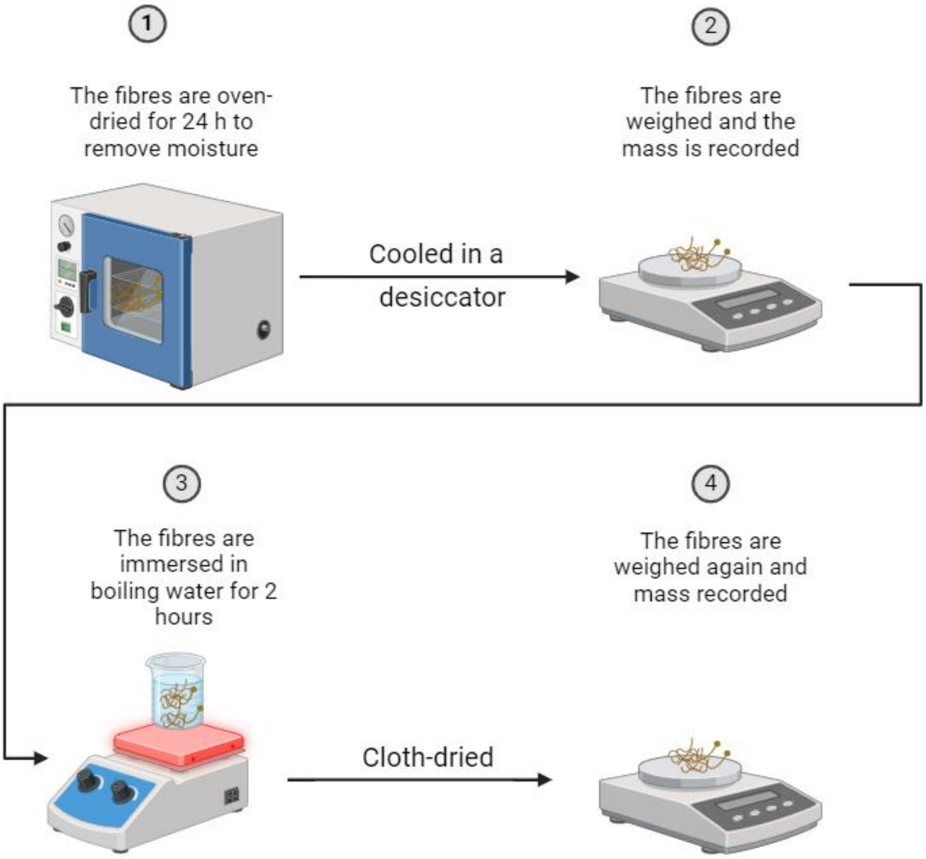

Figure 3.7: Fibre moisture absorption test schematic

Figure 3.8: (a) Fibre weighing (b) Boiling test (c) Oven-drying.

3.3.3 Thermogravimetric Analysis of Fibre

Thermogravimetric analysis (TGA) is a method of thermal analysis where the weight of a sample is monitored as it is exposed to an increasing temperature in a controlled environment. This technique allows for the measurement of changes in sample mass in response to temperature changes. TGA is usually performed as a change in sample mass as temperature increases or how mass changes when the sample is heated isothermally in a controlled atmosphere as a function of time (Menczel, 2020). Thermal stability study on fibres is a pivotal parameter in enhancing the composite properties and to ascertain the operating limit of the fibres (Kandemir et al., 2020). The inherent low degradation properties of natural fibres compromise their ability to reinforce, thus resulting in weaker composite materials. The structural constituents of natural fibres exhibit sensitivity to varying temperature ranges. It has been reported that the thermal stability of natural fibres can be improved by eliminating some portions of hemicellulose and lignin through chemical treatments (Heckadka et al., 2023).

In this work, TGA was used to evaluate the thermal stability of Mokolwane fibres by characterizing the sample mass loss of untreated and treated fibres due to temperature increase. This is carried out to understand how the alkali treatment affects the stability of Mokolwane fibres. The fibres were first pulverized to prepare for the experiment. The TGA701 Thermogravimetric Analyzer equipment in Figure 3.9 was used to evaluate the thermal stability. The different configurations of pulverized fibres evaluated were untreated fibres, 2%NaOH, 4NaOH, and 6%NaOH treated fibres.

The TGA701 Thermogravimetric analyser has 20 crucibles located on a carousel, hence a maximum of 20 samples can be evaluated simultaneously. A mass of 1 gram of each sample were placed in the 4 crucibles of the analyser. The TGA701 software allowed for labelling each sample by position of the crucible on the carousel and monitoring the mass of each as heat is applied. The pulverized fibres were heated from 30°C to a 700°C at a controlled heating rate of 10°C/min and the mass of the samples monitored and recorded throughout. The Windows® - based operating software on the connected computer was used to input parameters before the experiment as well as an output of data and graphs during the experiment. After the testing was done, the recorded results of weight versus temperature were then further analysed using ORIGIN software.

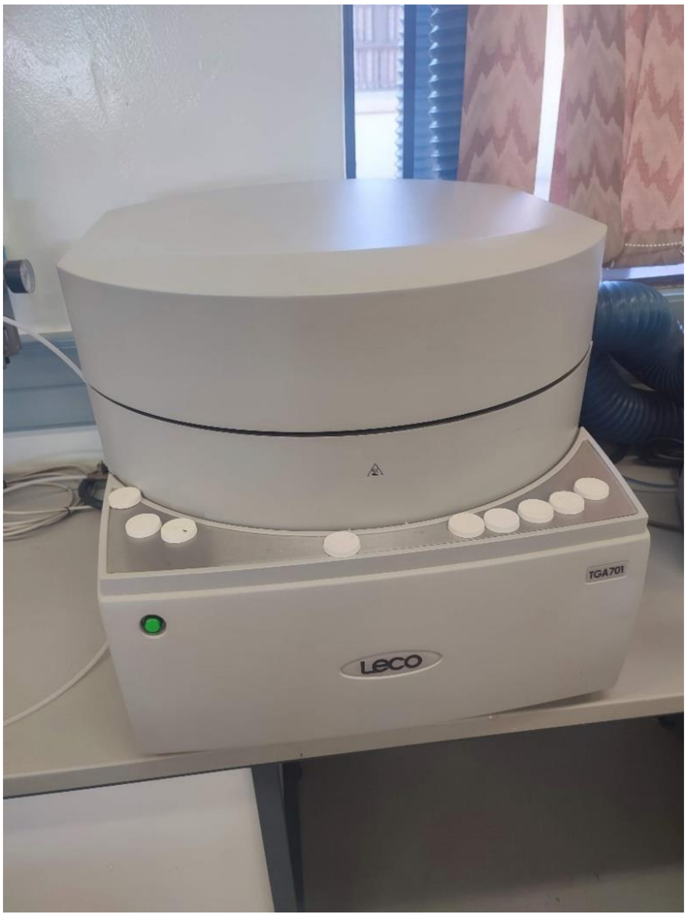

Figure 3.9: TGA701 Thermogravimetric Analyzer

3.3.4 Fourier Transform Infrared Spectroscopy (FTIR) of Fibres

Fourier transform infrared spectroscopy(FTIR) is a technique employed for the identification of characteristic absorptions, emissions, and photoconductivity of compounds to compare them with spectral libraries (Gore, 1958). Infrared spectroscopy relies on the response of molecular vibrations to electromagnetic radiation. When subjected to irradiation, the atoms within a molecule, held together by valence forces, absorb radiation at frequencies corresponding to their molecular vibrational modes, as elucidated by Koenig (1999). Since the vibrational energy levels are unique to each molecule, the resulting vibrational spectrum is often likened to a distinctive molecular fingerprint.

This here allows identification of organic compounds present in the mokolwane fibres by detecting different functional groups present, because each functional group has distinctive vibrational spectra. The presence of chemical constituents of natural fibres, like cellulose, hemicellulose, lignin, and pectin can be determined by establishing the functional groups from FTIR Spectra.

Furthermore, this method helps to assess the effect of alkali treatment on the chemical constituents of the fibre surface by observing the change in their molecular fingerprint. FTIR spectroscopy does this by detecting the changes in the chemical functional groups present in the fibres post-treatment. This in-depth information derived from FTIR Spectroscopy, helps ascertain the effect of chemical treatment on the Mokolwane fibres. The intensity of the absorption bands indicates the degree of alkali treatment and the extent of structural modification of the fibres. The information garnered from the use of this method allows the processing methods to be optimized to get improved fibre quality and performance.

To prepare the untreated and treated fibres for FTIR spectroscopy, they were first pulverized to meet the required size of the FTIR equipment. FTIR Spectroscopy was performed at the University Physics department on the Bruker Vertex 70V vacuum FT-IR spectrometer equipped with Attenuated Total Reflectance (ATR) mode to examine functional groups' changes in the fibre surfaces. Spectra spanning the range of 4000-1000 cm^{-1} were obtained for all the treated and untreated fibres. The FTIR equipment is shown in Figure 3.10. Figure 3.11 shows a schematic diagram of the FTIR Spectrometer. The data collected was analysed using ORIGIN software.

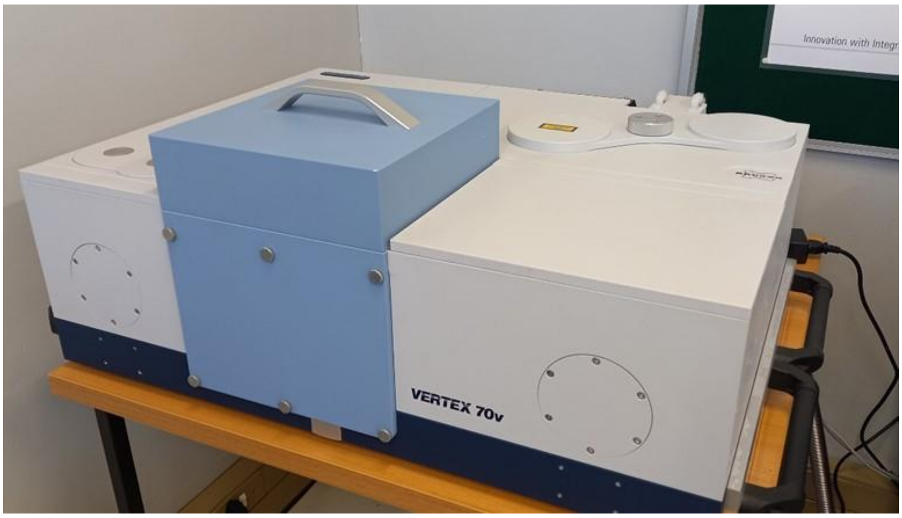

Figure 3.10: Bruker Vertex 70V vacuum FT-IR spectrometer

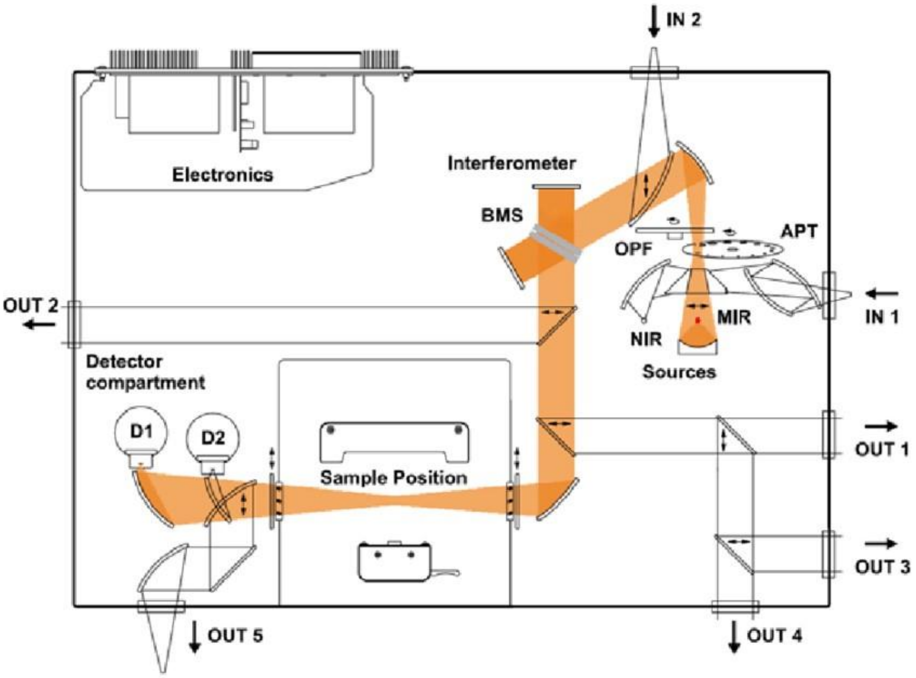

Figure 3.11: Schematic top diagram of VERTEX 70 FTIR Spectrometer (Panah, 2017.)

3.3.5 Scanning Electron Micrographs of Fibres

Both untreated and treated fibres were imaged using the JEOL JSM-7100 Field Emission Electron Microscope to investigate the differences in their surfaces. The effect of alkali treatment as NaOH is increased was studied by observing the morphological changes to the fibre microstructure. The Scanning Electron Microscope (SEM) utilizes electrons, rather than light, to generate highly magnified images. It employs an electron gun to emit a beam of electrons from the microscope's apex. This electron beam traverses a vertical trajectory within the microscope, contained within a vacuum environment. The SEM is connected to the computer interface to allow for easier magnification and observations. The computer setup and the JSM-7100 microscope are shown in Figure 3.12. A thin layer of conductive paste was applied on the specimen stub before the fibres were placed to secure it. A single extracted fibre was placed on an individual stub. The fibres were then chromium coated using a Q150T Turbo-Pumped Sputter Coater/Carbon coater before they were placed in the microscope for analysis. The fibres were observed under the microscope at magnification of x40 to x600. During observations, the appearance of an amorphous surface on the fibre microstructure was assessed in raw and alkali treated fibres. Photographs from this investigation are shown in the subsequent chapter along with the analysis.

Figure 3.12: JEOL JSM-7100 Field Emission Electron Microscope setup

3.4 Experimental Evaluation of Mokolwane Fibre Reinforced Composites

3.4.1 Density

The density of the fibre reinforced composites was determined to evaluate the discrepancies in material properties as the fibre content and alkali treatment were varied. The composites were cut to dimensions of 20 x 20 mm shown in Figure 3.13. To calculate the densities of the composites, the mass of each specimen was found using the mass balance. The volumes were calculated after measuring the lengths, widths and heights of the specimens using a vernier callipers. For each configuration, 10 replicates were measured, and the density of each specimen was calculated by the Equation 3.2 shown below. The average density was calculated from the 10 densities. For this work, the density variation because of alkali treatment was not assessed because the changes were observed to be only marginal.

$$\rho = \frac{m}{V}$$

Equation 3.2

Figure 3.13: Composite density specimen

3.4.2 Moisture Absorption of Composites

Moisture absorption of the composites was evaluated to find the total percentage of water absorbed when the samples were well saturated after undergoing long-term immersion. Given natural fibre-based composites' inherent affinity to absorb moisture, it is crucial to conduct a study on their water absorption characteristics to assess their suitability for industrial applications. This is an important parameter because moisture absorption has been reported to be detrimental to the matrix-fibre bonding and the mechanical performance of the resulting composites. It is therefore imperative to avoid usage of fibres that are prone to moisture uptake when designing and fabricating fibre reinforced composites for industrial purposes. Alkali treatment of the fibres prior to composite fabrication is employed to reduce their hydrophilicity.

The fibre percentage in a composite is another factor that influences the water uptake, and together with the alkali treatment are both evaluated in this moisture absorption test.

The composite specimens were cut to dimensions of 20 x 20 mm in preparation for the water uptake test, performed in accordance with ASTM D570-98. Ten specimens were prepared for each of the 13 configurations. Prior to the test, the samples were oven-dried at 80°C to remove moisture absorbed from the atmosphere, and then cooled in a desiccator. The initial weight of the samples was measured using a weighing machine accurate to 0.0001g. Subsequently, the samples were immersed in distilled water at 23°C for 24 hours and then dried with a lint-free cloth. Afterward, the samples were weighed, and the weight recorded before they were reinserted into the distilled water. This process was repeated at 24-hour intervals until an equilibrium state of water content in the composite was assumed to be reached. The percentage moisture absorption of the specimens was then evaluated by Equation 3.3.

$$W_A\% = \left(\frac{W_1 - W_0}{W_0}\right) * 100$$

Equation 3.3

Where $W_A\%$ is the %moisture absorption, W_0 is the initial weight of the specimen and W_1 is the final weight of the specimen.

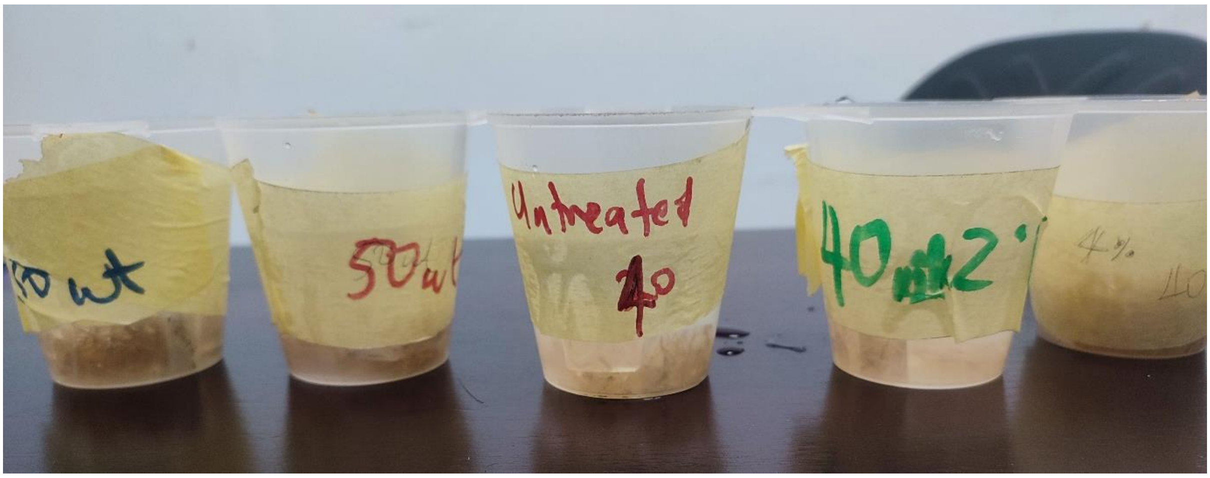

Figure 3.14: Moisture absorption and thickness swelling setup.

3.4.3 Thickness Swelling of Composites.

The thickness swelling investigation evaluates how the composites would perform when exposed to moisture. Excessive thickness swelling of composites during their operational lifespan can severely compromise the mechanical performance of parts fabricated from these materials. Components manufactured for marine applications would be exposed to moisture, hence their dimensional stability is of paramount importance. The composite specimens in the dimensions of 20 x 20 mm were prepared for the thickness swelling test of the composites, which was performed in accordance with the ASTM 570-98 standard. 10 replicates of samples of each composite configuration were prepared for this test.

The initial thickness of the samples was measured with the use of a digital vernier calliper before they were immersed in distilled water. The thickness was then measured at 24 h intervals. The thickness swelling of the composite samples was calculated by Equation 3.4.

$$T_s\% = \left(\frac{T_1 - T_0}{T_0}\right) * 100$$

Equation 3.4

Where $T_s\%$ is the percentage thickness, T_0 is the initial thickness of the samples, and T_1 is the thickness after soaking. The thickness swelling vs immersion duration graph was plotted on ORIGIN software incorporating the results.

3.4.4 Tensile Test

The Tinius Olsen H50KT Universal Testing Machine (UTM) that was used to evaluate the tensile strength and modulus of the Mokolwane fibre reinforced polyester composites. The UTM subjects the test specimen to a controlled tensile force and measures the maximum amount of force the specimen can withstand before breaking. Materials with good tensile strength offer good performance, reliability, and functionality in structural applications.

This test is done to evaluate the influence of alkali treatment and fibre loading on the structural integrity and durability of the composites. The test specimens of this study were cut to sizes of 150 x 25 x 5 mm in accordance with the ASTM standard D3039 to prepare them for the tensile tests. The prepared samples and the UTM are shown in the Figure 3.15. The UTM was run at a crosshead speed of 2mm per minute, with the sample gauge length at 80mm.

The tensile strength and modulus of each specimen were recorded. Five samples of each configuration were tested, and the mean values calculated to ascertain reliability and accuracy of the experiment results.

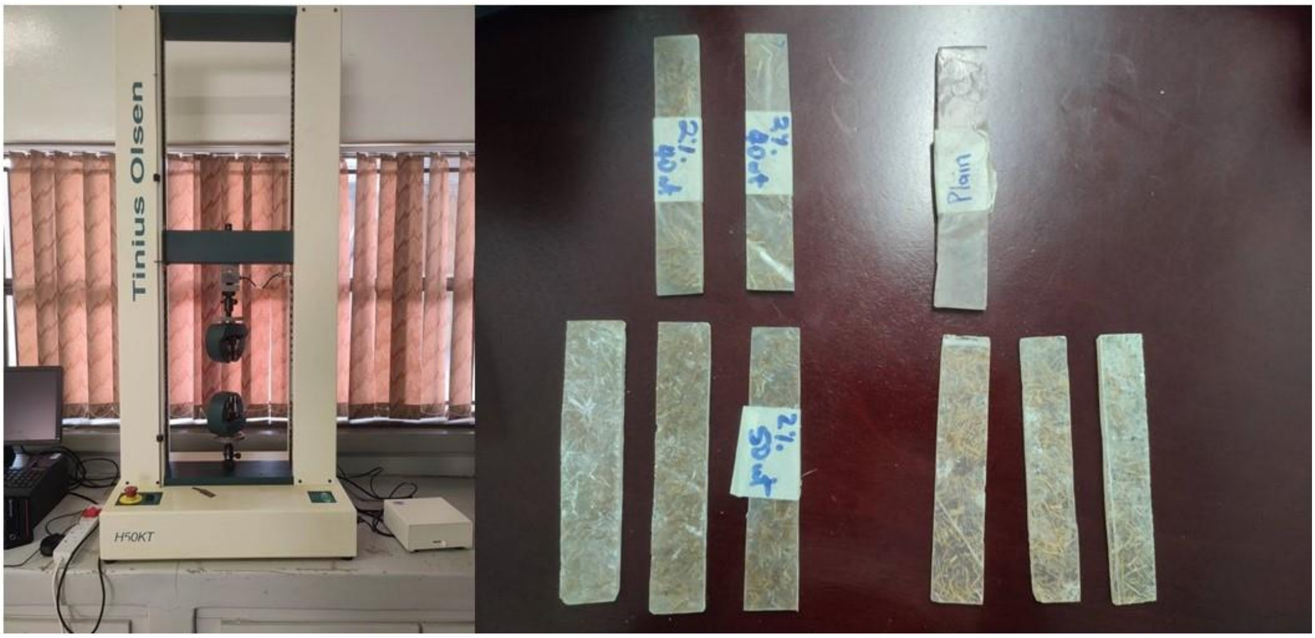

Figure 3.15: (a) Tinius Olsen H50KT Universal Testing Machine (b) Tensile test samples

3.4.5 Flexural Test

The 3-point bending test was performed on the H50KT Universal Testing Machine (UTM) to evaluate the flexural strength and modulus of the fibre reinforced composite and the influence of fibre treatment and fibre loading. For each configuration, 3 samples were tested, and the average values were calculated to ascertain the repeatability of the experimental procedure. The flexural test specimens were cut in accordance with the ASTM standard D790 with length 125mm, width 13mm and thickness 5 mm using the rock cutting machine at the University Geology laboratory. The crosshead speed of the UTM was set to 2mm per minute.

The flexural strength and modulus and were calculated using Equations 3.5 and 3.6. The flexural strength of the composites was calculated using Equation 3.6

$$\sigma_{max} = \frac{3P_{max}L}{2bh^2}$$

Equation 3.5

$$E = \frac{L^3}{4bh^3}\left(\frac{P}{\Delta}\right)$$

Equation 3.6

Where σ_{max} is the flexural strength, $P_{max}$is the maximum load, and L, b and h are the specimen span, width, and thickness respectively, E is the flexural modulus of elasticity, and (P/Δ) is the slope of the load-deformation relationship. In addition, the standard deviation of the results was calculated to find the error bars of the results.

3.4.6 Impact Test

Impact test of the fibre reinforced composites measures their ability to withstand sudden loads before fracturing. Determining the impact energy required to cause failure under high-velocity loads serves as quality control measure to ensure that the material would be able to meet design and application requirements. This is done by inducing sudden fracture by a swinging pendulum and measuring the impact energy absorbed.

The impact test specimens were cut to 64 x 13 x 5 mm from composite panels in accordance with the ASTM standard D256. A 2mm, 45° notch was cut in each specimen in accordance with the standard guidelines as shown by the specimen schematic in Figure 3.16(a) shows the impact test specimen schematic and the machine used in this study. Five samples of each composite configuration of varying degrees of fibre loading and alkali treatments were evaluated by this test. The Izod impact tests were conducted at the university materials testing laboratory on the Tinius Olsen Model Impact 503 machine shown on Figure 3.16(b).

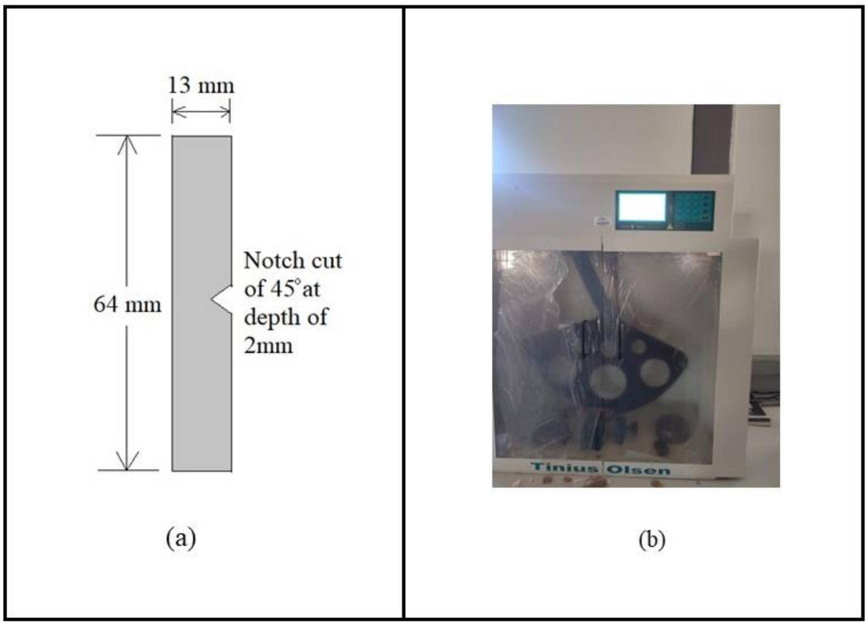

Figure 3.16: (a) Impact test specimen schematic (b) Tinius Olsen Model Impact 503

3.4.7 Scanning Electron Micrographs of Composites

Mokolwane fibre reinforced composited were observed under the JEOL JSM-7100 Field Emission Electron Microscope in the University Geology department to the effect of NaOH treatment and fibre volume fraction on the matrix-fibre bonds. The SEM setup is illustrated in Figure 3.12. A thin layer of conductive paste was applied on the specimen stub before the specimen was placed to secure it. The specimen was then chromium coated using a Q150T Turbo-Pumped Sputter Coater/Carbon coater before they were placed in the microscope for analysis. Due to their high conductivity, coating materials enhance the signal-to-noise ratio during SEM imaging, resulting in higher quality images. After the specimen were placed inside the microscope, they were observed on the computer screen with the use of the equipment knobs and the computer interface to focus and magnify the observations as necessary. The composite samples were observed to check for presence of voids, cavities, and agglomerations at magnifications of x50 to x850 on the SEM.

3.5 Chapter Summary

This chapter outlines the methodological framework employed to fulfil the objectives and aims of this dissertation. It begins with the harvesting, extraction, and preparation of fibres through alkali treatment, followed by characterization testing. These tests encompass fibre diameter distribution, moisture absorption, thermal analysis, FTIR testing, and SEM analysis. Subsequently, both untreated and treated Mokolwane fibres were utilized to manufacture fibre-reinforced polymer composites with polyester, which underwent further testing. The fabricated composites underwent examination for density, moisture absorption, thickness swelling, tensile strength, flexural strength, and impact energy. Additionally, SEM analysis was employed for further analysis. Throughout the chapter, pertinent illustrations, explanations, and equations have been incorporated to provide a comprehensive understanding of the process. The findings of this work are presented in the following chapter.

CHAPTER 4: RESULTS ANALYSIS

4.1 Introduction & Overview

In this chapter, the results obtained from the procedural steps outlined in the preceding chapter are presented. It is divided into two main sections (excluding the introduction and summary); (i) Characterization of Mokolwane fibres and (ii) Experimental evaluation of Mokolwane fibre reinforced polyester composites. The first section concentrates exclusively on delineating the properties of both untreated and alkali treated fibres. The aim of these findings is to discern the distinction between the two, with the goal of comprehending the impact of alkali treatment on the resulting composites.

The next part aims to examine the outcomes related to water absorption, mechanical characteristics, and morphological properties of composites reinforced with Mokolwane fibre. This section delves into the impact of both fibre content and alkali treatment on the composites. The discussion in this segment involves a comparison of the outcomes from this novel fibre-reinforced composites with those obtained by other researchers in the field of natural fibre reinforced polymer composites. Additionally, an exploration of the underlying mechanisms influencing the performance of various composite configurations is undertaken.

4.2 Characterization of Mokolwane Fibre

4.2.1 Fibre Diameter Distribution

Characteristics like chemical composition, flexibility, tensile properties etc of fibres vary significantly among different species. Furthermore, these properties also vary in the same fibre specie as conditions such as their growing and extraction influence these variations. Consequently, the resulting composites exhibit notable diversity in mechanical, thermal and water absorption properties, highlighting the significant role played by these variations (Moigne et al., 2011). This study carried out a survey of the fibre diameters of this extracted novel fibre in the aim to characterize the fibre properties.

The results showing variation of mokolwane fibre diameter are shown in Figure 4.1. The results show that majority of fibres extracted for this work are in the range 450-500 µm. The median fibre diameter was found to be 465 µm. In a work by Garat et al. (2018) to analyse the morphometric variations in natural fibres by automated laser scanning, median diameters of Palm, Sisal, Hemp, Flax and nettle were found to be 145,163. 88, 91 and 90 µm respectively. Literature has reported that wide variability in cross sectional dimensions is the result of botanical origins of the fibres as well as the scale of measurement performed (elementary fibre cells or fibre bundles) (Yan et al., 2014). During extraction, mokolwane fibres were highly compact in the leaflets likely resulting in fibres that have larger cross-sectional shapes unlike other contemporary natural fibres. It is important to note that the term *fibre* refers to a bundle of fibrils. These fibre diameters are also influenced by the extent of extraction from the leaflets as well as the ability of water retting to loosen the fibre bundles. Through analysis of the cross-sectional profile of Jute, Kenaf, Curaua, and Flax, Kandemir et al., (2020) observed a range of shapes, spanning from rectangular to circular and elliptical. The common way to characterize fibre diameter involves taking a couple of measurements along the fibre length and calculating the average. The outcomes are likely to be biased depending on the predominant orientation of the fibres observed under the optical microscope.

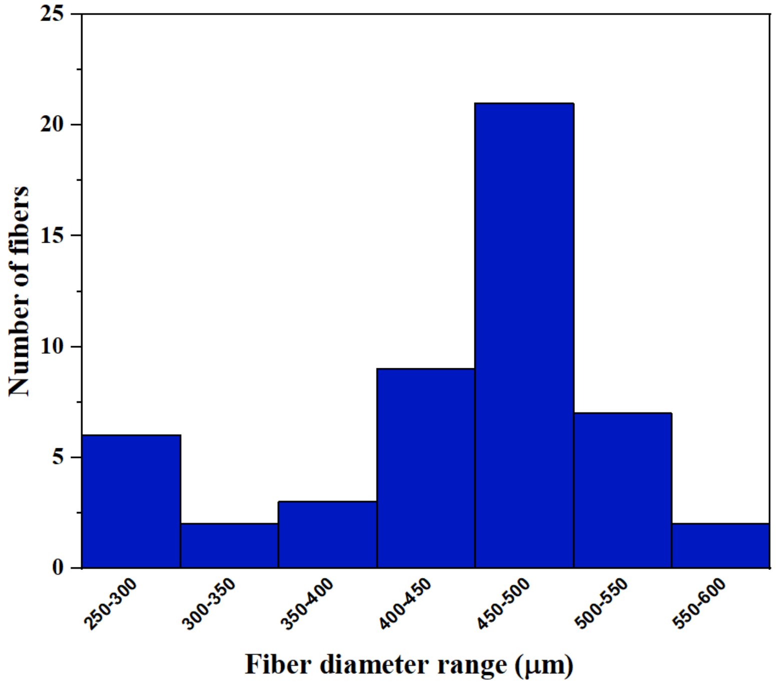

Figure 4.1: Mokolwane fibre diameter distribution

4.2.2 Moisture Absorption of Fibres

The results from the moisture absorption test of Mokolwane fibres are depicted in Figure 4.2. The raw fibres were found to have the highest moisture uptake from the tested specimens. Water, being a highly polar compound, readily attracts the polar -OH groups present in all cellulosic fibres. The impact of alkali treatment on the moisture absorption tendency of Mokolwane fibres become apparent through the observed reduction in moisture absorption by 33%, 46%, and 52% after the fibres have undergone treatment by 2%NaOH, 4%NaOH, and 6%NaOH respectively. Significant portions of natural fibres consist of cellulose and hemicellulose, which contribute to the inherent hydrophilic nature of the fibres. The results of the test can be assumed to be an outcome of hemicellulose removal from the fibres by NaOH. These results are corroborated by those of Begum et al., (2021), who was able to reduce Areca and Banana fibres' water uptake by treating them with alkali. Ramadevi et al., (2012) was able to obtain the lowest moisture absorption of Abaca fibres by treating them with 20% NaOH. The employment of such elevated concentrations of alkali, however, is deemed detrimental to the overall properties of the fibres, thereby compromising their capacity to reinforce polymer matrices.

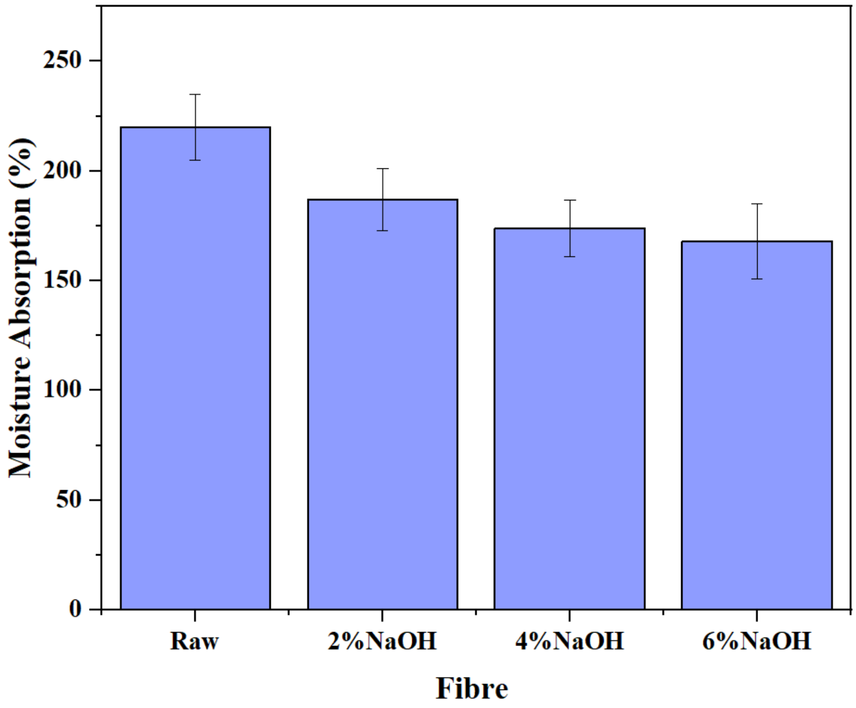

Figure 4.2: Moisture absorption of Mokolwane Fibres

Table 4.1: Moisture absorption of Mokolwane Fibres

	Moisture Absorption
Raw fibres	220%
2%NaOH treated fibres	187%
4%NaOH treated fibres	174%
6%NaOH treated fibres	168%

4.2.3 Thermogravimetric Analysis of Fibres

As described in the previous chapter, thermogravimetric analysis enables the tracking of changes in the mass of a sample with respect to temperature, providing valuable insights into the decomposition parameters of the fibre. Results from TGA have practical significance in evaluating the temperature conditions necessary when fabrication mokolwane fibre reinforced composites. To facilitate reading, it is convenient to depict the TGA curve by illustrating the percentage mass loss against temperature in °C. For this work, a comparative thermogravimetric analysis of untreated and alkali treated Mokolwane fibres was carried out in a temperature range of 30 °C - 700°C. The results are a necessary component to study the degradation properties of the fibres and the ensuring that the resulting composite is thermally stable in its applications. The parameters contributing to thermal stability include the initial degradation temperature, the major degradation temperature, and the final degradation temperature. Low degradation temperatures of natural fibres are detrimental to their reinforcing effects, resulting in weak composites. Because of this, the effect of alkali treatment on the thermal performance of Mokolwane fibres is investigated.

The degradation of the fibres shown in Figure 4.3 occurred in stages for the fibres. The degradation peak at 105°C to 112°C corresponds to vaporization of existing moisture in the fibres. The mass loss of 8%-10% occurs during this stage. In addition to this observed weight loss being associated to moisture content vaporization, fibre volatile extractives like wax contents on Mokolwane fibre surface are also evaporated (Oladele et al., 2019; Selvaraj et al., 2023).The second peak of degradation which occurs at 255°C to 280°C is ascribed to the pyrolysis of lignin from the fibres and lesser degradation of hemicelluloses (Izwan et al., 2020). This showed a mass loss of 11.4% for the untreated fibre specimen. Treated Mokolwane fibres exhibited lower mass loss during the stage at 4% to 6%.

Majority of weight loss was recorded during the third stage of degradation, which is ascribed to cellulose degradation (Bessa et al., 2021). In this stage depolymerization of hemicelluloses and complete breakdown of glycosidic links in α-cellulose occurs. The onset of this stage is at 258℃ to 280℃ and mass loss of 57.7%, 50%, 48% and 46% is observed for untreated, 2%NaOH-treated, 4%NaOH-treated, and 6%NaOH-treated fibres respectively. It can be seen from the TG curve that no significant weight drop is observed on the fibres till 258℃, specifying that the thermal stability is approximately around that temperature. Similar thermal stabilities have been observed in various studies of natural fibres like *Cissus quadrangularis* stem, *Furcraea foetida* and *Stretzlitzia reginae* fibres (Indran & Raj, 2015; Lemita et al., 2022). The effect of alkali treatment on the fibres to alter the thermal stability was observed to be marginal but positive. The result from the thermogravimetric analysis still signifies that the low thermal stability of Mokolwane fibres limits it from being coupled with high temperature processing polymeric matrices. For comparison, synthetic precursors, like glass fibres, soften between 700 and 1000℃(Y et al., 2019).

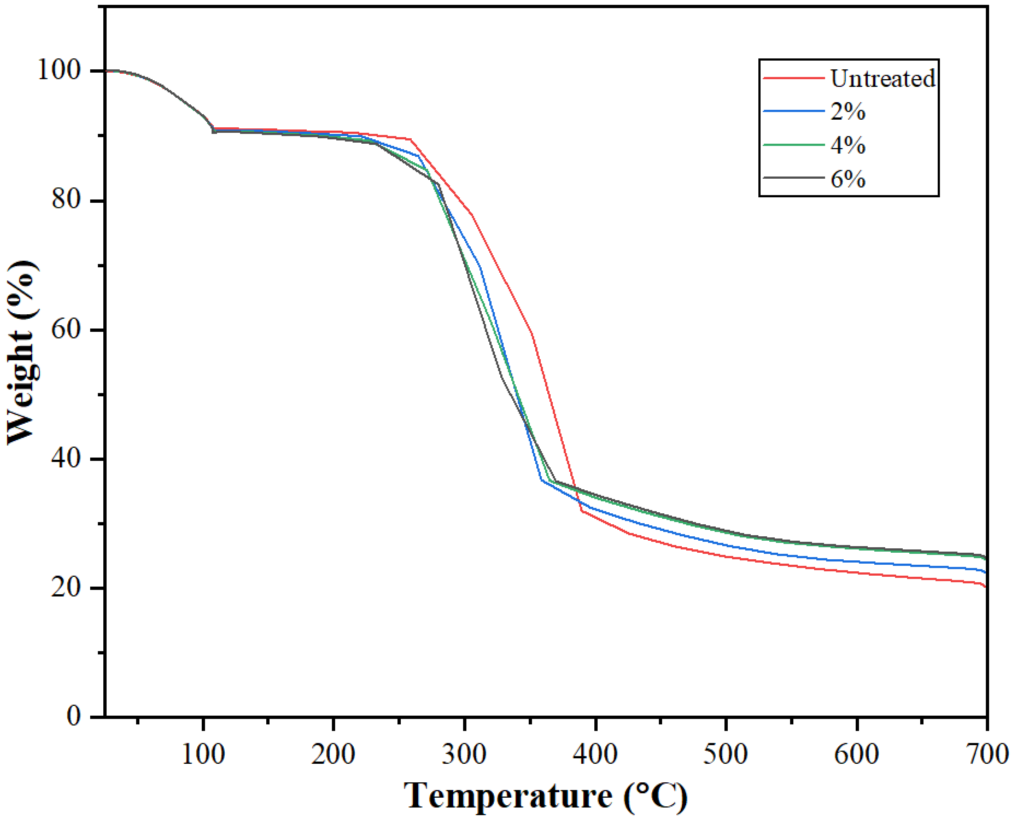

Figure 4.3: TGA curve of Mokolwane Fibre

Table 4.2: Information from TGA results

Materials	T at 10% of weight loss	T at 50% of Weight Loss	Residue (%) at 700
Untreated	235.7	364.7	19.61
2%NaOH	215.5	340.0	21.96
4%NaOH	190.5	340.7	23.89
6%NaOH	179.4	335.2	24.21

4.2.4 Fourier Transform Infrared Spectroscopy

FTIR Spectroscopy was employed to discern the chemical constituents of both the untreated and treated Mokolwane fibres. It was used to confirm the changes after the fibres were treated. Figure 4.4 illustrates the FTIR spectra of both the untreated control specimen and the treated samples, providing useful insights into the functional groups and molecular bond structure within the 4000 cm^{-1} to 1000 cm^{-1} range. The treated Mokolwane fibres exhibited similar spectra but the differences in absorption bands intensities is observed in alkali treated specimens.

It can be gleaned from the graph that the typical cellulose peaks are altered in the fibres after undergoing alkali treatment. The U-shaped broad absorption band at 3500 cm^{-1} to 3300 cm^{-1} for both treated and untreated fibre is associated with the vibrational stretching of hydroxyl (O-H) groups that are a component of cellulose (Amroune et al., 2015). The peak intensity of the absorption band in the FTIR spectra was increased after the fibres underwent alkali treatment. Cao et al., (2012) posits that the breaking of the hydrogen bonds between the O-H groups of cellulose and hemicellulose is the likely explanation.

The absorption bands at around 1740 and 1240 cm^{-1} are associated with carbonyl double bond(C=O) stretching and C-O functionalities of acetyl groups that make up hemicellulose (Sreekumar et al., 2008). The spectra exhibited the disappearance of these bands with an increase in alkali treatment on the fibres. Their disappearance denotes that alkali treatment significantly decreases their presence on the fibres. The band at 1625 cm^{-1} is associated with the bending mode of absorbed water, and the one at 1593 cm^{-1} is assigned to the aromatic skeletal vibrations coupled with C-H in-plane deformations (Reddy et al., 2013). The peak at around 2920 cm^{-1} is associated with stretching vibrations of the aliphatic chains of the C – H functional group in polysaccharides (Bessa et al., 2021; Hospodarova et al., 2018). However, no appreciable differences have been noted for the following absorption peaks: 2920, 1593, and 2920 cm^{-1} peaks.

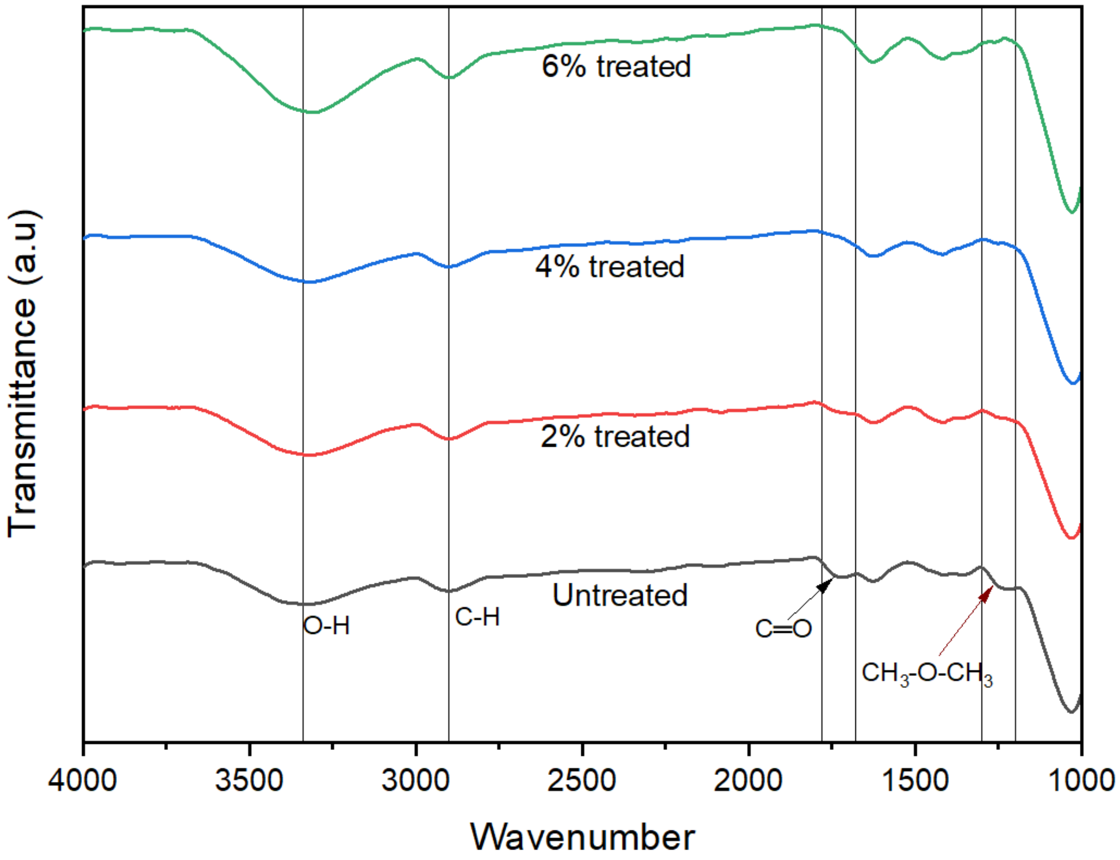

Figure 4.4: FTIR Spectra of Mokolwane fibres

4.2.5 Morphology of Fibres

The fibre samples were evaluated under the Scanning Electron Microscope (SEM) to investigate their morphology as well as the influence of alkali treatment on this property. Figure 4.5 presents the images taken by SEM of the fibre samples. Untreated sample shows closely packed microfibrils in the fibre sample with a presence of amorphous layer on the fibre surface. The amorphous layer on a lignocellulosic fibre includes macromolecular components such as hemicellulose, lignin, pectin, and wax. This layer hinders the fibre from efficiently bonding to the polymeric matrix due to its hydrophilic nature. The SEM micrographs further show the removal of this amorphous layer after the fibre samples had undergone alkali treatment. Mercerization is effective in breaking down the lignocellulosic complex, and dissolving lignin and hemicellulose, hence exposing the microfibrils contained within. Since hemicellulose is a microfibril cementing constituent, the microfibrils are exposed by the alkali treatment. Exposing microfibrils increases the effective surface area that bonds with alkali therefore increasing the wettability of the fibres. Smoother surface and increased surface area of Mokolwane fibres in figure 4.4 are a proof of the effectiveness of alkali treatment to separate lignocellulosic fibre as also found by Asim et al. (2016) on Kenaf and Pineapple leaf fibres.

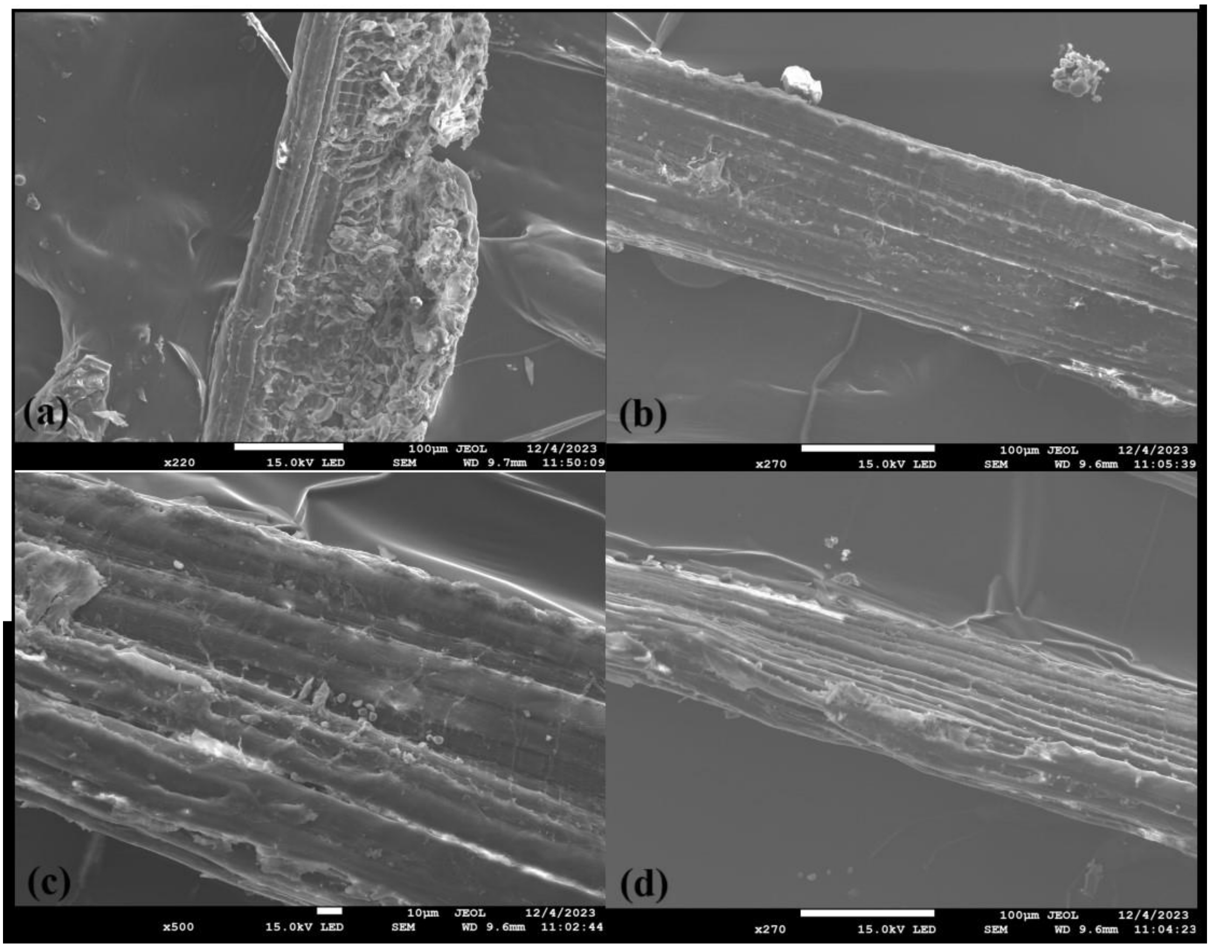

Figure 4.5: SEM images of Fibres (a) Untreated (b) 2%NaOH treated fibre (c) 4%NaOH treated fibre (d) 6%NaOH treated fibre.

Table 4.3: Properties comparison of Mokolwane fibre with other natural fibres.

Fibre	Diameter (μm)	Density (g/cm^3)	Moisture absorption	Thermal Stability (°C)	References
Mokolwane	250-600	0.96	220%	258	This work
Sisal	120	1.45	76.7%	-	Sabarish, (2007)
Ziziphus Mauritania	570.2	1.132	-	280	Vinod et al., (2022)
Coir	-	1.438		278.8	(Madueke et al., (2021)
Oil palm	150-500	0.7-1.55	120% (24 hours)	-	Rama Rao & Ramakrishna, (2022)
Bamboo	161-584	0.49	107.65%	-	Buson et al., (2018); Mousavi et al., (2022)
Kenaf	81	1.4	-	-	Tholibon et al., (2016)

4.3 Experimental Evaluation of Mokolwane Fibre Reinforced Composites

4.3.1 Density of Composites

The mechanical properties of natural fibres are affected by intricate interplays among various external factors and inherent structural features spanning molecular, macromolecular, and microscopic levels. Density and other composite physical properties are dependent on factors like void presence, humidity, fibre loading and resin viscosity (Mahdavi et al., 2010; Radzi et al., 2019). Furthermore, the physical and mechanical properties of the developed composites are significantly impacted by manufacturing process parameters such as temperature and mixing duration, leading to the formation of high voids due to air entrapment, consequently affecting the composite density (Awad et al., 2023).

The measured density of the composites is presented in the Table 4.3 below. In addition, the density of Mokolwane fibre was found to be 0.96 g/cm^3.The method chosen to find the composite density likely has an influence on the density results. A common way to calculate composite density is the displacement method because it accounts for void presence. Other factors include presence of debris on the fibre surface, the fibre specie, and its maturity level (Madueke et al., 2021). Additionally, calculating density is likely to be influenced by the cut finish when samples were cut from the moulds. The difference between calculated densities of treated and untreated composites were found to be very marginal in this work. Notable differences were however observed when fibre volume fraction was varied on the composites. The density of plain polyester was to be 1.22 g/cm^3. The influence of how composite density varies with fibre volume fraction can be deduced from the table shown. 1.15, 1.10 and 1.07 g/cm^3 were found for 40 wt.%, 50 wt.%, and 60 wt.% composites respectively. The increase in fibre loading is reported to correlate with the void fraction in composites (Tripathy et al., 2016; Zainudin et al., 2020).

Table 4.4: Density of Mokolwane fibre reinforced composites

Specimen	Density (g/cm^3)
Mokolwane Fibre	0.96
Plain Polyester	1.22
40 wt.% composite	1.15
50 wt.% composite	1.10
60 wt.% composite	1.07

4.3.2 Moisture Absorption of Composites

Using the results collected from the moisture absorption test of the test specimen, plots showing the moisture absorption versus the immersion time were created and presented in Figure 4.6. It is observed that there was an initial rapid water receptivity by the composites during the first day of immersion. All the composite configurations showed the rate of water receptivity to decline with longer soaking time, eventually reaching an equilibrium of state at around 480 hours, where water absorption becomes static. The water uptake becomes static as the composites becomes saturated and can no longer be able to absorb any more moisture. The neat composite had the lowest water uptake of 0.74% from the immersed specimens. The homogeneity in the neat specimen led to a more compact structure with less voids.

A higher rate of water uptake for composites with higher fibre composition can be gleaned from the figure. The composites with 60 wt.% fibre loading ended up with higher water uptake than the 40 wt.% and 50 wt.% composites signifying correlation between fibre loading and propensity to absorb water. The percentage water gain for the 60 wt.% composites is at 3.95-4.40% which drops down to 1.90-2.65% and 1.19-1.52% for composites with 50 wt.% and 40 wt.% fibre content respectively. From this, it can be deduced that higher fibre content increased water uptake capacity of the composites. Higher fibre content increased the cellulose content in the composites, hence led to more free OH^- group. The OH^- group that are present in the cellulose form hydrogen bonds with moisture particles. This is consistent with findings from Bera et al., (2019) who found that triple layers of luffa fibre to reinforce epoxy composite absorbed more moisture than single and double layered composites. In addition to the increase of free OH groups, an increase in the fibre fraction results in an increase in presence of voids because of insufficient wetting of the fibres by the resin. Moisture absorption of natural fibre polymer composites is an important parameter to keep track of as it leads to decline in composites properties. Water transport kinetics in composites has been described to rely on several factors, including polymer matrix viscosity, fibre morphology, temperature, humidity, presence of voids, defects, and microcracks along the composite interface (Lazrak & Hammi, 2022; Radzi et al., 2019). The primary mechanism through which water uptake occurs in fibre reinforced composites is via micro gaps that exist between the polymer chains. The second process is the capillarity transport through gaps, voids and micro cracks present in the fibre-matrix interface because of poor fibre wettability. The third mechanism is through composite swelling that leads to crack propagation (Manaila et al., 2021; Santos et al., 2019).

The influence of mercerization on the water uptake of the composites can also be deduced from the graph. The results show that fibre treatment reduced the water uptake of the composites. Increasing the alkali concentration in the treatment enhanced the water repellence of the composites although at 60% fibre, this pattern was not seen. In general, alkali treated fibre reinforced composites absorbed far less moisture than untreated fibre reinforced composites. The relative differences in moisture absorption between alkali treated fibre reinforced composites and untreated fibre reinforced composites can be accounted for by looking at the effect of alkali treatment on the fibre surface. Alkali treatment removes the amorphous layer present on the fibre surface that is responsible for the fibre's hydrophilicity. The layer contains cellulose which comprises of OH^- that reacts with water molecules. Consequently, the reduction of hydrophilic OH- groups enhance the fibre's resistance to moisture. Among 40% fibre loading composite specimens, 4%NaOH treatment was able to effectively reduce water absorption from 1.46% in untreated samples to 1.19%.

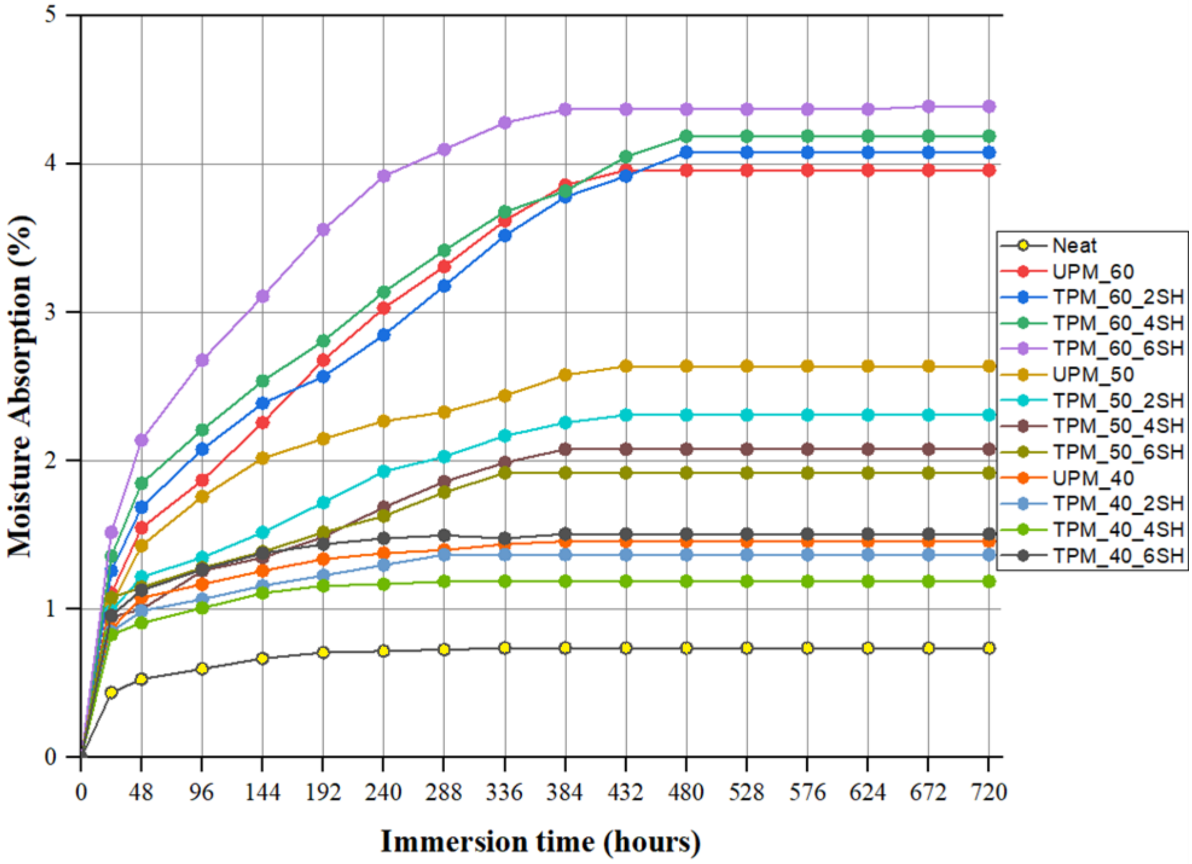

Figure 4.6: Moisture Absorption of Mokolwane fibre reinforced polyester composites.

Table 4.5: Moisture absorption of composites after long-term immersion

Sample	Moisture Absorption (%)
Neat	0.74
UPM_40	1.46
UPM_50	2.64
UPM_60	3.96
TPM_40_2SH	1.37
TPM_50_2SH	2.31

TPM_60_2SH	4.08
TPM_40_4SH	1.19
TPM_50_4SH	2.08
TPM_60_4SH	4.19
TPM_40_6SH	1.51
TPM_50_6SH	1.92
TPM_60_6SH	4.39

4.3.3 Thickness Swelling of Composites.

The dimensional stability of the composites was assessed over a period of 720 hours with the thickness swelling test, providing valuable insights for determining their suitability for various end uses. The results showing the percentage of the swelling ratio of mokolwane fibre reinforced composites due to water uptake with respect to immersion duration are presented in Figure 4.7. The water absorption of the composites occurs through the capillaries resulting in swelling of the samples. The neat composites were used as control for the experiment. The results show that they were more resistant to water absorption hence they swelled less. The homogenous hydrophobic resin does not absorb moisture particles because it is more compact and has less voids than the heterogenous fibre reinforced composites. It has been reported that the capacity of composites to absorb moisture and thickness swelling is linked with the density, void presence and fibre-matrix interfacial bond (Nunna et al., 2012). The findings from this test showed a similar trend to the moisture absorption test, whereby the initial moisture diffusion into the composites led to a faster swelling rate before stability is reached. Stability is achieved once the composites attain saturation, effectively countervailing the diffusion of water molecules into them. The highest thickness swelling was observed for UPM_60 at 4.01% followed by TPM_60_2SH and TPM_60_4SH at 3.53% and 3.29% respectively. The findings indicate that Mokolwane fibre-reinforced polyester composites, particularly those with a higher content, exhibited increased susceptibility to swelling due to moisture absorption. The heightened propensity for swelling can be attributed to several factors, including the heightened presence of free OH- groups, pores/voids within the material, and suboptimal fibre distribution and fibre-matrix interface (Kaymakci et al., 2017). These elements collectively contribute to a decrease in dimensional stability and an increase in thickness swelling within the composites. Masoodi and Pillai, (2012) documented that the presence of micro-crack on the composite surface is initiated by internal pressure within the polymer matrix caused by the swelling of hydrophilic natural fibres. In the study by Kazi et al., (2022), woven Roselle fibre epoxy composites were found to retard the capillary action of moisture absorption and thickness swelling than plain composites.

The impact of alkali treatment on the thickness swelling of composites is notable. Specifically, composites featuring alkali-treated fibres exhibit reduced swelling compared to those with untreated fibres. This phenomenon can be attributed a decrease in cellulose content within the fibre composites. Cellulose, being composed of OH- groups, forms hydrogen bonds with water molecules, and the reduction in cellulose content through alkali treatment consequently mitigates the swelling effect. The best moisture absorption and thickness swelling performance was found to be TPM_40_4SH at 1.19% and 1.78% respectively. It can therefore be deduced that this is the optimum fibre loading and alkali treatment to promote good interfacial adhesion, with minimal void presence and sufficient fibre wetting.

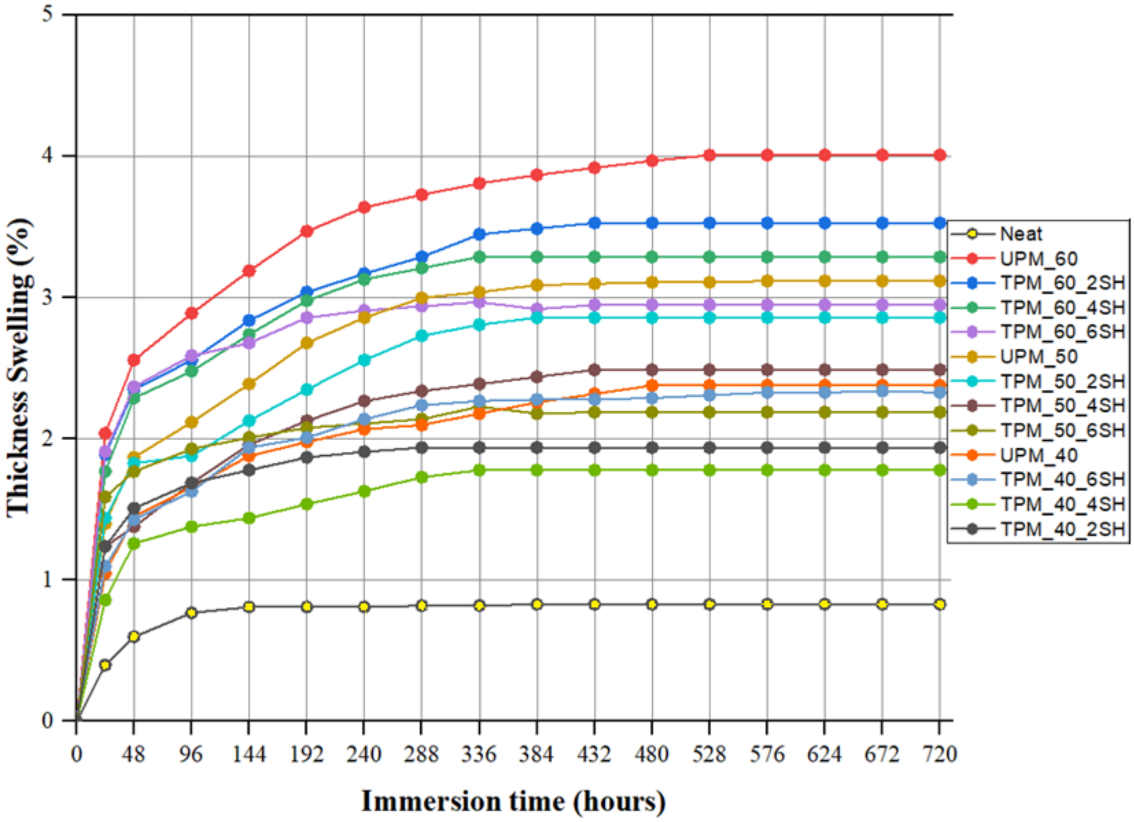

Figure 4.7: Thickness swelling of Mokolwane fibre reinforced composites.

Table 4.6: Thickness swelling of composites after long-term immersion.

Sample	Thickness swelling (%)
Neat	0.83
UPM_40	2.38
UPM_50	3.12
UPM_60	4.01
TPM_40_2SH	1.94
TPM_50_2SH	2.86
TPM_60_2SH	3.53
TPM_40_4SH	1.78
TPM_50_4SH	2.49
TPM_60_4SH	3.29
TPM_40_6SH	2.33
TPM_50_6SH	2.19
TPM_60_6SH	2.95

4.3.4 Tensile Strength of Composites

Uniaxial tension was performed on the samples to assess how much tensile stress the composites can endure before breaking. The results showing the effect of fibre loading (40 wt.%, 50 wt.% and 60 wt.%%) and alkali treatment (2,4 and 6%NaoH) on the tensile properties of Mokolwane fibre reinforced polyester composite are presented in Figure 4.8. The investigation showed that at 40 wt.% of fibre loading, low tensile strength and modulus were experienced. Poor dispersion and fibre distribution is a possible reason for this, as the resin is unable to efficiently transmit the applied stress to the fibres. Tensile properties were enhanced by increasing the fibre loading to 50 wt.%, implying that the applied stress was being efficiently applied to the fibres by the polyester resin. The addition of fibre beyond 50wt% fibre loading resulted in a decline in the tensile properties of the composites. This poor fibre-matrix compatibility is attributed to the insufficient wetting of the fibres by the polyester resin, hence poor bonding between the two and loss in tensile properties. Additionally, higher fibre fraction results in agglomeration and fibre-fibre interactions of the discontinuous mokolwane fibres (Ismail & Ishak, 2018).

The results show that surface treatment improved the tensile properties of the composites. The findings were able to show that tensile properties initially increased and then decreased with an increase of concentration of alkali treatment undertaken. The most optimum treatment can be observed to be 4%NaOH. The tensile strength and modulus of the composites were improved by 8.90-13.0% and 7.73-11.1% after they were treated by 4%NaOH. Significant enhancement of tensile properties of alkaline-treated is a result of removal of impurities such as pectin, fats, and lignin present in the fibre surface (Narayana & Rao, 2023; Yan et al., 2012). This optimizes the interfacial adhesion of the fibres to the matrix allowing them to withstand tensile loads. Increasing alkali treatment beyond 4%NaOH is observed to reduce the tensile properties of the composites. This can be attributed to fibre damage by excessive alkali treatment. The effect of alkali treatment on Mokolwane fibres has been shown in section 4.2.5 of this thesis. The micrographs show that with increasing alkali concentration, there is a progressive increase in fibre fibrillation. However, with very high alkali concentration degradation of cellulose occurs (Raharjo et al., 2023). This has been corroborated by Das & Chakraborty, (2008) with their study on Bamboo fibres.

Other researchers have found higher tensile strengths by incorporating hybrid fibre reinforced composites. Sathees Kumar, (2020) investigated effect of fibre loading on sisal, jute and sorghum hybrid polyester reinforced composites and found tensile strengths ranging at 174 - 245 MPa. Higher tensile properties were also found in the investigations on unidirectional fibre reinforced composites. Ren et al., (2019) found tensile strengths and modulus ranging at 220 - 260 MPa and 21-27 GPa respectively when investigating the effect of alkali treatment on mechanical properties of Kenaf fibre reinforced epoxy unidirectional composites.

A key observation from this test is that whilst TPM_50_4SH was observed to have the best performance in tensile performance, prior experimentation findings showed this specimen to absorb more moisture—and thus be more susceptible to swelling—than a specimen with the same alkali treatment concentration but lower fibre loading. A clinical investigation on the mechanical properties of specimens that have been immersed in moisture is required to investigate this phenomenon.

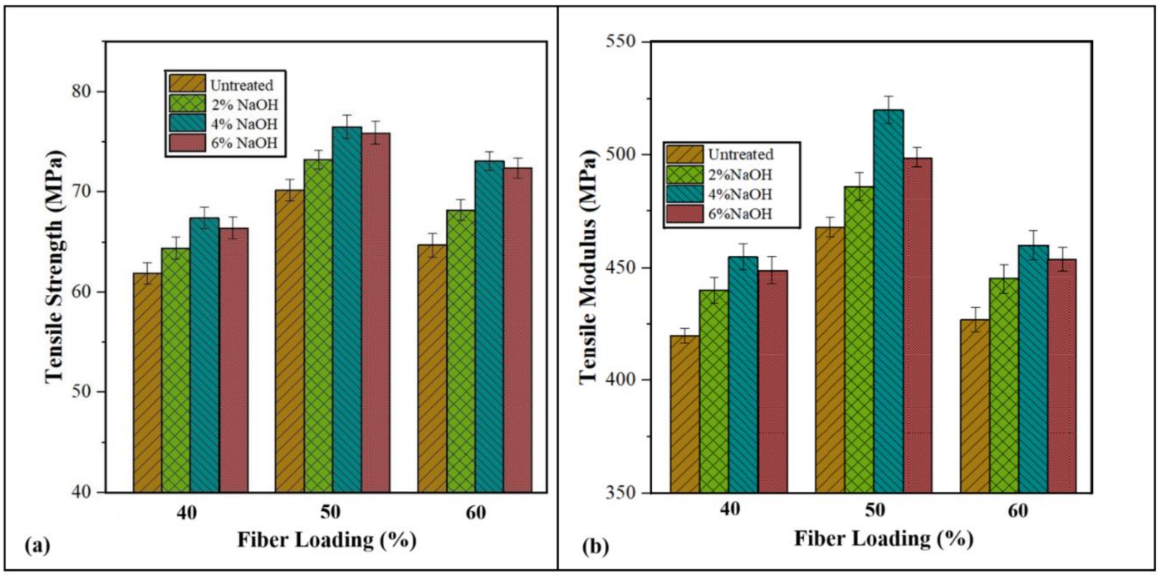

Figure 4.8: Tensile (a) Strength and (b) Modulus of Mokolwane fibre reinforced polyester composites.

4.3.5 Flexural Strength of Composites

The results describing the effect of NaOH treatment and influence of fibre loading on flexural strength and modulus of Mokolwane fibre reinforced composites are presented in Figure 4.9. Flexural strength is a crucial information in material selection to ensure that the composites are employed efficiently to withstand bending loads. The specimens were observed to fail by crack initiation and propagation in the bottom surface when they underwent three-point bending. The results illustrated in the figures generally exhibited a similar pattern to the tensile properties of the composites.

The flexural strength is notably influenced by the proportion of fibre content when incorporated with a polymer matrix. An improvement of flexural properties is exhibited by the composites when the fibre weight percentage is increased. The maximum flexural strength and flexural modulus of the composites are 124 MPa and 2.67 GPa obtained at fibre loading of 50 wt.% and alkali treatment of 4% NaOH. At 50 wt.% fibre loading the fibres are properly dispersed and oriented within the matrix to withstand the bending stresses incurred during the flexure test. Beyond 50 wt.% fibre loading a decrement is observed in the flexure properties. This can be attributed to insufficient fibre wetting by the polyester matrix, leading to voids and formation of agglomerations within the composite. An increase in fibre loading from 40 wt.% to 50 wt.% shows appreciable improvement in flexural strength of fibre reinforced composites.

SaravanaKumar et al. (2021) noted that addition of Kenaf and Pineapple leaf fibres to polyester enhanced flexural strength and inhibited composite deflection during the bending tests. However, excessive fibre composition indicated ineffective matrix-to-fibre load transfer.

Alongside optimal fibre content, optimal fibre treatment results in strong interfacial bonding, allowing for elastic deformation of composites. The results showed a gradual flexural strength improvement with an increase of NaOH concentration. This indicated that specimen treated with 4%NaOH have better holding zones with minimal fibre debonding, separation, or pull-out systems. However, at higher alkali concentration of 6%NaOH, a reduction in the flexural properties of the composites was observed. A pattern of an immediate increase in flexural properties followed by an eventual decrease is shown throughout research, and is consistent with findings from Kamaruddin et al. (2022) and Bekele et al. (2023).

Mahdavi et al. (2010) posited that the composite manufacturing processes like Injection moulding, or improved fibre processing techniques could potentially improve the resulting mechanical properties like flexural strength. Other authors noted the influence of polyester with a coupling agent to further improve flexural strength of composites. Alsewailem and Binkhder, (2014) found that diphenylmethane 4-4’ diidocyanate(DPMI) and ethylene propylene grafted with maleic anhydride(EP-g-MA) were effective in modifying the morphology of Date Pits reinforced polymer composite.

It is worth noting, from tensile and flexural test results, that optimizing fibre loading yields an appreciable improvement in mechanical performance over optimizing alkali treatment. This is consistent with the work from Kommula et al., (2013), and so, it can be inferred that interfacial adhesion is more reliant on optimum fibre loading over alkali treatment, but a synergy exists when the interplay of the two parameters is fine-tuned.

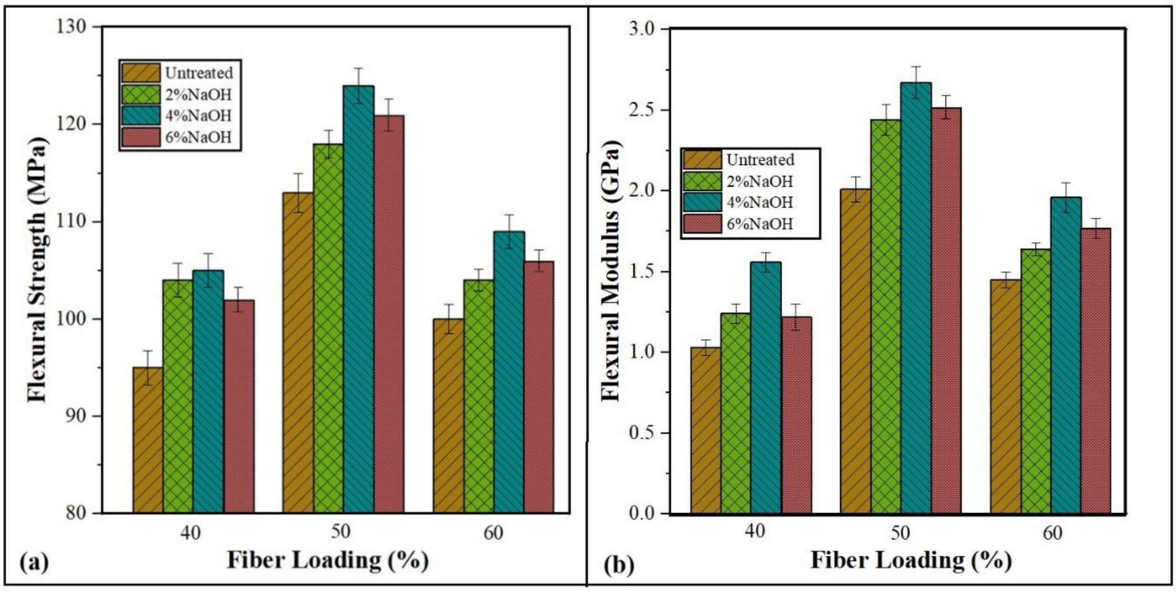

Figure 4.9: Flexural (a) Strength and (b) Modulus of Mokolwane fibre reinforced polyester composites.

4.3.6 Impact Energy of Composites

The impact strength of a composite is defined as the capacity to absorb and dissipate energies when exposed to a shock loading. Composites can be vulnerable to various impacts, such as accidental tool drops, strikes from hailstones, or collisions with low-flying objects in their structural lifespan. During the impact test, specimens with notches, secured in the Universal Testing Machine (UTM), fracture due to the impact of a swinging hammer. By measuring the requisite energy to instantiate a fracture in a specimen, useful information can be deduced on proper application of the material in industrial applications. This section discussed the influence of fibre loading and surface treatment by mercerization on the impact properties of mokolwane fibre reinforced composites as delineated on Figure 4.10.

The addition of Mokolwane fibres up to 50 wt.% from 40 wt.% improved the impact energy by 123%, 119%, 65% and 60.6% for untreated, 2%NaOH, 4%NaOH and 6%NaOH treated composites. This signifies a good bonding and stress distribution when the composites absorb impact energy from sudden impact. Incorporation of lignocellulosic fibres creates a hindrance to the propagation of an initial crack at the specimen notch. This subsequently leads to the generation of additional cracks along the weak fibre/matrix interface, propagating longitudinally in the direction of the specimen's length (Salman et al., 2016). Das et al., (2018) similarly, in their study on Jute fibre reinforced polypropylene composites, found a fibre loading of 50 wt.% to exhibit maximum strength. The authors asserted that at this fibre loading the fibres are properly oriented and homogenous throughout the matrix, hence uniform distribution of applied stress. Huzaifah et al., (2019) also reported that fibre interaction with the notched crack allows for energy absorption upon impact that ultimately slows down fracture. At lower fibre content, the fibres are insufficient to absorb the impact energy hence the low impact energy exhibited by the specimens at 40 wt.% fibre loading.

Figure 4.10 shows a decrease in the impact energy absorbed when fibre content is increased beyond 50 wt.% across all the specimen configurations. Fibre-to-fibre interactions are high due to high fibre composition in 60 wt.% fibre loading specimens. The specimens are prone to micro-crack formations in the composite surface and agglomerations, diminishing the ability to withstand shock impact loadings.

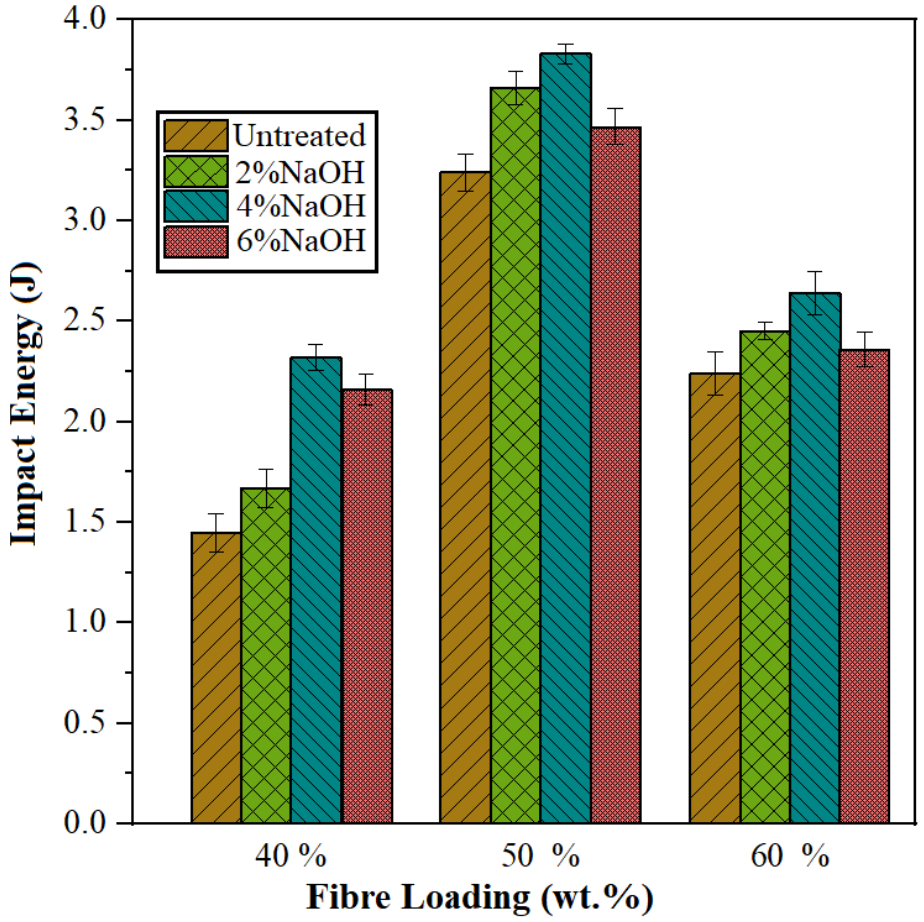

Figure 4.10: Impact energy of Mokolwane fibre reinforced polyester composites.

Another pattern from the results is the influence of alkali treatment on the composites. The impact energy of the 40 wt.%, 50 wt.% and 60 wt.% composites were increased by 60%, 18.2%

and 17.9% respectively when treated by 4%NaOH. This outcome indicated a notable enhancement in the impact testing performance following the alkali treatment of the composite. Alkali treated improved the fibre-matrix bonding producing a composite with enhanced crack propagation resistance. The optimum alkali treatment concentration was found at to be 4% NaOH. A further increase in alkali concentration was found to be detrimental to the impact properties of the composites. This is ascribed to the higher rigidity and enhanced brittleness that consequently diminish the capacity of the composite to absorb impact energy (Prasanthi et al., 2022).

4.3.7 Scanning Electron Micrograph (SEM) of Composites

The composites were observed under the SEM to assess their microstructure and fibre-matrix interaction. Untreated fibre-reinforced composites are observed in Figure 4.11 show that the fibres in the composite are intact. This explains why mechanical properties are compromised due to the inability of the resin to diffuse through elementary fibres. The figure also shows homogenous zones of polyester resin throughout the composites. This likely occurs due to the mismatch between hydrophilic fibres and the hydrophobic polyester matrix, alongside impurities and surface irregularities on the Mokolwane fibre. This observed poor fibre distribution is detrimental to the stress transfer capability of the resin to the fibre reinforcement. When the composite is subjected to tensile, bending, and impact loads, the inherent resin brittleness leads to failure.

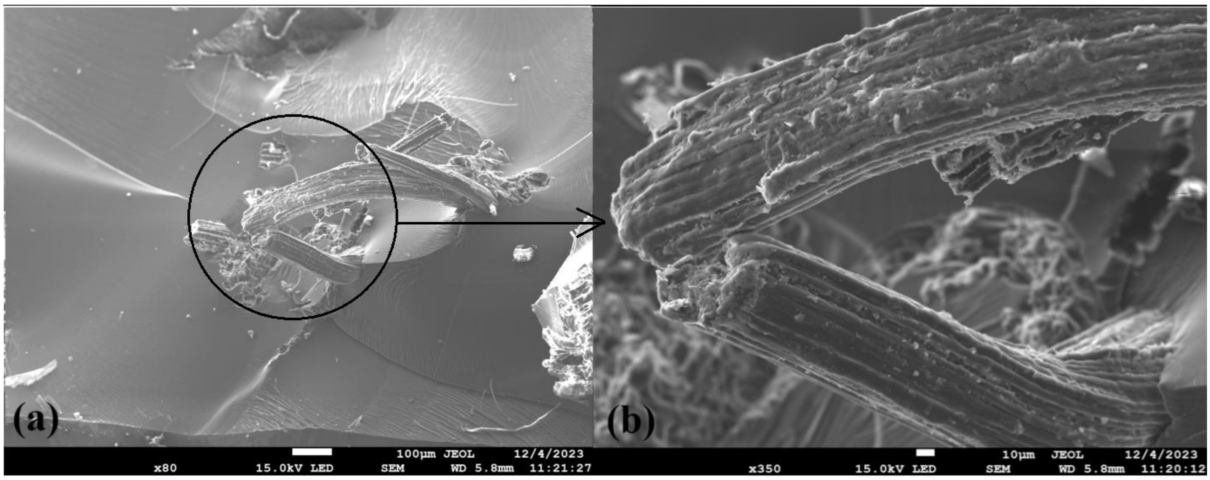

Figure 4.11: SEM images of Untreated fibre composite (UPM_40)

Figure 4.12 shows the microstructure of TPM_50_4SH. The figure shows good distribution of elementary fibres throughout the matrix. This shows that NaOH was effective to separate the fibres into fibrils, allowing for good resin diffusion throughout the fibres. This composite has been found to possess good mechanical properties as a result. Good fibre interfacial bonding is instrumental when composites are used to manufacture industrial applications components so that they will be less prone to moisture absorption and weakened mechanical properties.

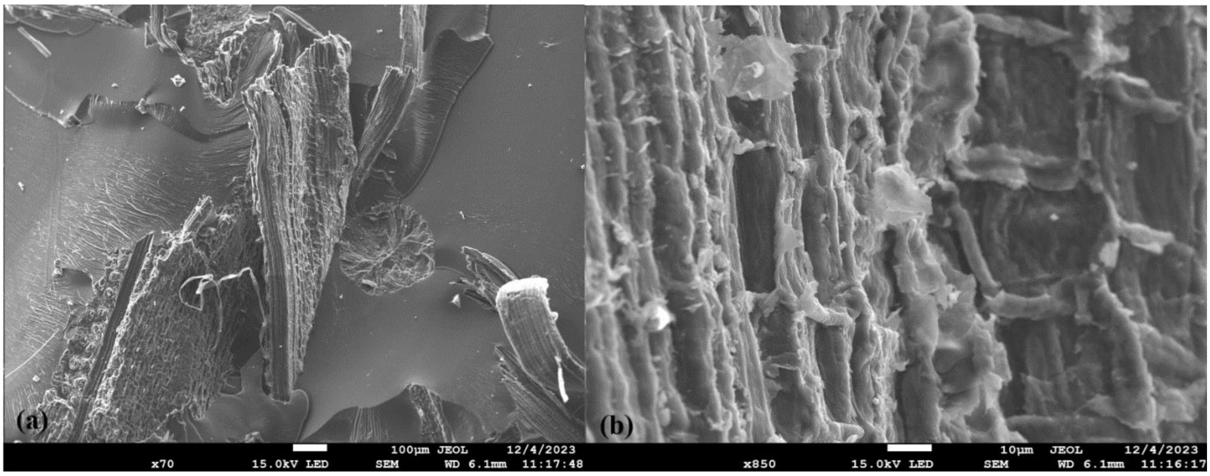

Figure 4.12: SEM images of TPM_50_4SH

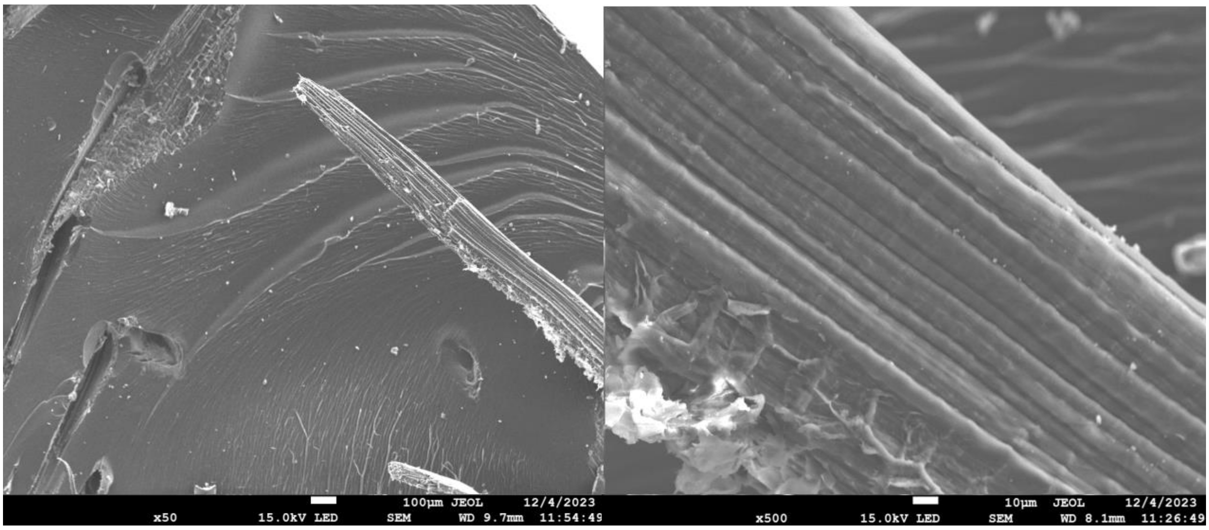

Figure 4.13 SEM images of TPM_60_6SH

Figure 4.13 shows the microstructure of Mokolwane fibre reinforced polyester composite with 60% fibre loading as well as treated with 6%NaOH prior to fabrication. There is significant agglomeration of the elementary fibres, likely because of high fibre fraction. The micrograph also shows notable fibrillation because of alkali. Figure 4.5d has already shown how alkali treatment is evident on the fibre surface when 6%NaOH has been employed. The excessive fibre treatment has been observed to diminish the composite's mechanical properties in this work.

Table 4.7: Comparison of mechanical properties of Mokolwane reinforced polyester composite with other natural fibre composites.

Composite	Tensile Strength (MPa)	Tensile Modulus(GPa)	Flexural Strength(MPa)	Flexural Modulus(GPa)	Impact Energy (J) /Strength	Reference
Alkali treated Mokolwane & Polyester	76.5	0.52	124	2.67	3.83	This work
Alkali treated *Ziziphus mauritiana* fibre & epoxy	55.08	1.83	59.48	7.496	4.25	Vinod et al., (2022)
Alkali treated Enset, sisal & epoxy	122.56	-	116.30	-	24.17 kJ/m^2	Bekele et al., (2023)
Oil palm fibre & Polypropylene	25-30	~3	~32	~3	42.6 kJ/m^2	Ramli et al., (2012)
Silane treated *Caryota urens* & Epoxy	164	6.56	237	6.52	4.65	Arun Prakash et al., (2022)
Sansevieria, NaOH treated sisal and polyester	48.47	2.72	69.17	3.33	16.34 kJ/m^2	Premkumar et al., (2022)
Coccinia grandis fibre & Epoxy	37	2.1	78	2.4	32.78 kJ/m^2	Ramasamy et al., (2022)
Wheat straw & epoxy	15.8	1.32	35.5	2.2	31.2 kJ/m^2	Mittal & Sinha, (2017)
Polyester + Hemp Fibre	28.87	0.38	60.06	1.86	-	Neves et al., (2019)

4.4 Chapter Summary

This chapter outlined the outcomes of the experimental work of this dissertation. The results show that after alkali treatment, the moisture absorption of fibres is reduced. This is posited to be a result of hemicellulose removal that reduces the hydrophilicity. Hemicellulose removal is corroborated by findings from FTIR analysis. The influence of alkali treatment is further seen on the thermogravimetric and morphology analysis of the fibres. The findings from the composite mechanical tests show that increasing fibre loading, and alkali treatment is beneficial. However, excessive fibre loading, and treatment leads to insufficient fibre wetting and fibre cellulose degradation, respectively. The optimum fibre loading, and alkali treatment were found to be 50 wt.% and 4%NaOH respectively.

CHAPTER 5: DISCUSSION & CONCLUSION

5.1 Introduction & Overview

In this dissertation, a composite was synthesized from Mokolwane fibres and polyester resin to evaluate its viability as a material to be used in industrial applications. Several tests were conducted on the fibre, composite, and the influence of alkali treatment and fibre content were assessed to achieve the research aims of this study. The previous chapters presented the thesis premise, reviewed the available literature on natural fibre reinforced composites, outlined the methodological framework carried out, and delineated the outcomes from the experiments conducted in this work. In this chapter these results are further assessed to look at the possible industrial applications of the composite, contributions of research, and the possible future works.

5.2. Discussion of Results & Industrial Applications of Mokolwane Fibre Reinforced Composites

5.2.1 Discussion of Results

Extracted Mokolwane fibres for this work were found to be mostly in the 450-500 μm range, with a median fibre diameter of 465 μm. The fibre extraction technique is likely to determine the resulting diameter of the fibres. This study determined that moisture absorption of Mokolwane fibres was reduced by 33%, 46%, and 52% after they underwent alkali treatment by 2%NaOH, 4%NaOH, and 6%NaOH respectively. This is postulated to be a result of hemicellulose removal by NaOH treatment. The thermogravimetric analysis of a Mokolwane fibres showed them to have a thermal stability of 258 °C, but no appreciable changes because of alkali treatment. In addition, the fibres' thermal stability is significantly lower than those of synthetic fibres, meaning that they cannot be applied in very high temperature processing techniques. FTIR analysis of fibres denoted a presence of polysaccharides in raw fibres. Spectra showed disappearance of some absorption bands and peaks as result of alkali treating Mokolwane fibres. Further analysis by SEM showed a presence of a deleterious layer on the compact raw fibres. This layer was observed to disappear after alkali treatment as well as defibrillation of the fibre into elementary fibres.

The composites were tested for water absorption characteristics by moisture absorption and thickness swelling long-term immersion tests. The rates of both these were found to positively correlate with fibre volume fraction of composites. This has been deduced to be due to a higher presence of free OH- groups, susceptibility to agglomerations and voids, and insufficient fibre wetting by polyester resin. Alkali treatment was found reduce water absorption and thickness swelling of the composites due to the reduction of hydrophilic OH- groups. Mechanical properties the composites were evaluated by the tensile, flexural and impact test. Composites fabricated with 50 wt.% of fibre treated with 4%NaOH were found to be ideal from the tested samples with tensile strength, flexural strength, and impact energy of 76.5MPa, 124 MPa, and 3.83J, respectively. Positive correlation of an increase of both alkali treatment and fibre volume fraction with mechanical properties was exhibited by composites. However, beyond 50 wt.% fibre volume and 4%NaOH treatment no improvements were observed. High alkali concentration is deemed harmful to the fibres, undercutting their ability to efficiently stress transfer under tensile, bending or impact loads. Higher fibre loadings are deemed detrimental to the overall mechanical properties of a composite as they lead to agglomerations, voids and cracks diminishing its ability to withstand loads.

5.2.2 Incorporation of Mokolwane Fibre Reinforced Polyester Composite in Industrial Applications

Given the results derived from test performed on Mokolwane fibre reinforced composite, possible applications of the composite in industrial applications can be assessed.

The automotive industry has embraced natural fibre composites as a viable material choice, with notable application seen in the door panelling of Bavarian Motor Works' (BMW) i3 model. Incorporating Kenaf fibre reinforced polypropylene, BMW has showcased the potential for utilizing natural fibre composites in automotive manufacturing (Schmiedel et al., 2014). In a research study by Zampaloni et al., (2007), Kenaf fibre reinforced polypropylene composite were reported to have tensile and flexural strengths of 45 MPa and 55 MPa, respectively. These values closely align with those observed in Mokolwane fibre reinforced polyester composites, underscoring their suitability in vehicle door panel applications. Mokolwane fibre reinforced polyester composites show significantly lower moisture uptake than Kenaf fibre reinforced PP composites(around 18%), as found by Lee et al., (2013). Further studies by Loganathan et al.,

(2022) suggests that Kenaf fibres exhibit comparable decomposition patterns and thermal stability to Mokolwane fibres, meeting the manufacturing temperature requirements for door panel applications. It’s noteworthy that BMW’s i3 door panels are coated with a protective film, shielding the composite from mechanical, chemical, and ultraviolet light exposure, thus mitigating potential degradation concerns.

Another natural fibre, flax, has found use as a reinforcement for epoxy resin, for application in semi-structural use like body panels, to make cars lighter and more energy efficient. This composite has found application as a carbon fibre reinforced epoxy replacement in McLaren’s Formula 1 seats, and Porsche Cayman 718 GT4 CS MR’s full bodykit. A study by Cerbu, (2015) has reported that the tensile and flexural strengths of flax fibre reinforced epoxy composite are 68.3MPa and 102MPa, respectively, hence they are comparable to those of Mokolwane fibre reinforced polyester composite found from this study. Hybridizing the flax-epoxy composite with carbon fibre has been found to substantially increase the tensile properties enough to utilize the composite in structural applications (Dhakal & Sain, 2019). Comparable performances of the two composites opens ventures to do the same for Mokolwane fibre reinforced polyester composites to use them in automotive applications.

Fibres derived from palm trees, such as those from date palms, have been studied as effective additives for concrete blends, particularly for improving flexural strength (Althoey et al., 2022; Aminova & Sikora., 2022). Consequently, Mokolwane fibres could potentially find application in today's construction industry to enhance the performance of structural elements. Other palm fibres, like Oil palm fibre, are used in the construction of windows, door frames, insulation panels, and roofings (Abdollahiparsa et al., 2023). Oil palm is shown in **Table 4.3** to possess similar physical properties to Mokolwane in terms of density, diameter, and moisture absorption. The widespread presence of Mokolwane trees, combined with their good regenerative abilities, could significantly enhance the construction industry in Botswana, while reducing reliance on importation.

5.3 Contributions of Research

Characterization of Mokolwane Fibre. The research findings presented herein significantly advance our understanding of a novel material. Through an extensive examination of its physical properties, water absorption capabilities, thermal behaviour, and morphological structure, this study contributes to the broader revalorization of natural fibres. By providing a comprehensive reference point, it equips future researchers with the necessary tools to delve into investigations involving this fibre or other emerging variants. Furthermore, this research elucidates the extraction process, treatment methodologies, and characterization techniques essential for studying this fibre. This knowledge not only facilitates further exploration but also serves as a foundational framework for conducting comprehensive experiments. Moreover, by highlighting the potential of this fibre as an alternative material in the sustainable development of natural fibre composites for industrial applications, this work is significant in the field of materials and design engineering. It prompts a reconsideration of conventional approaches, advocating for the integration of this fibre into the discourse on sustainable materials.

Mechanical Properties of Mokolwane Fibre Reinforced Polyester Composite. This svelte dissertation delves into an examination of the tensile, flexural, and impact properties of a novel fibre-reinforced polymer composite. Through meticulous evaluation, the study investigates the effects of fibre volume fraction and alkali treatment on the mechanical characteristics of the composite. By shedding light on optimal fibre composition and treatment concentrations, this research provides invaluable insights for future endeavours in material engineering. Armed with this knowledge, researchers can make informed decisions regarding fibre selection and treatment methodologies. Furthermore, the comprehensive analysis of mechanical properties presented in this study serves as a valuable reference for material engineers venturing into the exploration of new materials for both structural and non-structural applications across diverse industries.

Water Absorption of Mokolwane Fibre Reinforced Composites. In addition to assessing the mechanical performance of Mokolwane fibre-reinforced polyester composites, we also evaluated their moisture absorption and thickness swelling. These properties are crucial for understanding their suitability for various applications, particularly in industries such as marine engineering, where materials are often exposed to prolonged immersion in water. High levels of moisture absorption and thickness swelling can significantly compromise the mechanical integrity of composites. Therefore, investigating these aspects becomes imperative, especially considering the stringent regulations aimed at promoting sustainability in product manufacturing, particularly in terms of their end-of-life scenarios. Consequently, our study serves as a vital benchmark for further research into natural fibre composites, particularly concerning their viability for marine applications. By emphasizing these critical parameters, we aim to encourage further assessments and advancements in this field, ultimately driving innovation towards more sustainable and durable materials.

Sustainability. The United Nations' Sustainable Development Goals (SDGs) 11-15 underscore the urgency of addressing climate change, promoting responsible production processes, and safeguarding marine ecosystems. The prevalent reliance on petroleum-based products contributes significantly to greenhouse gas emissions, while synthetic fibres are a primary source of microplastic pollution in oceans. Developing lightweight products that are biodegradable not only reduces emissions but also mitigates the environmental impact of waste accumulation. This shift towards sustainable materials ensures the responsible use of resources and aligns with the objectives outlined in the SDGs. Furthermore, this research serves as a catalyst for innovation, prompting material engineering researchers to explore the potential of natural fibre-reinforced composites. By fostering a re-evaluation of conventional materials and encouraging the adoption of eco-friendly alternatives, it paves the way for a more sustainable future.

Research Outputs:

- **Seisa, K.**, Chinnasamy, V., & Ude, A. U. (2022). "Surface Treatments of Natural Fibres in Fibre Reinforced Composites: A Review." *Fibres & Textiles in Eastern Europe*, 30(2), Article 2. https://doi.org/10.2478/ftee-2022-0011

- **Seisa, K.**, Chinnasamy, V., & Ude, A. U. (2024). "Optimizing Mode I Fracture toughness in Fibre Reinforced Composites: A Review." Submitted to *Journal of Composite Materials.*

- **Seisa, K.**, Chinnasamy, V., & Ude, A. U. (2024). "Effect of fibre treatment on moisture absorption and mechanical properties of Mokolwane fibre reinforced polyester composites." Submitted to *Fibers and Polymers*.

5.4 Future Works

Treatment variations. Several surface treatments like acetylation and silane treatment have been noted by researchers for their ability to improve fibre-matrix interfacial adhesion(Seisa et al., 2022). There is gap to investigate how they could be used with the composite that is the focal point of this research. In addition to that, the influence of different variations of treatments has not been explored. These include treatment duration, combinations of different treatments as well as the environment conditions during treatments.

Manufacturing Technique Influence. Various ways to fabricate fibre reinforced composites have been outlined in the literature review of this dissertation along with their advantages and drawbacks. However, due to inordinate time and resources required to undertake them, their impact on the properties was not thoroughly investigated. Optimizing the manufacturing process and its attendant parameters would ensure production of high-quality materials.

Hybridization. Exploring how the incorporation of Mokolwane fibre with another natural or synthetic fibre to reinforce polyester performs. Materials synthesized from hybridization would make great substitutes for conventional materials in structural and non-structural applications as they have been touted for their superior properties over single-fibre composite.

Fracture Toughness & Hardness studies. The incorporation of natural fibre reinforced composites in industries like aerospace, where they are susceptible to high velocity impact loads (such as bird strikes or hailstorms), manifestly provides fertile grounds for study of their performance in mode I, mode II, and mixed modes fracture toughness tests. Various reinforcement techniques that include interleaving, z-pinning, stitching, and multi-scale reinforcement to improve fracture toughness of the composite should also be explored. Similarly, the resistance of the material to localized deformation can be explored using micro hardness tests.

Life Cycle Analysis (LCA) studies. Despite their positive ecological effect, the full-scale use of natural fibre composites in industrial applications poses various environmental problems, that include but not limited to, eutrophication from fertilizer use, emission of nitrogen and phosphorus during cultivation, and large arable-land requirements. Before integrating Mokolwane fibre reinforced polyester composite in industrial applications, conducting a comprehensive LCA study is essential. The study should analyse the ecological impact throughout the product entire life cycle, encompassing raw material extraction, production, usage, and end-of-life scenarios. Moreover, LCA studies should incorporate economic impacts aspects, such as life cycle costing, and assess the composite’s durability. This holistic approach provides valuable insights for material engineers engaged in research and technological development using bio composites. However, a significant challenge lies in the absence of novel materials like Mokolwane fibres from product databases such as SimaPro, Ecoinvent and GABI, which are commonly used by LCA practitioners for inventory analysis. Addressing this gap will be crucial for accurate assessment and informed decision-making in sustainability-oriented design and manufacturing processes.

5.5 Conclusion

The objectives of this dissertation were pursued to evaluate the viability of mokolwane as a reinforcement in fibre reinforcement composites. The fibre was characterized by a series of tests aimed at understanding its intrinsic properties, and its suitability. Through the thermal stability, moisture absorption, and physical tests undertaken the fibre was observed to give out similar performance akin to other natural fibres studied. The surface treatment by alkali has been observed, through morphology examination by SEM, to dissolve the amorphous layer on the fibre surface. Moisture absorption and thickness swelling of composites were investigated, and deduced that, increase of fibre loading led to a higher water uptake, whilst alkali treatment effectively reduced that. The mechanical performance of the composites was observed to be influenced by both the alkali treatment concentration and fibre loading, with the best performance observed at 4%NaOH and 50wt.%, respectively. Comparative analyses yielded that the fibre and the composite have similar performance with other fibres and natural fibre composites.

5.6 Chapter Summary

This chapter aimed to interpret the results obtained during this study. The main theme was to recommend, based on the outcomes of the research, how the composite can be incorporated in industrial applications. Future works on the topic will look at how varying surface treatments, manufacturing techniques, hybridization affects properties. Additionally, there is a need for comprehensive assessments concerning the lifecycle of the material and studies on fracture toughness to further enhance our understanding and optimize the performance of the composite in practical applications.

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