PHYSICAL, MECHANICAL AND THERMAL PROPERTIES OF BAMBOO-KENAF FIBER REINFORCED POLYLACTIC ACID HYBRID COMPOSITES

By

ABIR KHAN

Thesis Submitted to the School of Graduate Studies,
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DEDICATION

To my beloved parents, wife and daughter

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Chairman : Professor Mohd Sapuan bin Salit @ Sinon, PhD, PEng
Faculty : Engineering

The global crisis of plastic pollution has intensified the demand for sustainable alternatives, especially in the furniture industry. While natural fiber-reinforced composites are emerging as eco-friendly solutions, research on biodegradable matrices like polylactic acid (PLA) reinforced with bamboo (BF) and kenaf (KF) fibers remains limited. This study addresses this gap by developing and characterizing three hybrid (30BF-70KF, 50BF-50KF, and 70BF-30KF) and two non-hybrid (BF-PLA and KF-PLA) composites using twin screw extrusion and compression molding techniques. The physical properties (density, void content, crystallinity via XRD, and chemical interactions via FTIR), mechanical properties (tensile, flexural, compressive, impact, and hardness), and thermal properties (Thermogravimetric analysis-TGA and Differential scanning calorimetry-DSC) were thoroughly analysed. Moreover, water absorption, surface wettability and flammability were also analysed. Key findings include that BF reinforcement reduced the composites' density to

1.1826 g/cm^3, while the inclusion of KF increased it to 1.2479 g/cm^3. BF50-KF50 hybrid composite achieved the lowest void content (0.27%) and the highest dimensional stability, while the BF-PLA composite exhibited the highest tensile (25.95 MPa) and compressive strengths (173.15 MPa). Thermogravimetric analysis (TGA) revealed the highest onset degradation temperature (484°C) in the BF30-KF70 composite. BF and KF increased moisture uptake, with BF50-KF50 hybrid composite exhibited the lowest water absorption (0.5 hours: 0.85%, 2 hours: 1.38%) and thickness swelling, indicating optimal dimensional stability. Higher kenaf content correlated with increased biodegradability, with KF-PLA being the most soluble at 7.15%. Water vapor permeability (WVP) analysis revealed that denser composites like KF-PLA have lower WVP (5.121845×10^{-10} gs^{-1}m^{-1}Pa^{-1}), suitable for moisture-sensitive application. Enhanced hydrophilicity was observed with BF50-KF50 showing the lowest contact angle (61.47°). Flammability tests (UL-94) rated all composites a V-2 rating, and LOI results indicated that BF-PLA and KF-PLA had improved LOI values (21.88% and 21.79%) compared to pure PLA (19.03%). BF50-KF50 achieved an LOI of 21.10%, indicating optimal flame resistance. These results demonstrate the potential of BF-KF/PLA hybrid composites as sustainable materials for eco-friendly furniture application.

Keywords: Biocomposites, Bamboo Fibers, Kenaf Fibers, Polylactic Acid

SDG: GOAL 12: Responsible Consumption and Production, GOAL 13: Climate Action

Abstrak tesis yang dikemukan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

SIFAT-SIFAT FIZIKAL, MEKANIKAL DAN TERMA KOMPOSIT HIBRID Asid POLIKAKTIK BERTETULANG BULUH/KENAF FIBER

Oleh

ABIR KHAN

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Krisis global pencemaran plastik telah meningkatkan permintaan terhadap alternatif lestari, terutamanya dalam industri perabot. Walaupun komposit diperkuat gentian semula jadi muncul sebagai penyelesaian mesra alam, penyelidikan mengenai matriks biodegradasi seperti asid polilaktik (PLA) diperkuat dengan gentian buluh (BF) dan kenaf (KF) masih terhad. Kajian ini menangani jurang ini dengan membangunkan dan mencirikan tiga komposit hibrid (30BF-70KF, 50BF-50KF, dan 70BF-30KF) dan dua komposit bukan hibrid (BF-PLA dan KF-PLA) menggunakan teknik penyemperitan skru kembar dan pengacuan mampatan. Sifat fizikal (ketumpatan, kandungan lompang, penghabluran melalui XRD, dan interaksi kimia melalui FTIR), sifat mekanikal (tegangan, lenturan, mampatan, hentaman, dan kekerasan), dan sifat terma (Analisis Termogravimetri-TGA dan Kalorimetri Pengimbasan Pembezaan-DSC) dianalisis secara menyeluruh. Selain itu, penyerapan air, kebolehbasahan permukaan dan kemudahbakaran juga dianalisis. Penemuan utama termasuk pengukuhan BF mengurangkan ketumpatan komposit kepada 1.1826 g/cm³, manakala penambahan KF meningkatkannya kepada 1.2479 g/cm³. Komposit hibrid BF50-KF50 mencapai kandungan lompang terendah (0.27%) dan kestabilan dimensi tertinggi, manakala komposit BF-PLA menunjukkan kekuatan tegangan (25.95 MPa) dan mampatan (173.15 MPa) tertinggi. Analisis termogravimetri (TGA) mendedahkan suhu permulaan degradasi tertinggi (484°C) dalam komposit BF30-KF70. BF dan KF meningkatkan penyerapan kelembapan, dengan komposit hibrid BF50-KF50 menunjukkan penyerapan air terendah (0.5 jam: 0.85%, 2 jam: 1.38%) dan pembengkakan ketebalan, menunjukkan kestabilan dimensi optimum. Kandungan kenaf yang lebih tinggi berkorelasi dengan peningkatan biodegradasi, dengan KF-PLA paling larut pada 7.15%. Analisis kebolehtelapan wap air (WVP) menunjukkan bahawa komposit yang lebih padat seperti KF-PLA mempunyai WVP lebih rendah (5.121845×10^{-10} $gs^{-1}m^{-1}Pa^{-1}$), sesuai untuk aplikasi sensitif kelembapan. Peningkatan hidrofilisiti diperhatikan dengan BF50-KF50 menunjukkan sudut sentuh terendah (61.47°). Ujian kemudahbakaran (UL-94) memberikan penarafan V-2 kepada semua komposit, dan keputusan LOI menunjukkan bahawa BF-PLA dan KF-PLA mempunyai nilai LOI yang lebih baik (21.88% dan 21.79%) berbanding PLA tulen (19.03%). BF50-KF50 mencapai LOI 21.10%, menunjukkan rintangan api optimum. Keputusan ini menunjukkan potensi komposit hibrid BF-KF/PLA sebagai bahan lestari untuk aplikasi perabot mesra alam.

Keywords: Biokomposit, Serabut Buluh, Serat Kenaf, Asid Polilaktik

SDG: MATLAMAT 12: Penggunaan dan Pengeluaran yang Bertanggungjawab
MATLAMAT 13: Tindakan Iklim

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

BF	Bamboo Fiber
KF	Kenaf Fiber
PLA	Polylactic Acid
NFRPC	Natural Fiber Reinforced Polymer Composites
DP	Direct Polycondensation
AP	Azeotropic Polycondensation
ROP	Ring Opening Polymerization
TMPD	N,N,N',N'-tetramethyl-1,4-phenylenediamine
MWCNT	Multi-Walled Carbon Nanotubes
OMMT	Organically Modified Montmorillonite
ASTM	American Society for Testing and Materials
FTIR	Fourier Transform Infrared
XRD	X-Ray Diffraction
UTM	Universal Testing Machine
FESEM	Field Emission Scanning Electron Microscopy
TGA	Thermogravimetric Analysis
DTG	Derivative Thermogravimetry
DSC	Differential Scanning Calorimetry
WVP	Water Vapor Permeability
LOI	Limiting Oxygen Index

LIST OF SYMBOLS

^{0}	Degree
^0C	Degree Celsius
ρ	Density
gm	Gram
hr.	Hour
Pa	Pascal
MPa	Mega Pascal
GPa	Gega Pascal
%	Percentage
µm	Micrometre
mm	Millimetre
C_i	Crystallinity index
W_i	Initial weight
W_f	Final weight
T_i	Initial thickness
T_f	Final thickness

CHAPTER 1

INTRODUCTION

1.1 Background of the Research

Every year, 8 million metric tons of plastic waste make their way into the oceans due to the prevalent use of synthetic plastics and poor disposal methods (Kurtela & Antolović, 2019). It is estimated that by 2025, over 100 million metric tons of plastic waste from coastal regions, which is not properly managed, will enter the oceans (Liu et al., 2022). Consequently, there is an increasing emphasis on sustainable practices in the production of furniture and other consumer goods. There is a growing consumer demand for the incorporation of environmentally friendly materials in the production process to reduce greenhouse gas emissions. For example, a study on wood-based furniture suggests that materials significantly contribute to the greenhouse gas emissions associated with these items, representing 38-90% of total emissions (Linkosalmi et al., 2016). Moreover, the escalating global concerns about climate change and the decreasing availability of petrochemical resources have heightened the appeal of natural fiber reinforced composites (NFRC) for various uses (Kumar et al., 2022). NFRCs offer several advantages, such as a lower carbon footprint, natural environmental compatibility, and better physical and mechanical properties than synthetic alternatives (Nazrin et al., 2021).

1.2 Problem Statements

The escalating crisis of plastic pollution, driven by the widespread use of non-biodegradable synthetic plastics, has spurred a critical need for sustainable alternatives in the furniture industry. While natural fiber reinforced composites (NFRCs) have shown promise, research into the potential of bamboo and kenaf fiber-reinforced biodegradable matrices like polylactic acid (PLA) remains limited. Most previous studies have focused on using these fibers in epoxy matrices, which, although improving mechanical properties, still pose environmental concerns due to their non-biodegradable nature. This research aims to address this gap by exploring the development of hybrid composites using PLA as a matrix to reduce environmental impact. Specifically, the study will comprehensively evaluate the physical, mechanical, and thermal properties of these hybrid composites, as well as their water absorption, surface wettability, and flammability characteristics. By examining these crucial properties, this research seeks to advance our understanding of bamboo and kenaf fiber-reinforced PLA hybrid composites and their potential as sustainable alternatives in furniture manufacturing.

1.3 Research Objectives

The general objective of this research is to develop and characterize hybrid composites reinforced with bamboo and kenaf fibers using polylactic acid as a matrix. The specific objectives to achieve this goal include:

1. To evaluate physical, mechanical and thermal properties of bamboo and kenaf fibers reinforced polylactic acid (PLA) hybrid composites.

2. To evaluate water absorption, surface wettability and flammability properties of bamboo and kenaf fibers reinforced polylactic acid (PLA) hybrid composites.

1.4 Significance of the Research

The significance of this research lies in its comprehensive investigation of bamboo (BF) and kenaf (KF) fiber-reinforced polylactic acid (PLA) hybrid composites. The research advances understanding of the physical, mechanical, thermal, and moisture-related properties of these eco-friendly materials. The study highlights the superior mechanical and thermal properties of BF-PLA composites, particularly in terms of tensile and compressive strength, making them suitable for load-bearing applications. The research also focuses on the water absorption, surface wettability, and flammability of these composites, demonstrating their potential for moisture-resistant and flame-retardant applications. Together, these studies underscore the versatility and sustainability of BF-KF/PLA composites for various industrial applications, paving the way for future research into advanced materials with enhanced properties.

1.5 Research Scope and Limitation

This research focuses on the development and characterization of BF-KF/PLA hybrid composites for potential application in the furniture industry. The study encompasses:

- Fabrication of three hybrid (30BF-70KF, 50BF-50KF, and 70BF-30KF) and two non-hybrid (BF-PLA and KF-PLA) composites using twin screw extrusion and compression molding techniques.
- Evaluation of physical properties such as density, void content, crystallinity (via XRD), and chemical interactions (via FTIR).
- Evaluation of mechanical properties through tensile, flexural, compressive, impact, and hardness tests.
- Morphological analysis of fractured surfaces using Field Emission Scanning Electron Microscopy (FESEM).
- Analysis of thermal properties using Thermogravimetric Analysis (TGA) and Differential Scanning Calorimetry (DSC).
- Investigation of water absorption characteristics, surface wettability, and flammability of the composites.

While this research provides valuable insights into BF-KF/PLA hybrid composites, several limitations are identified:

- Only specific fiber ratios (30:70, 50:50, 70:30) are explored for hybrid composites; other ratios may offer different results.
- The fabrication methods are limited to twin screw extrusion and compression molding, potentially limiting the scope to other manufacturing techniques.
- The research does not involve long-term durability tests or the prototyping of actual furniture products.
- Economic feasibility and large-scale production challenges are not addressed.

- Advanced surface treatments and incorporation of nanomaterials (e.g., carbon nanotubes, graphene nanoplates) are beyond the scope but are recommended for future research.

- The study does not assess the composites' performance under varying environmental conditions (e.g., extreme temperatures, humidity).

1.6 Structure of Thesis

The following are the specifics of the thesis structure:

Chapter 1

This chapter outlines the research background, defines the problem statements, and details the objectives of the study. Additionally, it discusses the importance and the breadth of the research work and provides an overview of the thesis structure.

Chapter 2

This chapter provides a comprehensive review of the literature related to the topic of this research. Additionally, it addresses the research gaps identified during the study, discussing how they are explored within the chapter.

Chapter 3

This chapter focuses on the methodologies employed in this research, including the selection of materials, preparation processes, fabrication techniques, testing procedures, and methods of data collection.

Chapter 4

This chapter presents the results and discussion of the research work, divided into six sections. The first three sections focus on the physical, mechanical, and thermal properties of bamboo-kenaf fiber-reinforced polylactic acid hybrid composites. It examines the impact of varying bamboo and kenaf fiber loading on the composites' physical properties (density, void content, chemical interactions and crystallinity), mechanical properties (tensile, flexural, compressive, impact and hardness), and thermal characteristics.

The remaining sections explores the water absorption, surface wettability and flammability properties of bamboo-kenaf fiber-reinforced polylactic acid hybrid composites. Specifically, it investigates how different loading of bamboo and kenaf fiber affect the water absorption, rainwater absorption, thickness swelling, water solubility, water vapor permeability, water contact angle and flammability properties of the composites.

Chapter 5

This final chapter summarizes the overall conclusions drawn from the entire research and offers recommendations for future enhancements and investigations related to this study.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In the field of composite research, natural fibers such as jute, bamboo, kenaf, flax, sisal, hemp, banana, and pineapple leaf fibers are increasingly used as reinforcing materials (Eyupoglu, 2020). Among these, bamboo (BF) and kenaf (KF) fibers are particularly noted for their excellent physical, mechanical, and thermal properties, attracting significant attention from researchers. These fibers are not only lightweight but also offer strength comparable to synthetic materials, making them ideal for various industrial uses. Their rapid growth and widespread availability also add economic benefits. When paired with biopolymers like polylactic acid (PLA), composites reinforced with bamboo and kenaf fibers offer a sustainable alternative to conventional plastic products. Notably, PLA is environmentally favorable compared to other plastics, emitting approximately 1600 kilograms of CO_2 per metric ton during its breakdown (Rajeshkumar et al., 2021). Furthermore, the use of agricultural raw materials in the production of PLA contributes to CO_2 absorption, enhancing its environmental benefits.

2.2 Mechanical Properties of BF-KF Fiber Reinforced Hybrid Composites

Several research have highlighted significant advancements in hybrid composites made from bamboo and kenaf fibers, particularly with an epoxy matrix. Ismail et al. (Ismail et al., 2019) reported improved tensile properties and reduced void content in a 50% bamboo-50% kenaf composite, achieving a tensile strength of 55.18 MPa and a modulus of 5.15 GPa. These improvements suggest the hybrid composites' suitability for high-strength, low-void applications. Safwan et al. (Safwan et al., 2018) proposed using bamboo and kenaf mats as reinforcement for epoxy resin polymer matrix, which enhanced the mechanical properties of neat epoxy. Bamboo mat composites showed the highest tensile strength (53.03 MPa), while kenaf mat composites had the highest tensile modulus (4.71 GPa). Scanning electron microscopy revealed better interfacial adhesion in bamboo mat-epoxy composites.

Studies have also explored the impact of nano additives like multi-walled carbon nanotubes (MWCNTs) and organically modified montmorillonite (OMMT) nanoclay. Prabhudass et al. (Prabhudass et al., 2022) found that MWCNTs significantly increased tensile strength (up to 67.3 MPa) and improved impact energy absorption in bamboo/kenaf fiber reinforced epoxy composites. These enhancements also improved thermal stability and structural integrity, making the material suitable for applications requiring strength and impact resistance. Chee et al. (Chee et al., 2021) examined the effect of OMMT nanoclay, reporting high tensile (55.82 MPa), flexural (105 MPa), and impact strengths (65.68 J/m) in OMMT-filled composites, highlighting their potential for lightweight, high-strength structural applications. Chee et al. (Chee et al., 2020) also noted that OMMT improved moisture resistance and thermal expansion behavior, indicating the material's suitability for applications requiring dimensional stability. While these studies show the promise of bamboo and kenaf fiber-reinforced epoxy composites, they also highlight environmental concerns due to epoxy's non-biodegradability (Kumar et al., 2018). Therefore, there is a critical need to explore sustainable composite materials derived from bamboo and kenaf fibers, marking an important area for future research.

2.3 Thermal Properties of BF-KF Fiber Reinforced Hybrid Composites

The glass transition temperature (T_g) and melting temperature (T_m) are critical in determining a polymer's characteristics. When a polymer exceeds its Tm, it begins to melt and flow, which significantly affects its mechanical properties due to increased molecular chain mobility. This flow reduces the polymer's viscosity, enhancing its processability. For polylactic acid (PLA), the processing temperature range is between 180 to 250°C (Farah et al., 2016). Under high temperature and humidity, PLA degrades within days (Wan et al., 2019). To expand PLA's industrial and automotive applications, efforts have been made to improve its thermal stability and reduce its thermal degradation rate. Kumar and Babu (Kumar & Babu, 2024) found that reinforcing PLA with ground bamboo significantly improved its T_g, T_m, and crystallization temperature (Tc). Similarly, Shukor et al. (Shukor et al., 2014) reported enhanced thermal stability of PLA with the addition of alkali-treated kenaf fibers. Given PLA's vulnerability to high-temperature degradation, understanding how bamboo and kenaf fibers enhance the composite's performance and mitigate degradation under adverse conditions is crucial.

2.4 Water Absorption and Surface Wettability Properties of BF-KF Fiber Reinforced Hybrid Composites

Understanding the water absorption behavior of bamboo-kenaf fiber-reinforced PLA hybrid composites is essential, as moisture significantly impacts their mechanical properties and durability (Mohammed et al., 2023). Due to their hydrophilic nature, these fibers tend to absorb moisture, causing swelling, matrix plasticization, and fiber-matrix interface degradation, which reduces tensile and flexural strength (Ali et al., 2018). Significant progress has been made in developing hybrid composites using bamboo and kenaf fibers, especially with an epoxy matrix (Chee et al., 2019, 2021; Chee et al., 2020; Ismail et al., 2019; Prabhudass et al., 2022; Safwan et al., 2018). Only Chee et al. (Chee et al., 2020) investigated the water absorption behavior of bamboo-kenaf epoxy hybrid composites, finding that incorporating nanoclay, particularly organically modified montmorillonite (OMMT), significantly mitigated water absorption and thickness swelling. The B/K/OMMT hybrid composites showed the lowest water absorption at 4.19% and minimal thickness swelling at 5.62%. These improvements were attributed to the uniform dispersion of nanoclay and strong interfacial adhesion between the fibers and the epoxy matrix. There is a substantial knowledge gap regarding the water absorption behavior of bamboo and kenaf fiber-reinforced hybrid composites, particularly because most studies have focused on epoxy polymer matrices, which pose environmental concerns due to their non-biodegradable nature (Kumar et al., 2018). Thus, exploring sustainable composite materials derived from bamboo and kenaf fibers is a crucial research area.

Surface wettability or water contact angle studies are significant for composite materials due to their impact on adhesion, moisture resistance, and overall mechanical performance. Wettability studies help in designing effective surface treatments and coatings for composites. PLA is known as a biocompatible, recyclable, biodegradable polymer with hydrophobic surface characteristics, indicated by its static water contact angle range of 75-85°, affecting its cell affinity in medical applications (Tümer et al., 2022). Controlling the wettability of PLA surfaces is highlighted as an important process in both academic and industrial studies, as it can significantly impact PLA's application potential. Lowering the contact angle of PLA surfaces can accelerate degradation processes, such as hydrolysis, microbial colonization, and surface erosion (Gorrasi & Pantani, 2018). Therefore, understanding the impact of bamboo and kenaf on PLA composites' surface wettability is crucial for optimizing these properties and enhancing their application potential.

2.5 Flammability Properties of BF-KF Fiber Reinforced Hybrid Composites

Recent research has explored enhancing the fire properties of PLA-bamboo composites through various methods. These methods include the interfacial phosphorus-silicon aerogel method (Niu et al., 2022a), the interfacial silicon-nitrogen aerogel method (Niu et al., 2022b), using recycled bamboo chopsticks (Wang & Shih, 2016), phosphate and urea-grafted bamboo charcoal (Zhang et al., 2021), and bamboo fiber modified with silica aerogel (Niu et al., 2022c). These studies incorporated flame retardant additives to assess the impact of bamboo fibers on the fire properties of PLA composites.

Young and Tsao (Young & Tsao, 2015) examined alkali-treated bamboo fibers in PLA composites, using halogen-free flame-retardant particles (DMOP-8307) for fire resistance. For kenaf fibers, Jusoh et al. (Jusoh et al., 2023) reported that alkali-treated kenaf fibers significantly enhanced the fire properties of polymer composites. However, no studies have yet examined the combined effect of bamboo and kenaf fibers on the fire properties of PLA composites.

2.6 Summary

The previous studies as highlighted in Table 2.1 have focused on the epoxy polymer composites which poses environmental concerns due to its non-biodegradability (Devansh et al., 2024; Kumar et al., 2018). The existing research also lacks comprehensive analysis of a wide range of physical, mechanical and thermal properties. Additionally, majority of the studies utilized hand layup technique which is less suitable for higher production rates required in industrial production (Elkington et al., 2015). These gaps in the current literatures highlight the need for a more comprehensive study on bamboo-kenaf hybrid composites using biodegradable matrices like PLA, which could address the environmental concerns in composite materials.

Table 2.1: Summary of the Existing Research on BF-KF Hybrid Composites

Materials	Fabrication Method	Research objectives	Limitations	Reference
Fibers: Bamboo & Kenaf Polymer Matrix: Epoxy	Compression Molding	Identify the best type of bamboo fiber	1.Used epoxy as the matrix 2.Tensile and impact properties only 3. Did not investigate thermal properties	Safwan et al., 2018
Fibers: Bamboo & Kenaf Polymer Matrix: Epoxy	Hand layup	Investigate void content, tensile, vibration, and acoustic properties	1.Used epoxy as the matrix material. 2.Used hand lay-up method 3. Tensile properties only 4.Did not investigate thermal properties	Ismail et al., 2019
Fibers: Bamboo & Kenaf Polymer Matrix: Epoxy Additives: Nanoclay (MMt, OMMT, HNT)	Hand lay-up	Investigate density, void content, water absorption, thickness swelling, and thermal expansion	1.Used epoxy as the matrix 2. Physical and thermal properties only	Chee et al., 2020
Fibers: Bamboo & Kenaf Polymer Matrix: Epoxy Additives: Nanoclay (MMT, OMMT, HNT)	Hand lay-up	Investigate mechanical and dynamic mechanical properties	1.Used epoxy as the matrix 2.Only tensile, flexural and impact properties 3.Lack of thermal analysis	Chee et al., 2021
Fibers: Bamboo & Kenaf Polymer Matrix: Epoxy Additives: Multi-walled carbon nanotubes (MWCNTs)	Hand lay-up	Investigate mechanical properties	1.Used epoxy as the matrix 2.Only tensile and impact properties	Prabhudass et al., 2022

As highlighted from literature review, there is a significant knowledge gap regarding bamboo and kenaf fiber reinforced PLA hybrid composites and their influence on physical, mechanical, and thermal properties. Moreover, there is lack of comprehensive study on water absorption, surface wettability and flammability properties. This research aims to fill that gap by examining two non-hybrid composites (BF-PLA and KF-PLA) and three hybrid composites (BF30-KF70, BF50-KF50, and BF70-KF30) in comparison to pure PLA. The study evaluates physical properties such as density, void content, crystallinity (XRD), and chemical interaction (FTIR). It also includes five mechanical tests: tensile, flexural, compressive, impact, and hardness. Thermal properties were analyzed using TGA and DSC, providing insights into the hybrid composites. Additionally, the study assesses water absorption, rainwater absorption, thickness swelling, water solubility, water vapor permeability, and surface wettability (water contact angle test). Flammability properties were also examined using UL-94 (vertical) and Limiting Oxygen Index tests.

CHAPTER 3

MATERIALS AND METHODS

3.1 Introduction

This chapter covers every aspect of methodology of this research. **Figure 3.1** shows the research methodology flow.

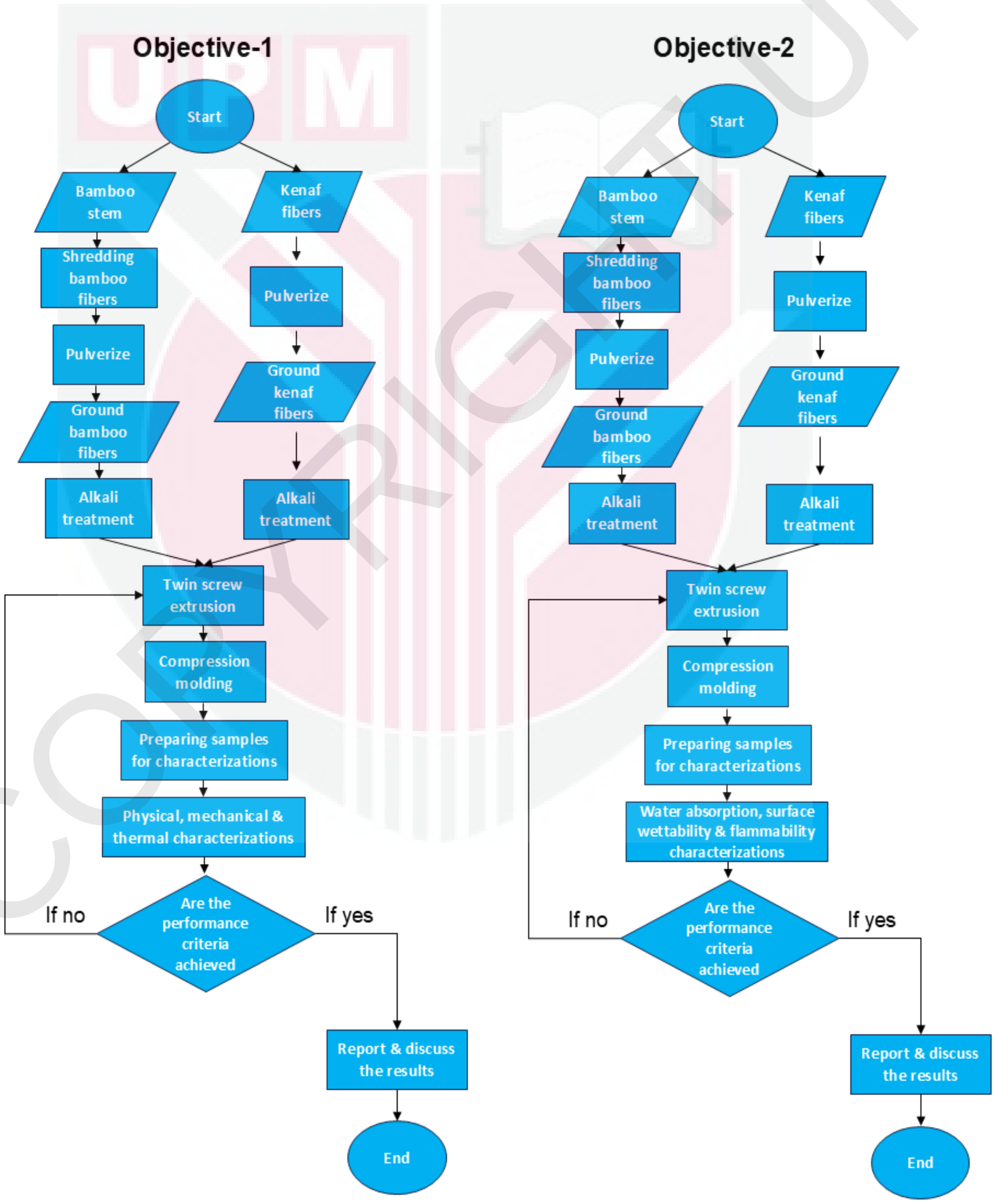

Figure 3.1 : Detailed Methodology of BF-KF/PLA Hybrid Composites

3.2 Materials

Bamboo stems (*Schizostachyum brachycladum*) and kenaf core fibers were supplied by Origin Fitrah Valley (Serdang, Malaysia) and National Kenaf & Tobacco Board (Malaysia) respectively. The bamboo stems were processed to ground form as depicted in **Figure 3.2**. The size of both the bamboo and kenaf fibers ranged from 500-1000 μm. Bamboo fibers have a measured density of 1.1g/cm^3, while kenaf fibers have a measured density of 1.4g/cm^3. The polylactic acid (PLA) 2003D grade and maleic anhydride used in this experiment were supplied by Natureworks LLC (USA) and Sigma Aldrich (USA) respectively.

Figure 3.2 : Bamboo Fiber Conversion Process from Stems to Powder Form

3.3 Alkali Treatment

Bamboo and kenaf fibers underwent alkali treatment using a 5% w/v NaOH solution. The solutions were stirred on a magnetic stirrer plate at the temperature of 80 °C for 4 hours and at 2000 rpm (**Figure 3.3**). Both fibers were washed with distilled water after the alkali treatment. The wash helped remove lignin, wax, fat, hemicellulose, impurities and lower-molecular-weight compounds. Lastly, an electric oven was used to dry the treated fiber at the temperature of 80 °C for 24 hours.

Figure 3.3 : Alkali Treatment Process of Bamboo and Kenaf Fibers

3.4 Fabrication of BF-KF/PLA Hybrid Composites

Bamboo and kenaf fibers were reinforced with PLA according to the proportions detailed in **Table 3.1**. Along with the fibers and PLA, 5 wt% maleic anhydride was also added. The composite filaments were prepared using a twin-screw extruder machine (Brand-Micromac). The feeder screw frequency was set at 5 hertz and the processing temperatures of the eleven zones in the extruder machine were set at 180°C, 180°C, 180°C, 185°C, 185°C, 190°C, 190°C, 195°C, 200°C, 205°C, and 210°C respectively. The extruded bamboo-kenaf/PLA filaments were cooled down to the ambient temperature in a water bath. Subsequently, the filaments were chopped into small pellets with a pelletizer and then dried at 80 °C for 5 hours to eliminate moisture. Following this, the dried pellets were loaded into a mold for compression molding. A processing temperature of 170 °C was set at the compression molding machine. Preheat time, pressing time and cold press time were set at 3, 3 and 5 minutes respectively. Composites in sheet form were developed at the dimensions of 150 mm (L) x 150 mm (W) x 3 mm (T). Smaller sized test specimens were cut out from the sheets according to the requirements of specific characterizations. The composites fabrication process is depicted in **Figure 3.4**.

Table 3.1 : Formulation of BF-KF/PLA Hybrid Composites

Composites	Weight Ratio %		PLA
	Bamboo Fiber	Kenaf Fiber	
Pure PLA	-	-	100
BF-PLA	25	-	75
KF-PLA	-	25	75
BF30-KF70	7.5	17.5	75
BF50-KF50	12.5	12.5	75
BF70-KF30	17.5	7.5	75

Figure 3.4 : Fabrication Process of BF-KF/PLA Hybrid Composites

3.5 Physical Characterizations of BF-KF/PLA Hybrid Composites

3.5.1 Density

Density of the composites were determined according to the ASTM D792 standard (Saba et al., 2019). At first, the mass (m) of each sample was measured using a Mettler Toledo XS205 balance (**Figure 3.5**). Samples were then submerged in distilled water to measure the volume (v) of water displaced. Finally, the density (ρ_{exp}) was determined using Equation 3.1. This process was conducted five times at 26 °C to find out the average density.

$$\rho_{exp} = \frac{m}{v} \quad (3.1)$$

Theoretical density is calculated using following Equation 3.2,

$$\rho_{theo} = \frac{1}{\frac{W_{BF}}{\rho_{BF}} + \frac{W_{KF}}{\rho_{KF}} + \frac{W_m}{\rho_m}} \quad (3.2)$$

Here, W_{BF}, W_{KF}, and W_m denote the weight fractions of the bamboo fiber, kenaf fiber, and PLA matrix respectively. The ρ_{BF}, ρ_{KF} and ρ_m corresponds to the densities of the bamboo fiber, kenaf fiber, and PLA matrix respectively.

Figure 3.5 : Density Test Procedure of BF-KF/PLA Hybrid Composites

3.5.2 Void Contents

Void percentages of the composites can be calculated using Equation 3.3 as follows:

$$\text{Volume fraction of void}, V_{void} = \frac{\rho_{theo} - \rho_{exp}}{\rho_{theo}} \times 100 \qquad (3.3)$$

The values of ρ_{exp} and ρ_{theo} are obtained from Equation (3.1) and (3.2) respectively.

3.5.3 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR was performed to detect the functional groups present in the PLA hybrid composites reinforced with bamboo and kenaf fibers. FTIR spectra were obtained using a Thermo Scientific iS10 (USA) spectrometer, with samples measuring 10 mm x 10 mm x 3 mm. The FTIR spectra were observed in the wavelength starting from 4000 cm^{-1} to 500 cm^{-1}.

3.5.4 X-ray Diffraction (XRD)

X-ray diffraction (XRD) test was performed to analyze the crystallinity of the composites using a Rigaku-Tokyo (Japan) 2500 X-ray diffractometer. This instrument was operated at a scan rate of 0.02 (θ) s^{-1}, across an angle scope of 5–60° (2θ), and was calibrated to a voltage of 40 kV and 35 mA current. The crystallinity index (C_i) was calculated by using Equation 3.4.

$$C_i\,(\%) = \frac{A_c}{A_c + A_a} \times 100 \tag{3.4}$$

Here, A_c and A_a denote the area of the crystalline regions and area of the amorphous regions respectively.

3.6 Mechanical Characterizations of BF-KF/PLA Hybrid Composites

3.6.1 Tensile Testing

Universal Testing Machine (INSTRON 3366, USA) was used to perform the tensile tests at ambient temperature as per the ASTM D638-10 standard (Junaedi et al., 2020). Universal Testing Machine (INSTRON 3366, USA-**Figure 3.6**) was used to perform the tests. The test specimens were prepared to the dimensions of 150 mm (L) x 25 mm (W) x 3 mm (T) mm. The crosshead velocity while performing the tensile test was 2.0 mm/ min. Gauge length of the samples were 80 mm.

Figure 3.6 : Tensile Test Procedure of BF-KF/PLA Hybrid Composites

3.6.2 Flexural Testing

Three-point bending test as per the ASTM D 790 standard were performed using INSTRON 3366 (USA-**Figure 3.7**) testing machine (Saharudin et al., 2019). Specimen dimensions were 130 mm (L) x 13 mm (W) x 3 mm (T). The crosshead velocity while performing the flexural test was 1 mm/ min and span length was 48 mm.

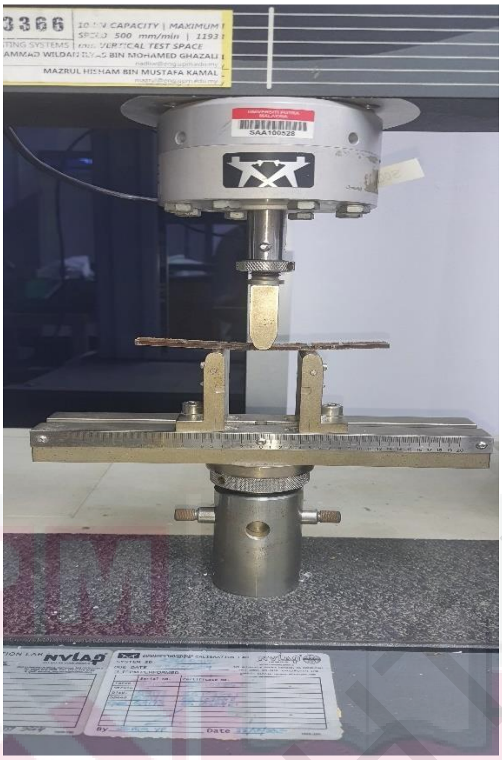

Figure 3.7 : Flexural Test Procedure of BF-KF/PLA Hybrid Composites

3.6.3 Compressive Testing

The compressive tests were conducted using a Universal Testing Machine (INSTRON 3366, USA-**Figure 3.8**) in accordance with the ASTM D695–15 standard (Sherwani et al., 2021a). A loading rate of 2 mm/min was employed, and test samples were cut out to dimensions of 7.94 mm (L) x 1.27 mm (W) x 3.2 mm (T).

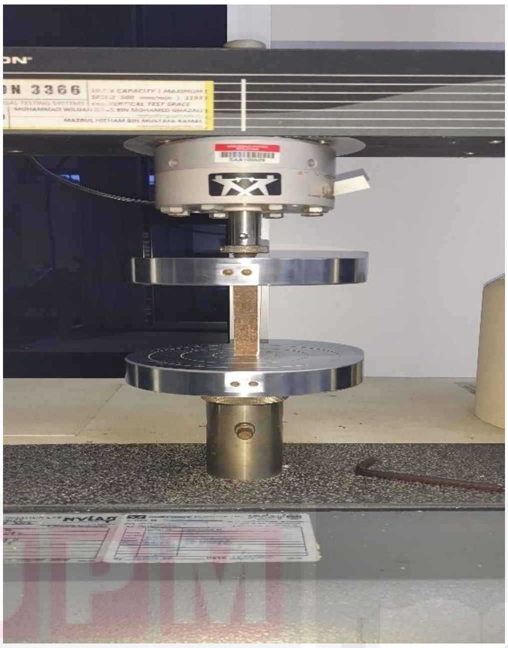

Figure 3.8 : Compressive Test Procedure of BF-KF/PLA Hybrid Composites

3.6.4 Impact Testing

Izod impact tests were conducted following the ASTM D256 (2018) standard, utilizing a Digital INSTRON CEAST 9050 pendulum apparatus (**Figure 3.9**) (Ramnath et al., 2016). Samples were cut out to dimensions of 60 mm (L) x 13 mm (W) x 3 mm (T) and were unnotched. The findings of this test will help to evaluate the material's toughness, demonstrating the capacity to absorb energy under plastic deformation, and highlights the brittle nature of the material when subjected to a sudden load.

Figure 3.9 : Izod Impact Test Apparatus

3.6.5 Hardness Testing

The indentation resistance of the composites was analyzed by Rockwell hardness testing, conducted as per the ASTM D785 standard (Rashid et al., 2017). The testing equipment employed was the Mitutoyo ATK 600 model manufactured by Mitutoyo Corporation (Kanagawa, Japan-**Figure 3.10**). A steel ball with a diameter of half an inch was used on scale S for the test. A force of 100 kilograms was exerted during the testing procedure. The resulting Rockwell hardness values, denoted as HRS, were obtained using a digital scale.

Figure 3.10 : Hardness Test Procedure of BF-KF/PLA Hybrid Composites

3.6.6 Field Emission Scanning Electron Microscope (FESEM)

Microstructural analysis of the tensile fractured samples was conducted in a Field Emission Scanning Electron Microscope (Nova NANOSEM 230, Netherlands) equipment. The equipment operates at a power of 5.0 kV. The FESEM analysis involved varying magnifications between 200x and 2000x. At first, the samples were platinum coated to enhance their electrical conductivity and to enhance the visualization of the surface texture. The coating process lasted for five minutes and was performed using a high vacuum sputter coater (Model-K575X, UK).

3.7 Thermal Characterizations of BF-KF/PLA Hybrid Composites

3.7.1 Thermogravimetric Analysis (TGA)

The thermal decomposition behavior of the bamboo-kenaf/PLA composites were examined through thermogravimetric analysis (TGA) to assess weight reduction as the temperature increase. This analysis utilized a TGA Mettler Toledo apparatus (Malaysia). The test was conducted in a nitrogen atmosphere at a flow rate of 40 mL/min, the temperature was gradually increased from 25 to 900° C at a rate of 10° C/min.

3.7.2 Differential Scanning Calorimetry (DSC)

A Mettler Toledo (Malaysia) differential scanning calorimetry (DSC) apparatus was employed to analyze the thermal characteristics of the bamboo-kenaf/PLA composite materials. The examination involved elevating the temperature of the specimens at a uniform rate of 10° C/min, ranging from 25 to 300° C. The test was conducted within an environment of nitrogen gas flowing at 40 mL/min.

3.8 Water Absorption Characterizations of BF-KF/PLA Hybrid Composites

3.8.1 Water Absorption Test

Five specimens, each measuring 10 mm x 10 mm x 3 mm, were initially dried in an oven with air circulation at 100 ± 2 °C for 24 hours to eliminate any existing moisture. Subsequently, the samples were submerged in regular tap water at ambient temperature (23±1°C) for durations of 0.5 hours and 2 hours

(**Figure 3.11**), following the guidelines from earlier research (Nazrin et al., 2020). The initial weight of the samples was recorded prior to submersion (W_i) and final weight after submission (W_f). The water uptake by the bamboo-kenaf fiber reinforced PLA hybrid composites was determined using Equation 3.5:

$$Water\ absorption\ (\%) = \frac{W_f - W_i}{W_i} \times 100 \tag{3.5}$$

Figure 3.11 : Water Absorption Test of BF-KF/PLA Hybrid Composites

3.8.2 Rainwater Absorption Test

For the rainwater absorption experiment, five specimens each measuring 10 mm x 10 mm x 3 mm were prepared as per previous study (Sherwani et al., 2021b). Initial (W_i) and final (W_f) weights were recorded for the samples before and after they were immersed in rainwater for 48 hours for 7 days at ambient temperature (23±1°C) (**Figure 3.12**). The average from the five specimens was used to determine the rainwater absorption percentage using Equation 3.6:

$$\text{Rainwater absorption } (\%) = \frac{W_f - W_i}{W_i} \times 100 \qquad (3.6)$$

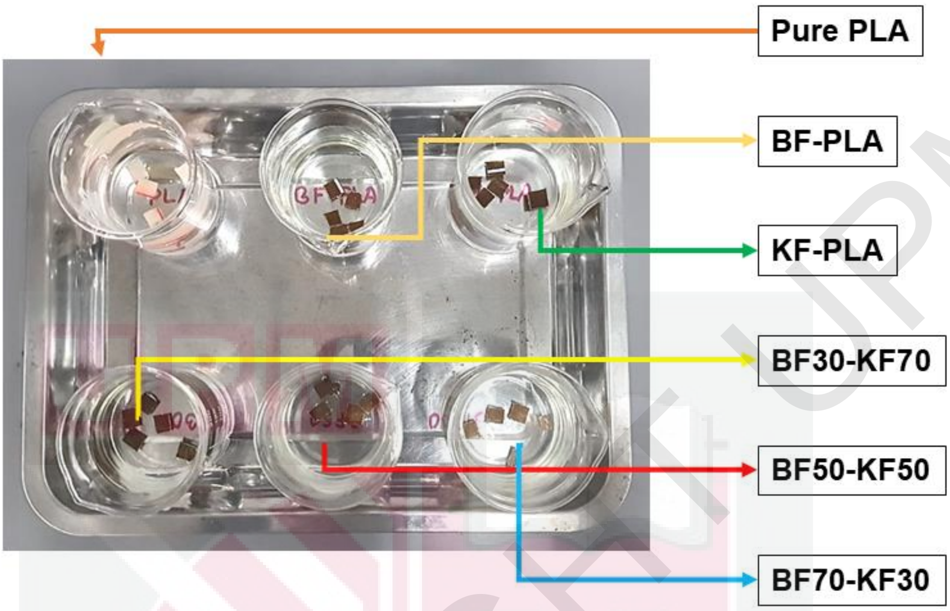

Figure 3.12 : Rainwater Absorption Test of BF-KF/PLA Hybrid Composites

3.8.3 Thickness Swelling Test

Five specimens, each 10 mm x 10 mm x 3 mm, were first dried in a circulating air oven at 100±2°C for 24 hours to eliminate any existing moisture, then soaked in regular tap water maintained at ambient temperature (23±1°C) for 0.5 hours and 2 hours, based on methodologies suggested in prior research (Nazrin et al., 2020a). The thickness swelling of the samples was assessed both before immersion (T_i) and after immersion (T_f) using a digital Vernier caliper (Brand: Duratool, Country: China) with an accuracy of 0.01 cm. The swelling ratio of the thickness for the composite was determined using Equation 3.7:

$$Thickness\ swelling\ (\%) = \frac{T_f - T_i}{T_i} \times 100 \quad (3.7)$$

3.8.4 Water Solubility Test

The water solubility test was performed according to the procedure described by previous studies (Irissin-Mangata et al., 2001; Nazrin et al., 2021b). Before commencing the test, the initial weight (W_1) of each specimen (10 mm × 10 mm × 3 mm) was recorded. The samples were then submerged in regular tap water at 25 °C temperature with constant stirring for 24 hours (**Figure 3.13**). Following this period, the insoluble parts of the samples were dried at 100 °C for an additional 24 hours. The dried insoluble remains were then weighed (W_2). The experiment was replicated three times for each sample. The solubility of each composite blend was determined using Equation 3.8:

$$Water\ solubility\ (\%) = \frac{W_1 - W_2}{W_1} \times 100 \quad (3.8)$$

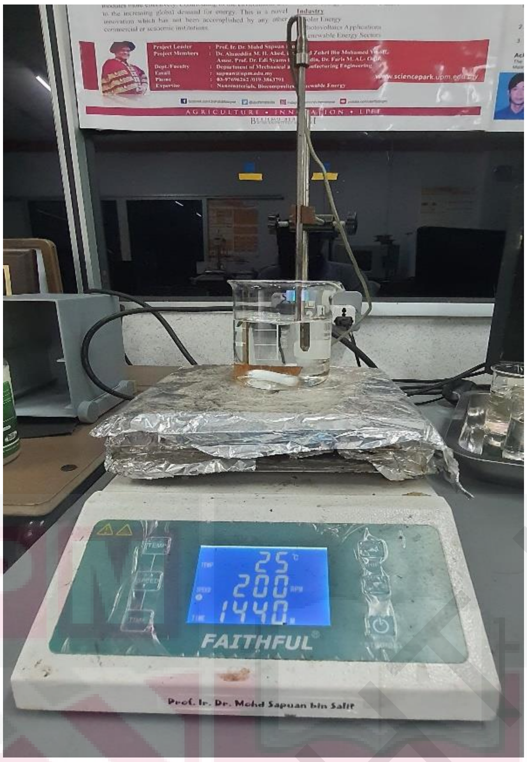

Figure 3.13 : Water Solubility Test Of BF-KF/PLA Hybrid Composites

3.8.5 Water Vapor Permeability (WVP) Test

The measurement of water vapor permeability was conducted following the ASTM E96-95 standard (ASTM, 2016). Prior to the test, the samples underwent conditioning in a desiccator, adhering to 53 ± 1% relative humidity (RH) and an ambient temperature of 23 ± 2°C. The procedure was carried out three times for consistency. Initially, 10 grams of silica gel were placed into the base of a cylindrical cup with a diameter of 20 mm (**Figure 3.14**). The samples, prepared in circular shapes, were then affixed to the tops of these cups, ensuring a 3 mm gap from the upper edge. The initial weights of these cups were carefully recorded before they were positioned in a controlled humidity environment (25°C, 75% RH). The weights were periodically monitored until an equilibrium state was achieved. WVP was determined from the weight gain of the permeation, measuring over 24 h for 7 days. Upon reaching equilibrium, the final weights were recorded and utilized to calculate the water vapor permeability (WVP), as detailed in the corresponding Equation 3.9.

$$Water\ vapour\ permeability\ (\%) = [(m \times d)/(A \times t \times P)] \quad (3.9)$$

Here the increased weight of the test cup is represented by m in grams, the thickness of the sample is denoted as d in millimeters, the area of the sample exposed is A in m^2, the time interval for permeation is t in seconds, and the partial pressure difference across the sample is P in Pascals.

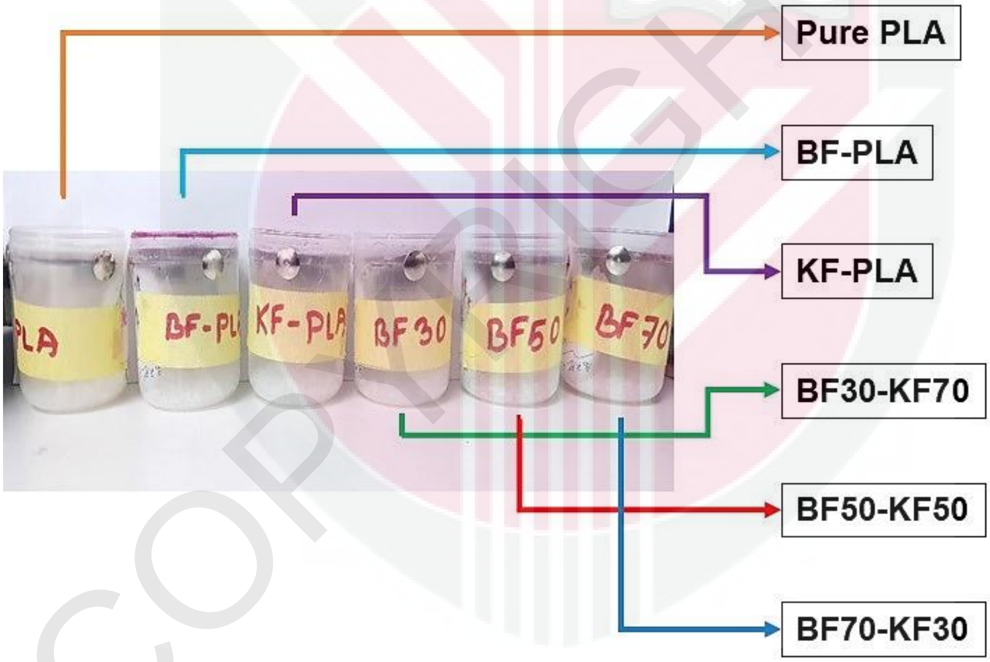

Figure 3.14 : Water Vapor Permeability Test of BF-KF/PLA Hybrid Composites

3.9 Surface Wettability Characterizations of BF-KF/PLA Hybrid Composites

3.9.1 Water Contact Angle Test

The surface wettability was measured following the guidelines of ASTM D7334-08 (ASTM 2008) on Theta Lite Optical Tensiometer instrument (TL100, Biolin Scientific-**Figure 3.15**) (Redzuan et al., 2019). Contact angle measurements were conducted using 7 μL droplets of distilled water at a temperature of 20°C. The testing room maintained a relative humidity of 65±5%. Measurements continued until no water droplets remained on the composites surface.

Figure 3.15 : Water Contact Angle Test Apparatus

3.10 Flammability Characterizations of BF-KF/PLA Hybrid Composites

3.10.1 UL-94 (Vertical) Test

The UL-94 vertical tests were carried out with a vertical burning tester (**Figure 3.16**). The specimens used were 125 mm × 13 mm × 3 mm. Each sample undergoes initial ignition for ten seconds, followed by extinguishing the flame, and then a second ignition for another ten seconds. The duration until the flame extinguishes is recorded, and any burning chars or liquid droplets falling are observed. This procedure was repeated five times for each sample.

Figure 3.16 : UL-94 (Vertical) Test Apparatus

3.10.2 Limiting Oxygen Index (LOI) Test

The LOIs of the specimens were evaluated in accordance with ASTM D2863 (Chee et al., 2020). Specimens were prepared, each measuring 75 mm × 10 mm × 3 mm. The specimens were ignited like candles (ignition from the top surface) as depicted in **Figure 3.17**. For each group of composites, consisting of 15 specimens, testing continued until the last 5 specimens showed a deviation of 0.15 vol% in oxygen concentration. The final LOI was calculated based on the burning pattern of these last 5 test specimens.

Figure 3.17 : Experimental Setup of LOI Test for BF-KF/PLA Hybrid Composites

3.11 Summary

This chapter outlines the materials, treatments, fabrication techniques, and characterization methods employed to develop and analyze BF-KF/PLA hybrid composites. The process begins with the preparation of raw materials, followed by an alkali treatment to improve fiber-matrix adhesion. The treated fibers are then used to fabricate hybrid composites using twin-screw extrusion and compression molding techniques.

As highlighted in Table 3.2 the physical properties of the composites, including density, void content, crystallinity (via XRD), and chemical interactions (via

FTIR), were characterized. Mechanical properties such as tensile, flexural, compressive strength, impact resistance, and hardness were assessed. Thermal properties were evaluated through thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC). Additionally, the composites' water absorption, surface wettability, and flammability were tested to ensure their suitability for eco-friendly applications, particularly in the furniture industry.

Table 3.2 : Summary of Characterizations Performed for BF-KF/PLA Hybrid Composites

Characterizations					
Objective 1			Objective 2		
Physical	Mechanical	Thermal	Water Absoprtion	Surface Wettability	Flammability
Density	Tensile	TGA	Water absorption	Water contact angle	UL-94 (vertical)
Void content	Flexural	DSC	Rainwater absorption		LOI
Crystallinity (XRD)	Compression		Thickness swelling		
FTIR	Impact		Water solubility		
	Hardness		Water vapor permeability		

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter contains the detail results and discussion of the all objectives of the research. The chapter is divided into six parts:

1. Physical properties of BF-KF/PLA hybrid composites.
2. Mechanical properties of BF-KF/PLA hybrid composites.
3. Thermal properties of BF-KF/PLA hybrid composites.
4. Water absorption properties of BF-KF/PLA hybrid composites
5. Surface wettability properties of BF-KF/PLA hybrid composites
6. Flammability properties of BF-KF/PLA hybrid composites

4.2 Physical Properties of BF-KF/PLA Hybrid Composites

4.2.1 Density Analysis

Density analysis offers significant insights into the dimensional stability of natural fiber reinforced composites (Kerni et al., 2020). As evidenced by **Figure 4.1**, the BF-KF/PLA hybrid composites are classified as low-density composites (less than 500 g/cm³) because of the lightweight nature of both the fibers and the PLA polymer. The incorporation of bamboo fiber into PLA (BF-PLA) results in a reduction of density to 1.1826 g/cm³. But with the addition of kenaf fiber (KF-PLA), the density is increased to 1.2479 g/cm³ as the density of the kenaf fibers in this study is higher compared to bamboo fibers. In the case of hybrid composites, the low-density bamboo fibers reduce the overall density, as observed in the BF70-KF30 hybrid composite (1.1505 g/cm³). Conversely, BF30-KF70, which has a higher kenaf percentage, exhibits a higher density of 1.2471 g/cm³, indicating that kenaf fiber contributes to an increase in the density of the composites, as evidenced in other studies (Hassan et al., 2022; Tawakkal et al., 2012). The lower density of composites with higher bamboo fiber reinforcement could be particularly beneficial for plastic furniture, where lower weight can enhance portability and ease of assembly.

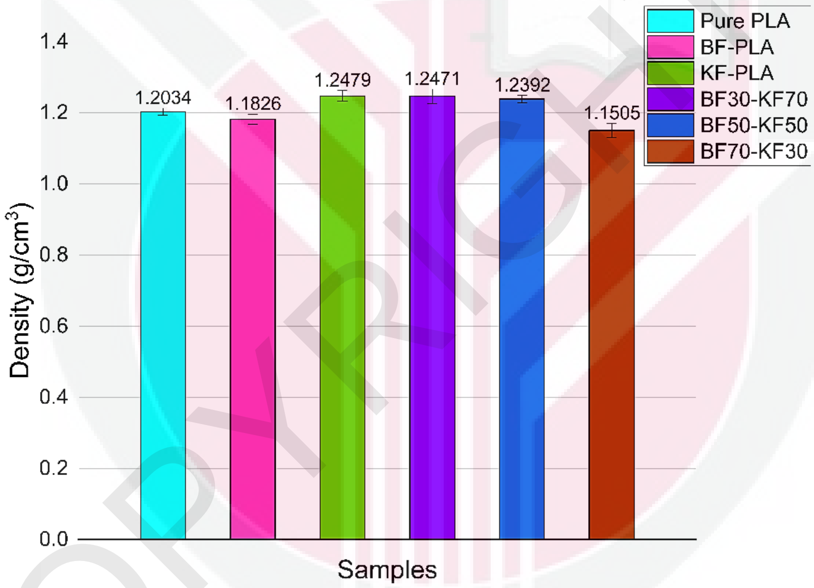

Figure 4.1 : Density Data of BF-KF/PLA Hybrid Composites

4.2.2 Void Content Analysis

Table 4.1 shows the experimental density, theoretical density and void content of the BF-KF/PLA hybrid composites. The experimental densities of all the composites are lower compared to the theoretical densities, likely due to voids forming during the fabrication process (Chee et al., 2020). Low-density composites tend to form voids or empty spaces within their structure which reduce the interfacial bonding between fibers and the matrix, leading to weaker mechanical properties (Taib & Julkapli, 2019). The void content arises because the polymeric phase is unable to effectively displace the trapped air within the composites (Yahaya et al., 2015). Additionally, the increase in void content was caused by the matrix's incomplete wetting of the fibers (Ismail et al., 2019). The results showed that pure PLA had a void content of 2.95%, likely due to the bubble generation during the extrusion and compression molding processes (Betanzos et al., 2016). The incorporation of bamboo and kenaf fibers resulted in a lower void content indicating denser packing of the composites. The lowest void content of 0.27% was observed for the 50:50 blend of bamboo and kenaf (BF50-KF50) which co-relates with the finding of Ismail et al. (Ismail et al., 2019). The highest void content in BF70-KF30 composite suggest poor interfacial adhesion between the fibers and polymer matrix.

Table 4.1 : Void Contents of the BF-KF/PLA Hybrid Composites

Sample	ρ_{theo}	ρ_{exp}	Void contents, V_{void}
Pure PLA	1.24	1.2034	2.95%
BF-PLA	1.205	1.1826	1.86%
KF-PLA	1.2755	1.2479	2.16%
BF30-KF70	1.2575	1.2471	0.83%
BF50-KF50	1.2425	1.2392	0.27%
BF70-KF30	1.2275	1.1505	6.27%

4.2.3 FTIR Analysis

The FTIR results were examined to detect the physical and chemical connections between PLA, bamboo, and kenaf fibers. The FTIR spectrum curves of the pure PLA and PLA composites reinforced with bamboo and kenaf fibers are shown in **Figure 4.2**. The pure PLA and fiber reinforced PLA composites demonstrated quite similar peaks as evidenced by the FTIR spectrum. All the fiber reinforced composite samples contain O–H groups of the cellulosic bamboo and kenaf fibers, demonstrated by wide absorption bands between 3200 cm^{-1} and 3500 cm^{-1} (Prabhudass et al., 2022). The characteristic stretching band for asymmetric and symmetric stretching of C–H of ethyl and ethylene groups in the pure PLA and all bamboo-kenaf fibers composites can be observed between 3050 cm^{-1} and 2830 cm^{-1} (Beniwal & Toor, 2024). The distinct absorption peak detected at 1745 cm^{-1} correspond to the stretching vibration of the –C=O of the ester group available in PLA (Nazrin et al., 2020). The peak observed at 1452 cm^{-1} and the minor band near 1365 cm^{-1} are linked to the stretching of CH_3 and the deformation of C-H bonds, respectively (Akindoyo et al., 2018). The sharp peaks at 1180 cm^{-1}, 1080 cm^{-1} and 1040 cm^{-1} indicate the presence of C-O bonds which is present in the chemical structure of maleic anhydride (Angin et al., 2023). The peak representing hydrocarbon bonds was identified at 865 cm^{-1} (Sánchez et al., 2019).

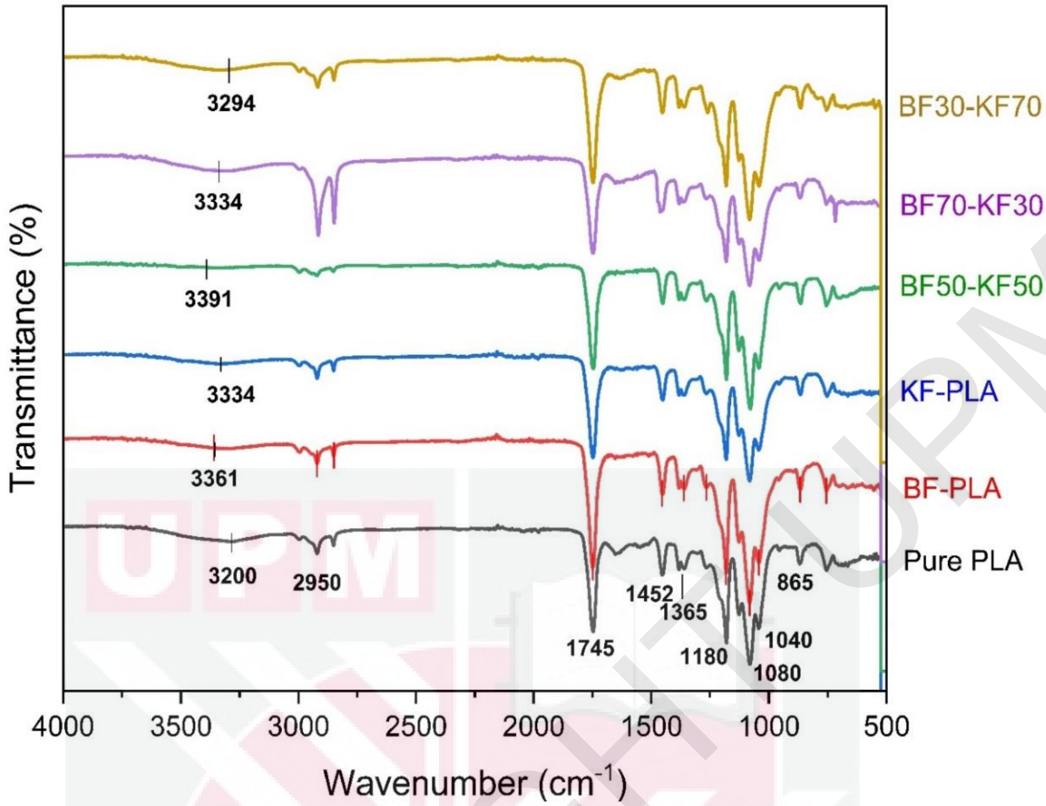

Figure 4.2 : FTIR Peaks of BF-KF/PLA Hybrid Composites

4.2.4 XRD Analysis

X-ray beams scatter in various directions when they encounter the crystal plane, allowing for the analysis of angles, crystal orientation, proportion of crystalline phases, and the strength of diffracted rays through XRD techniques (Lee, 2017). **Figure 4.3** demonstrates the diffraction patterns of both pure PLA and PLA composites reinforced with bamboo-kenaf fibers**.** Broad hump seen in the lower angle region (below 20°) across all samples is characteristic of the amorphous phase in polymers. This suggests that the semi-crystalline PLA polymer within the mixture predominantly displayed an amorphous nature (Mittal et al., 2015). This is associated with absence of a distinct crystalline peak typically linked to the (211) / (110) crystallographic plane (Bhasney et al., 2017). Typically, the pure PLA crystallization process is too slow to yield notable crystallinity, particularly under non-isothermal conditions (Zhai et al., 2009). In all the composite samples, sharp crystalline peak was observed at 21.5^0 corresponding to the (002) crystal plane (Kumar & Tumu, 2019). The intensity of the peak is highest in the BF70-KF30 sample which indicates a higher perfection of the crystal lattice. Das & Chakraborty (Das & Chakraborty, 2008) reported that alkali treatment of bamboo fiber led to the formation of new hydrogen bonds between cellulose chains. This phenomenon resulted in a tighter packing of cellulose chains, thereby increasing the observed crystallinity index. Pure PLA, BF-PLA and BF70-KF30 samples showed peak at 23.8^0 which is attributed to the (220) crystal planes (Cui et al., 2022). However, this peak is not visible in KF-PLA, BF50-KF50 and BF30-KF70 samples. In terms of crystallinity percentage, a trend has been observed that when the bamboo fiber percentage increases the crystallinity percentage also increases as evidenced by **Table 4.2**. The lower crystallinity observed in composites containing higher percentage of kenaf may be attributed to the higher hemicellulose content typically found in kenaf fibers compared to bamboo fibers (Malik et al., 2021). Hemicellulose tends to hinder the crystallization process of PLA by restricting polymer chain mobility and hindering the formation of nucleation sites. Unlike cellulose, which forms strong hydrogen bonds and crystalline regions, hemicellulose introduces more amorphous regions (Rao et al., 2023). Therefore, reduced crystallinity is observed in kenaf fiber reinforced composites. For potential applications in plastic furniture and consumer products, these findings suggest that composites with higher bamboo fiber content and increased crystallinity are likely to offer better mechanical properties and durability.

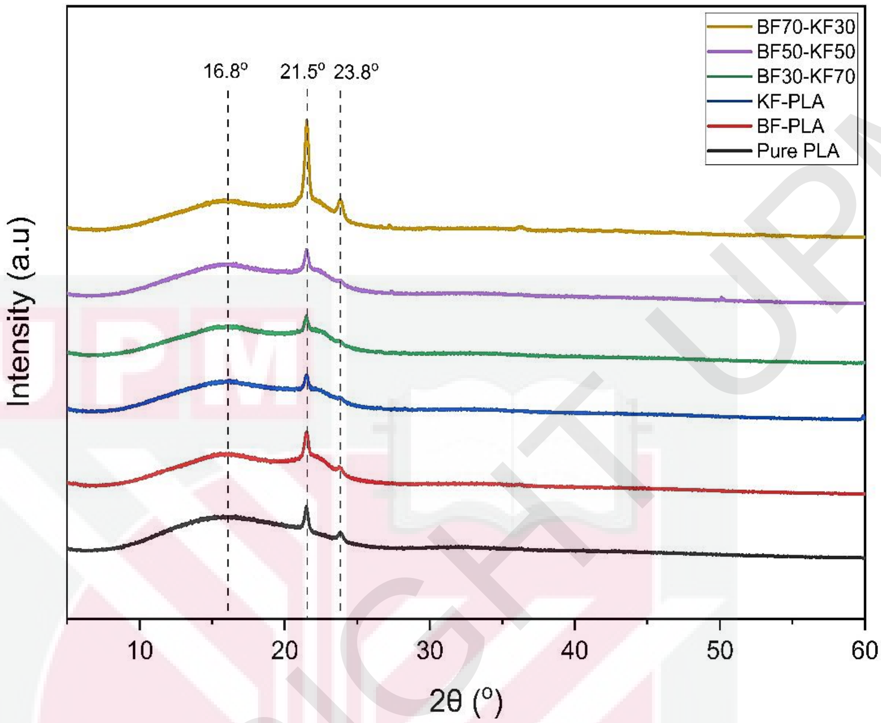

Figure 4.3 : XRD Patterns of BF-KF/PLA Hybrid Composites

Table 4.2 : Crystallinity Percentages of the Bamboo-Kenaf Fiber Reinforced PLA Hybrid Composites

Samples	**Crystallinity index, C_i (%)**
Pure PLA	47.87
BF-PLA	46.56
KF-PLA	22.05
BF30-KF70	23.78
BF50-KF50	32.17
BF70-KF30	38.05

4.3 Mechanical Properties of BF-KF/PLA Hybrid Composites

4.3.1 Tensile Properties Analysis

Tensile characteristics of composites are regulated by loading percentage of fiber, state of dispersion, structure of fiber, available functional groups, and fiber/matrix bonding (Ilyas et al., 2022; Safwan et al., 2018). Tensile strength values depicted in **Figure 4.4a and 4.4b** shows Pure PLA exhibited a tensile strength of 24.51 MPa and a Young's modulus of 610.13 MPa. The addition of bamboo fibers (BF-PLA) resulted in a slight increase in tensile strength to 25.95 MPa, while significantly enhancing the Young's modulus to 2199.15 MPa. This improvement in tensile strength and modulus suggests strong interfacial bonding between the alkali-treated bamboo fibers and the PLA matrix (Wang et al., 2018a). In contrast, the kenaf fiber composite (KF-PLA) showed a decrease in tensile strength to 8.45 MPa but still improved the Young's modulus to 1341.27 MPa. The higher tensile strength of bamboo composites showcases the superior strength of bamboo fibers compared to kenaf (Ismail et al., 2019). Among the hybrid composites, the BF50-KF50 composition demonstrated the most balanced properties, with a tensile strength of 17.82 MPa and a Young's modulus of 1980 MPa. The enhanced Young's modulus for all the fiber reinforced samples, indicate that these composites are stiffer and more resistant to elastic deformation than the pure PLA (Ilyas et al., 2022).

As evident from **Figure 4.4c**, the inclusion of bamboo and kenaf fibers reduces the elongation at break of PLA matrix hinting towards the stiff nature of the fiber reinforced composites. The lower elongation at break may be attributed to the micro-sized bamboo and kenaf fibers providing higher aspect ratio. In composites with high-aspect-ratio fibrils, the large interfacial area between the fibrils and the matrix enhances their contact. During elongation, the matrix exerts pressure on the fibrils, creating frictional forces that resist deformation. This resistance limits the formation of cavities (cavitation) and results in lower small elongations at break (Kuzmanović et al., 2020). The stress-strain curve illustrated in **Figure 4.4d** supports the observation of lower elongation at break for the fiber-reinforced composites. Apparently, all the curves exhibit linear behavior until the composites fail. The pure PLA sample demonstrate the highest strain and slightly less stress compared to BF-PLA. Therefore, the fiber-reinforced composites exhibit more brittle nature than the pure PLA.

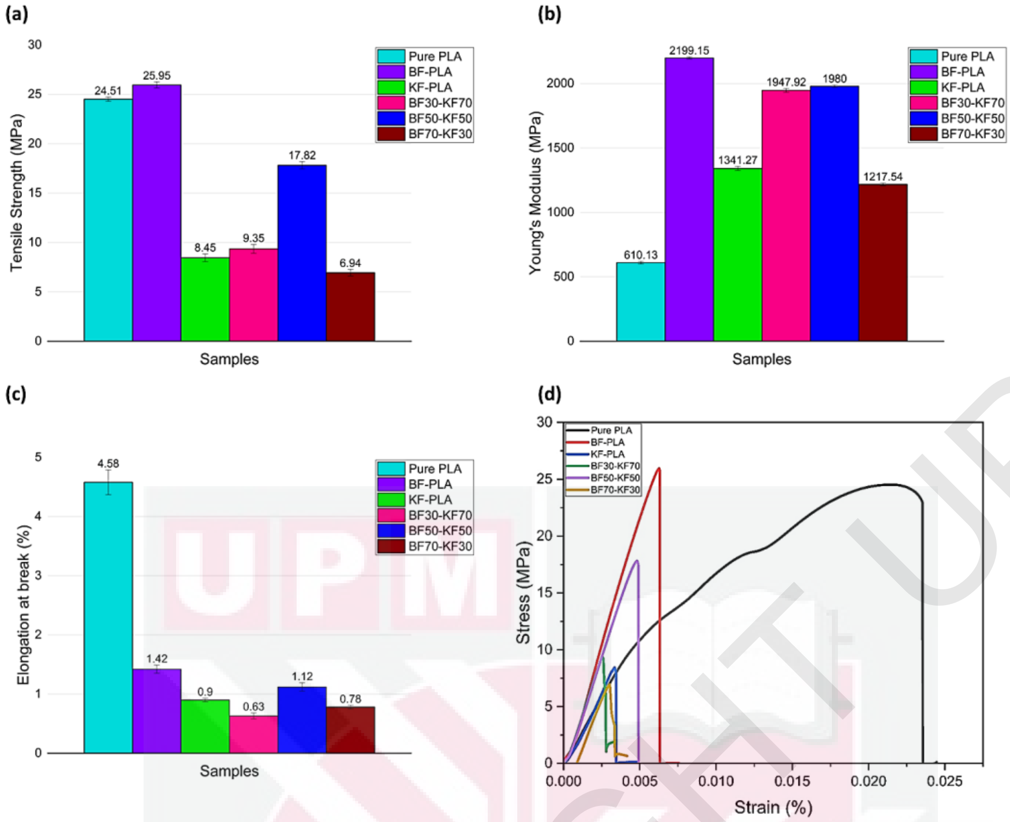

Figure 4.4 : (a) Tensile Strength, (b) Young's Modulus, (c) Elongation at Break and (d) Stress-Strain Curve Data of BF-KF/PLA Hybrid Composites

4.3.2 Flexural Properties Analysis

Flexural strength of a material is defined as the highest bending stress needed to fracture a beam when subjected to a three-point bending test (Prabhudass et al., 2022). **Figure 4.5a** shows the flexural strength data where pure PLA exhibited the topmost flexural strength at 71.09 MPa. However, a decrease in flexural strength was observed when PLA was reinforced with bamboo-kenaf fibers. BF-PLA sample recorded a second-best flexural strength of 45.53 MPa, indicating a better compatibility with PLA compared to kenaf-PLA composites, which demonstrated a significantly lower strength of 27.46 MPa. This reduction in flexural strength in the fiber-reinforced composites may be attributed to the irregular shapes of the ground bamboo and kenaf fibers. The irregular shape of the fibers hinders effective stress transfer between the fibers and the polymer matrix. Additionally, the weak interfacial adhesion between the fibers and the matrix contributes to the formation of voids (Lee et al., 2021). These voids act as stress concentrators and potential crack initiation sites, weakening the overall structure (Yussuf et al., 2010). These factors together lead to a mechanically weaker composite under bending load. The BF50-KF50 composite exhibited a flexural strength of 33.18 MPa, surpassing kenaf-PLA but not reaching the strength of bamboo-PLA, implying a partial compensatory effect. BF70-KF30 sample exhibited slightly lower flexural strength than the BF30-KF70 sample. A possible reason for this might be the higher kenaf content in the BF30-KF70 sample as the cellulose percentage in kenaf fibers is higher than bamboo fibers (Chokshi et al., 2022). This fact aligns with the general observation that plant fibers with more cellulose typically exhibit enhanced mechanical performances (Bismarck et al., 2005).

Flexural modulus is the ratio of stress to strain within a material's linear elastic region. It indicates the material's stiffness or resistance to bending when subjected to a three-point bending flexural test (Chee et al., 2021). The flexural modulus data in **Figure 4.5b** reveals that while BF-PLA has a lower flexural strength compared to pure PLA, it has a significantly higher flexural modulus. Ochi (Ochi, 2015) also reported that bamboo fiber reinforced PLA composites have higher flexural modulus. This indicates that although bamboo fibers reduce the maximum stress the composite can handle before failing, they greatly increase its stiffness. In contrast to bamboo fibers, kenaf fibers provide a minimal enhancement in stiffness. The hybrid composites (BF50-KF50, BF70-KF30, BF30-KF70) show a trend where the flexural modulus does not always increase with the flexural strength. For instance, the BF30-KF70 composite has a higher flexural strength than the BF70-KF30 composite, yet its flexural modulus is lower. This suggests that the BF30-KF70 composite is stiffer and resists bending better, but it is not as strong as the BF70-KF30 composite in terms of the maximum stress to withstand before failing.

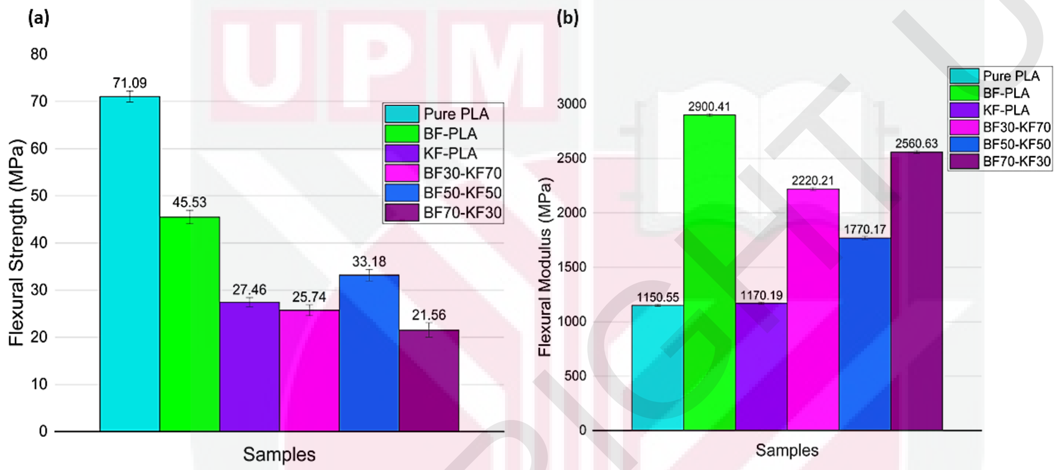

Figure 4.5 : (a) Flexural Strength and (b) Modulus Data of BF-KF/PLA Hybrid Composites

4.3.3 Compressive Strength Analysis

The reinforcement by bamboo and kenaf fibers in PLA matrix has significantly increases compressive strength, as seen with BF-PLA, KF-PLA and BF30-KF70 (**Figure 4.6)**. Notably, BF-PLA has the highest compressive strengths of 173.15 MPa. According to Rao et al. (Rao et al., 2010) the higher compressive strength of bamboo fiber may be attributed to the higher crystallinity achieved by removal of amorphous hemi-cellulose with alkali treatment. The data suggests that both bamboo and kenaf fibers are effective at reinforcing PLA, but their benefits are not simply additive, as seen in the hybrid samples. The hybrid samples BF50-KF50, BF70-KF30, and BF30-KF70 exhibit a range of compressive strengths. The BF30-KF70 shows second best compressive strength of 169.03 MPa indicating the best ratio of bamboo and kenaf fibers for hybridization when comes to compressive loading. The other two combination (BF50-KF50 and BF70-KF30) of hybrid samples showed poor compressive strength. The reason behind the poor strength may be attributed to the misalignment of fibers during the compression molding process. Fiber misalignment caused by limitation of production process is regarded as a primary factor contributing to compressive damage in composites (Yuan et al., 2019). Misaligned fibers can cause localized stress concentrations, leading to premature failure via buckling, or matrix cracking (Wilhelmsson et al., 2019). Furthermore, the misalignment of fibers reduces the extent of the elastic region by limiting their load-bearing capacity, while inducing the formation of a longer plastic region where fibers undergo buckling and kink-band formation (Bhowmick et al., 2012). This extended plastic region further compromises the composite's ability to resist compressive loads effectively.

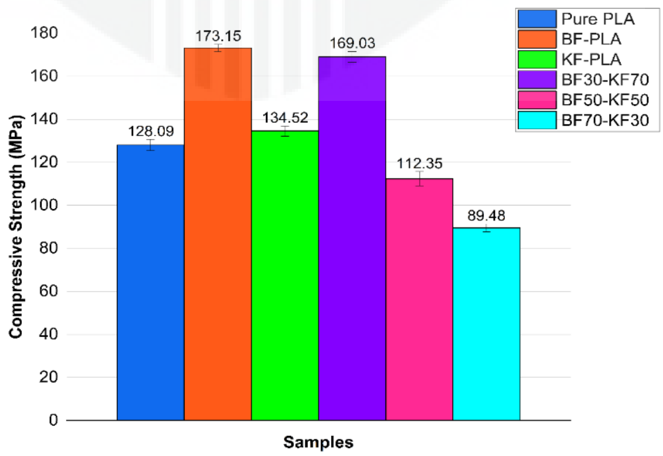

Figure 4.6 : Compressive Strength Data of BF-KF/PLA Hybrid Composites

4.3.4 Impact Properties Analysis

Izod's impact test offers valuable insights into the composites' ability to withstand sudden impacts or loads (Prabhudass et al., 2022). By measuring the energy absorbed during fracture, this test helps determine the material's toughness and resistance to impact, which is crucial for applications where durability and resilience are essential. **Figure 4.7** illustrates the fluctuations in impact strength of the PLA composites as bamboo and kenaf fiber reinforcements are applied. The BF30-KF70 hybrid sample exhibited the maximum impact strength at 6.6 Nm, which is approximately 17.86% greater than that of the pure PLA. Significant improvement in impact strength was noted also for the other hybrid and non-hybrid configurations. This enhancement in impact strength is likely to the alkali treatment on the fibers, which eliminated a significant proportion of lignin, wax, hemicellulose, pectin, and various surface impurities (Zahraoui et al., 2023). Another possible reason could be the reduction of distance between the particles or the thickness of the PLA matrix ligaments with the addition of bamboo and kenaf fibers (Shirvanimoghaddam et al., 2021). Therefore, the fiber incorporation influenced the transition of PLA matrix from brittle to tough. Moreover, the increased surface area provided by the micro sized bamboo and kenaf fibers help to improve the impact strength of PLA composites by toughening mechanisms such as stress concentration or shear yielding (Nadzri et al., 2020).

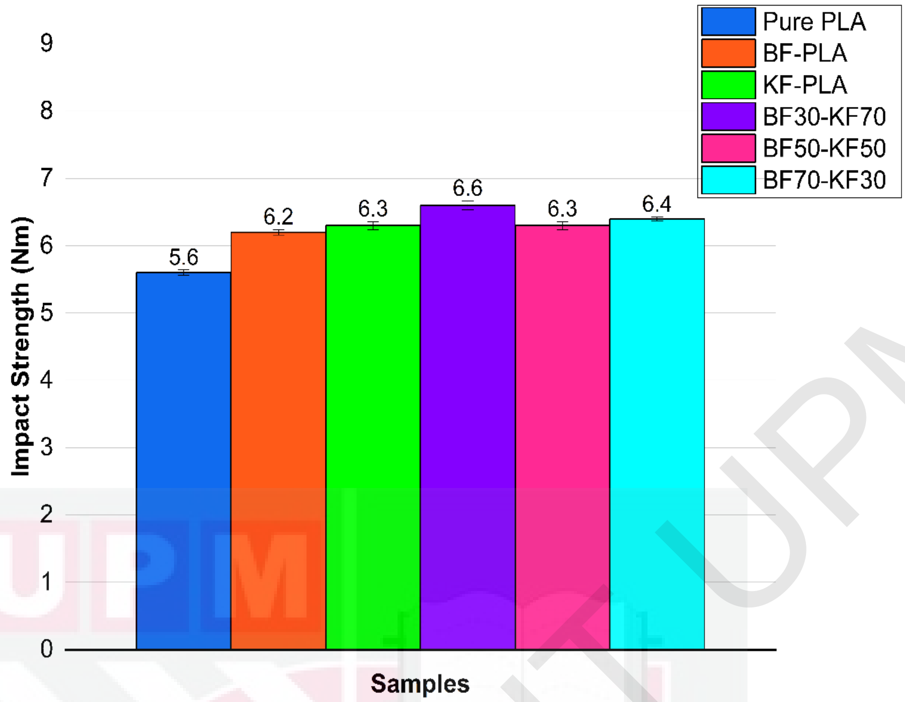

Figure 4.7 : Impact Strength Data of BF-KF/PLA Composites

4.3.5 Hardness Properties Analysis

Hardness is the quality or condition of being hard, measured by a material's resistance to various kinds of permanent shape change when a compressive force is applied (Ashok et al., 2018). It serves as a metric for a material's capacity to withstand surface indentation. **Figure 4.8** shows the hardness resistance values of pure PLA, non-hybrid and hybrid combinations of the composites. The hardness resistance value of the KF-PLA (69.2 HRS) is the highest among all the samples tested suggesting that the addition of kenaf fibers reduce the flexibility of PLA matrix. The possible reason of reduced flexibility may be attributed to the stiff nature of the kenaf fiber. Stiffness of kenaf fiber comes from the cellulose content which is higher compared to bamboo fiber (Akil et al., 2011; Ramesh, 2016). Among the hybrid samples, the BF50-KF50 sample showed the highest hardness value of 55.8 HRS which is 18.27% higher than the pure PLA. Only the BF70-KF30 hybrid sample showed lower hardness value than the pure PLA. The lower hardness value observed can be attributed to the indenter's inability to evenly compress a uniformly packed surface (Butt et al., 2020). This is influenced by surface irregularities or imperfections and variations in material composition near the surface of the sample. Since hardness values indicate wear resistance, harder materials are more effective at resisting friction and wear (Zainudin & Sapuan, 2009). Therefore, the BF-PLA, KF-PLA and BF50-KF50 have potential to be used as surface material in furniture or consumer products.

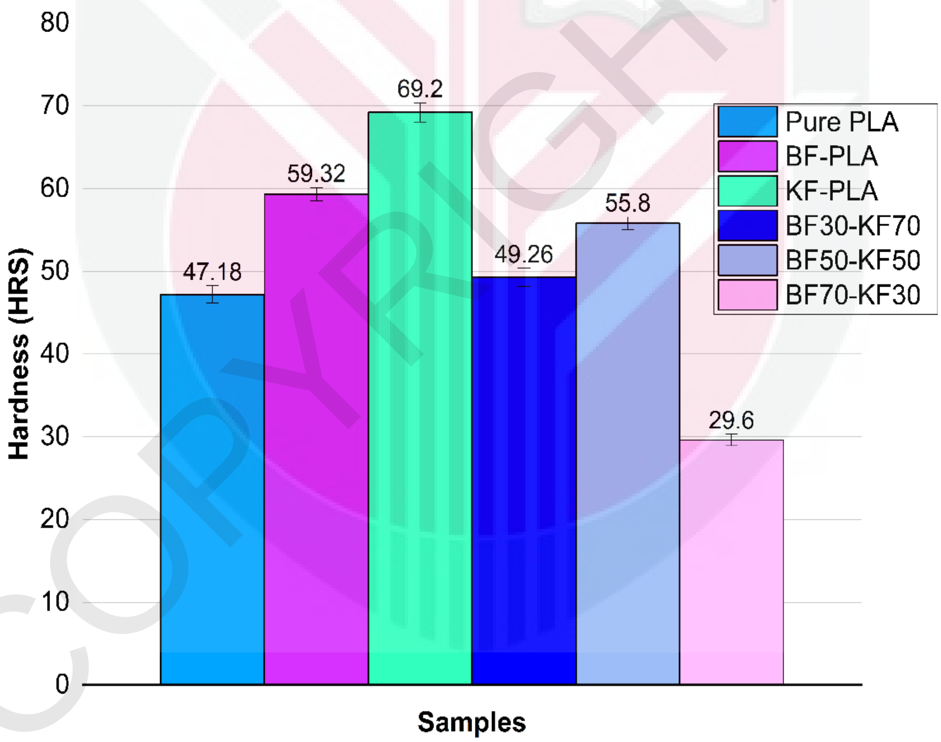

Figure 4.8 : Hardness Tests Data of BF-KF/PLA Hybrid Composites

4.3.6 FESEM Analysis

The tensile fractured samples were examined to reveal their microstructural characteristics. Tensile test results of the composites rely on the compatibility of bamboo and kenaf fibers within the PLA matrix. Microstructural analysis provides insights into both the adhesion at the interface between the fibers and the matrix, as well as the distribution of the fibers within the matrix (Olonisakin et al., 2022). The FESEM image of the tensile fractured pure PLA sample, depicted in **Figure 4.9a**, showcased a consistently fractured surface. But had some ridges can be observed indicating the material's brittle characteristics which is reported by previous studies (Hassan et al., 2020; Rasheed et al., 2021). **Figure 4.9b** shows that bamboo fibers are uniformly dispersed within the PLA matrix, indicating good interfacial compatibility. Alkaline treatment and action of maleic anhydride as the compatibilizer improve the interfacial bonding between bamboo fiber and PLA as reported by Liu et al. (Liu et al., 2012). Hence, the BF-PLA sample showed better tensile strength than the rest of the fiber reinforced samples. Poor tensile strength for KF-PLA sample is due to the weak interfacial bonding as evidenced from **Figure 4.9c.** Poor interfacial bonding hinders the transmission of stress from the polymer matrix to the fibers, resulting in reduced strength (Chung et al., 2018). This is evidenced by lower tensile strength observed during the mechanical testing of the KF-PLA composites. The BF50-KF50 hybrid composites showed better dispersion and interfacial fiber-matrix bonding. However, voids are observed as depicted in **Figure 4.9e**. Voids are areas within the polymer matrix where fibers fail to bond with the polymer, and a greater occurrence of these voids can significantly reduce the mechanical strength of the composites (Mehdikhani et al., 2019). The BF30-KF70 and BF70-KF30 hybrid samples contain debonding, fiber pullout, voids and agglomerated fibers as depicted in **Figure 4.9d** and **Figure 4.9f** respectively.

Teklal et al. (Teklal et al., 2018) reported that the pulling out of fibers from the matrix reduces the overall strength of the composite. To sum up, the FESEM results justify the tensile performances of the composites observed in the testing.

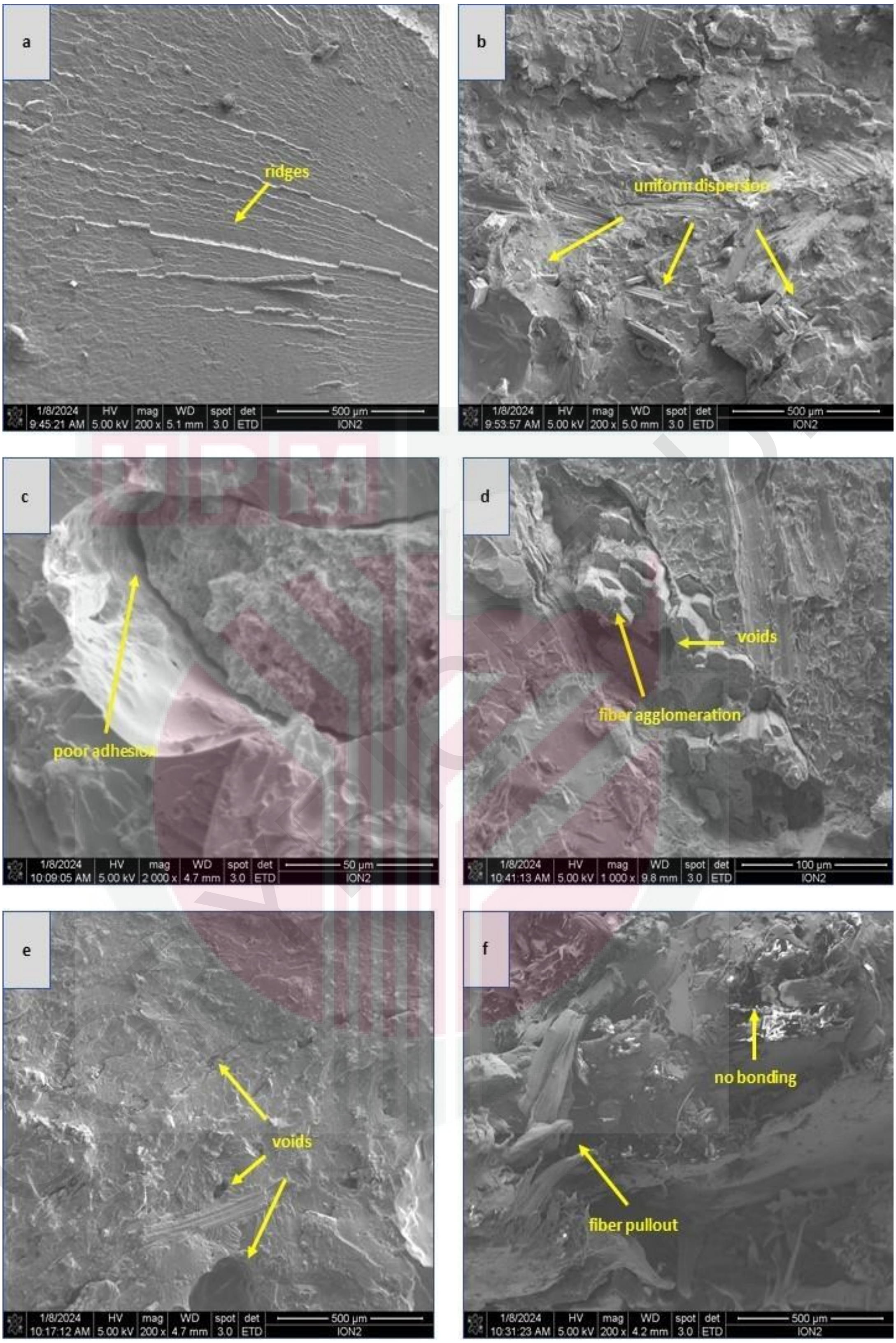

Figure 4.9 : FESEM Figures Showing a) Pure PLA, b) Bamboo-PLA, c) Kenaf-PLA, d) BF30-KF70 Hybrid, e) BF50-KF50 Hybrid and e) BF70-KF30 Hybrid Composites

4.4 Thermal Properties of BF-KF/PLA Hybrid Composites

4.4.1 TGA Analysis

The impact of incorporating varying amounts of bamboo and kenaf fibers on the thermal stability of PLA polymer was assessed. Degradation and decomposition behavior of polymer when exposed to heat reveal crucial data to enhance the processing properties (Pielichowski et al., 2022). **Table 4.3** represents the temperatures corresponding to 5%, 25%, 50%, and 75% mass reductions as well as the peak temperature of the maximum weight loss for the material. At 5% weight loss, the degradation temperature of the bamboo and kenaf fibers reinforced samples drop. The incorporation of bamboo and kenaf fibers to PLA led to higher the molecular mobility and decomposition of some other molecular chains. As a result, crystalization was developed due to plasticization and heterogeneous nucleation and consequently a reduction in the thermal degradation temperature occurred (Qian et al., 2016). The maximum degradation rate displayed a varying temperature with BF30-KF70 having the highest value (484 °C) followed by BF70-KF30 (482 °C), KF-PLA (479 °C), pure PLA (477 °C), BF-PLA and BF50-KF50 (470 °C). The char residue significantly increased as the bamboo and kenaf fibers were incorporated since both of these fibers are associated with producing a higher yield of charred products compared to PLA (Jawaid et al., 2022).

Table 4.3 : Thermal Degradation Temperatures of BF-KF/PLA Hybrid Composites

Samples	$T_{5\%}$ (°C)	$T_{25\%}$ (°C)	$T_{50\%}$ (°C)	$T_{75\%}$ (°C)	T_{max} (°C)	Char residue (%)
Pure PLA	306	341	354	364	477	0.194
BF-PLA	269	329	341	349	470	4.833
KF-PLA	266	324	336	346	479	5.443
BF30-KF70	264	322	334	355	484	4.736
BF50-KF50	266	324	336	346	470	3.435
BF70-KF30	295	321	332	443	482	3.783

The particular weight reduction depicted in the TGA curves (**Figure 4.10)** and distinct peak observed in the DTG curves (**Figure 4.11**) represent each stage of thermal degradation. The weight loss of the composites under thermal conditions occurs in three phases. The initial phase of weight reduction occurs prior to 300 °C, primarily due to the evaporation of water (Zou et al., 2022). Here the rate of weight loss in composites vary slightly across different bamboo and kenaf fibers concentrations. At approximately 100 °C, minor weight reductions were noted in the TGA graphs, which is likely due to moisture evaporating as the samples undergo the drying process (Dehbari et al., 2014). The second phase of weight loss takes place between 300 °C to 350 °C, representing the main thermal decomposition phase of the composites. The peak loss of mass can be attributed to the breakdown of hemicellulose and cellulose in fibers. This breakdown occurs a result of the dehydration of the cellulose structures (Xin et al., 2015). Weight reduction of the composites enters third phase once temperatures exceed 350 °C, with a noticeable slowdown in the pyrolysis process across various composite materials. In this phase, the mass retention of the bamboo and kenaf fibers reinforced composites were higher than pure PLA, demonstrating that incorporation of bamboo and kenaf plays a key role in enhancing PLA's stability under elevated temperatures. In this phase weight loss occurs because of the decomposition of non-cellulosic materials (Asim et al., 2020).

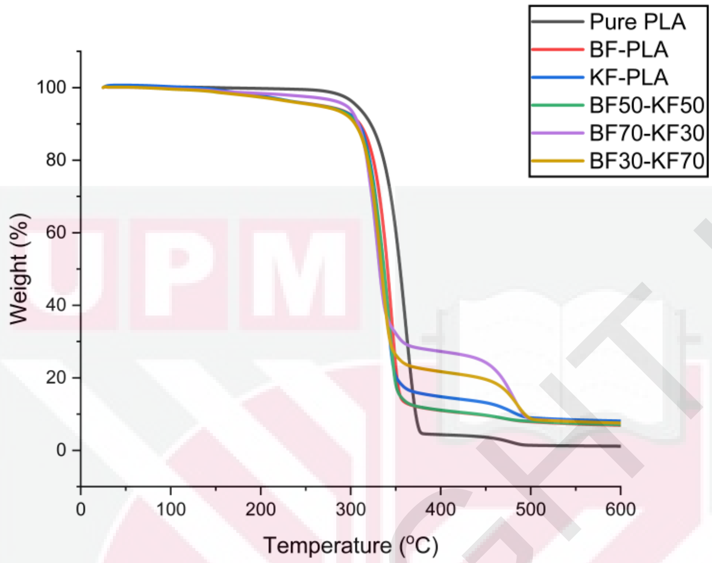

Figure 4.10 : TGA Curve of BF-KF/PLA Hybrid Composites

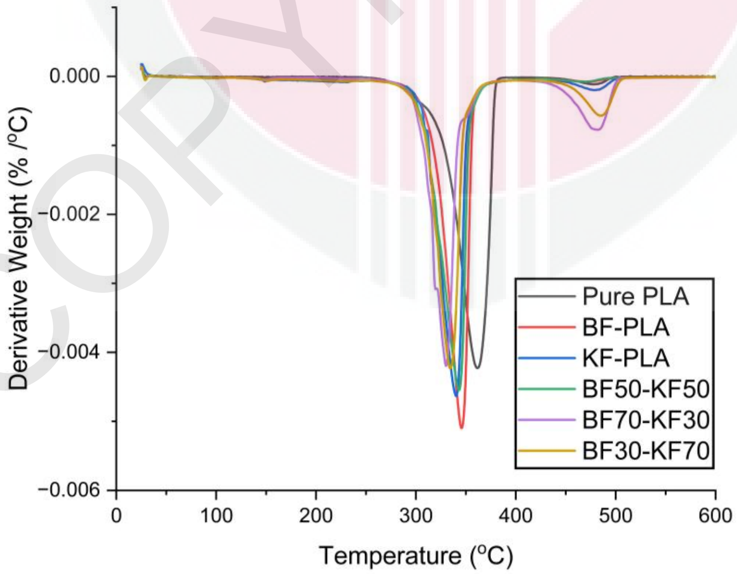

Figure 4.11 : DTG Curve of BF-KF/PLA Hybrid Composites

4.4.2 DSC Analysis

The thermal characteristics such as glass transition temperature (T_g), crystallization temperature (T_c), melting temperature (T_m), crystallization enthalpy (ΔH_c), and melting enthalpy (ΔH_m) obtained from differential scanning calorimetry (DSC) analyses of bamboo-kenaf fibers reinforced PLA composites are presented in **Table 4.4**.

Table 4.4 : DSC Results for BF-KF/PLA Composites

Sample	T_g (°C)	T_c (°C)	T_m (°C)
Pure PLA	53.60	107.83	156.71
BF-PLA	45.78	100.83	154.39
KF-PLA	45.51	97.46	154.06
BF50-KF50	46.50	98.76	154.66
BF70-KF30	50.76	100.10	155.89
BF30-KF70	46.33	98.14	153.48

Figure 4.12 shows the differential scanning calorimetry profiles for bamboo-kenaf fibers reinforced PLA composites undergoing both exothermic and endothermic processes. The DSC profiles exhibit a consistent pattern across all samples, with the identification of the exothermic and endothermic phases through the observation of several peaks. Observations reveal that the initial transition occurs in temperature range from 45 to 55 °C which signify the glass transition temperature (T_g) of PLA (Farah et al., 2016a). The incorporation of bamboo and kenaf fibers into PLA composites has been shown to reduce the T_g across all hybrid and non-hybrid samples. Among the fibers reinforced samples, BF70-KF30 hybrid sample showcased the highest T_g. This indicates the optimum percentage to increase in the glass transition temperatures for both bamboo and kenaf fibers are 70% and 30% respectively. Consequently,

a greater temperature is required for the transition of the material from a solid glass-like state to a more flexible, rubbery form (Azlin et al., 2022). When the composites are continuously heated, the samples undergo the exothermic crystallization phenomenon. During this process the composites make transition from a glassy state to an amorphous state. The cold crystallization temperature for PLA is highlighted by a peak in the temperature spectrum of 97–108 °C. The most rapid rate of PLA's cold crystallization has been reported to occur within the 95 to 115 °C temperature bracket (Iannace et al., 2014). The incorporation of bamboo and kenaf fibers induced nucleation of PLA polymer crystallization at a relative low temperature was initiated (Quiroz - Castillo et al., 2023; F. Wang et al., 2018b). Following this, the samples underwent heating up to 300 °C, revealing endothermic peaks. The endothermic peaks falling between 153-160 °C correspond to the melting temperatures (T_m) of PLA (Yang et al., 2008).

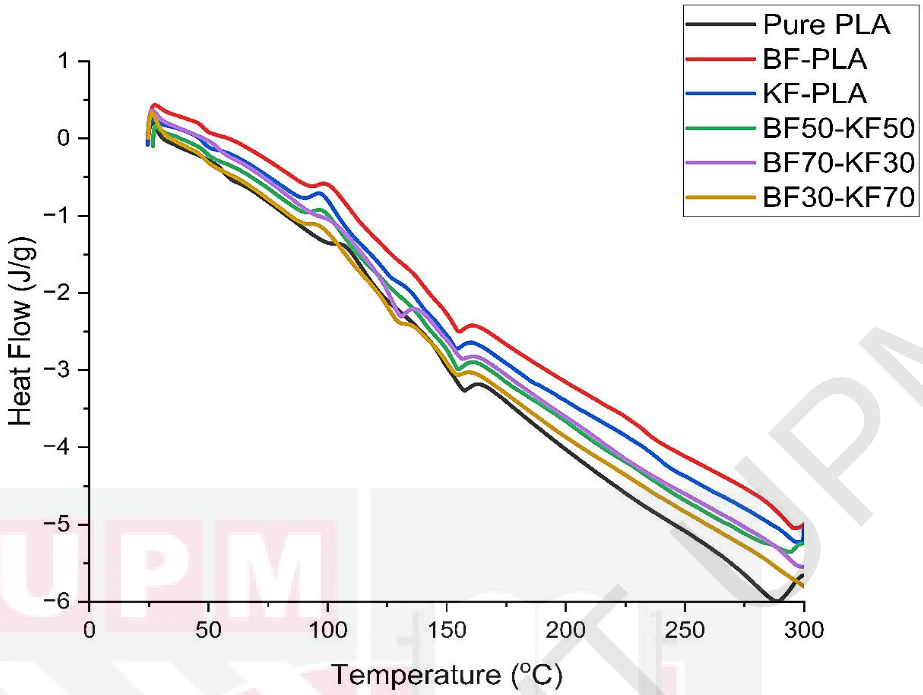

Figure 4.12 : DSC Thermogram of BF-KF/PLA Hybrid Composites

4.5 Water Absorption Properties of BF-KF/PLA Hybrid Composites

4.5.1 Water Absorption Test

The moisture absorption behavior was studied owing to the fact that the hydrophilic nature of natural fibers restricts the application of natural fiber reinforced composites in aquatic environments and water-related uses (Sanjeevi et al., 2021). **Figure 4.13** illustrates the mass of the composite specimens both in their initial state and following tap water absorption over a period of 0.5 hours and 2 hours. The data confirm the pronounced hydrophilic properties of the bamboo and kenaf fibers, which lead to increased moisture uptake. Hemicellulose present in both the fibers are responsible for moisture absorption behavior (Azwa et al., 2013). The reduced moisture absorption values observed for pure PLA sample can be attributed to the hydrophobicity nature of the PLA polymer (Farah et al., 2016b). KF-PLA sample was observed to have the highest water absorption percentage indicating poor fiber matrix adhesion. On the contrary, the BF50-KF50 hybrid composite sample recorded the lowest water absorption percentage among the fiber reinforced samples indicating better fiber matrix adhesion (Chandramohan et al., 2019). Therefore, in moist environment the 50:50 blend of bamboo and kenaf is expected to perform better in mechanical stress than the rest of the fiber reinforced composites.

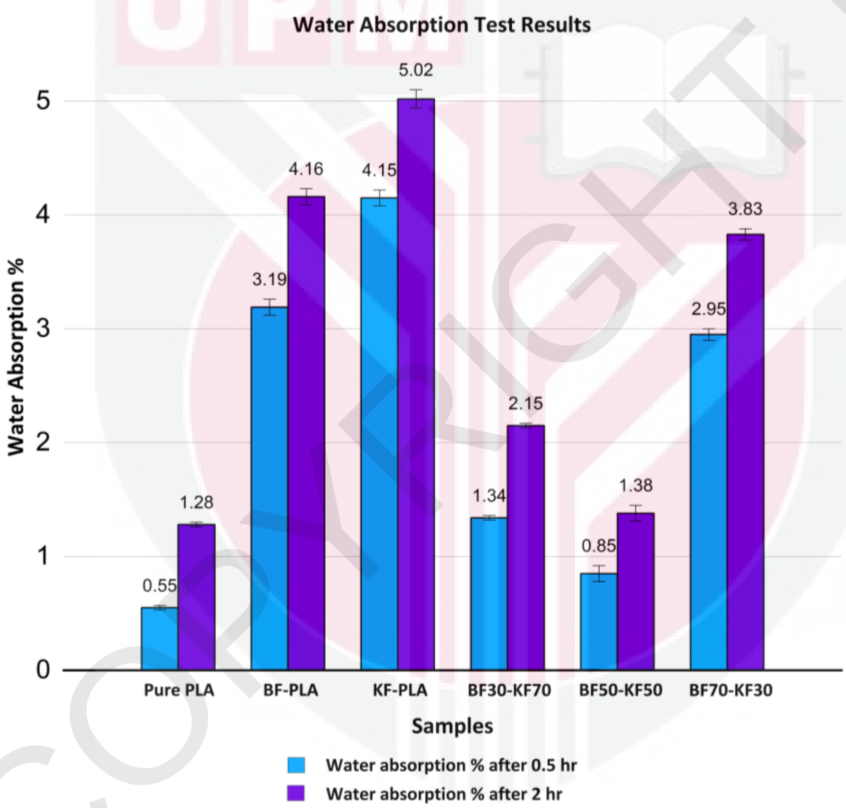

Figure 4.13 : Water Absorption Test Results of BF-KF/PLA Hybrid Composites

4.5.2 Rainwater Absorption

Composites based on natural fibers are sensitive to rainwater due to their hydrophilicity, making rainwater absorption characteristics important to study (Radzi et al., 2019). Rainwater absorption study was conducted particularly to understand the potential outdoor application of bamboo-kenaf/PLA hybrid composites. **Figure 4.14** depicts the rain water absorption percentage value for bamboo-kenaf/PLA hybrid composites. The BF-PLA and KF-PLA samples showed higher absorption rates, with KF-PLA recording the highest values, indicating poor fiber-matrix adhesion and the higher void contents of kenaf-PLA composite (Nunna et al., 2012). Among the hybrid composites, BF30-KF70 demonstrated moderate absorption levels, while BF50-KF50 displayed the lowest absorption among fiber-reinforced samples, suggesting optimal fiber-matrix adhesion and a balanced fiber composition. The BF70-KF30 showed an increasing trend in absorption. These results align with previous water absorption findings, where higher hydrophilicity and poor fiber-matrix adhesion led to increased water uptake, particularly in kenaf-reinforced composites. The consistent performance of BF50-KF50, with its balanced composition and lower water absorption, underscores its potential for products requiring moderate water resistance for outdoor application. Both the water and rainwater absorption behavior of the fiber reinforced composites reveal the scope for bacterial attack due to higher moisture absorption rates (Dinesh et al., 2020). Hence, the biodegradability of the PLA composites can be enhanced further.

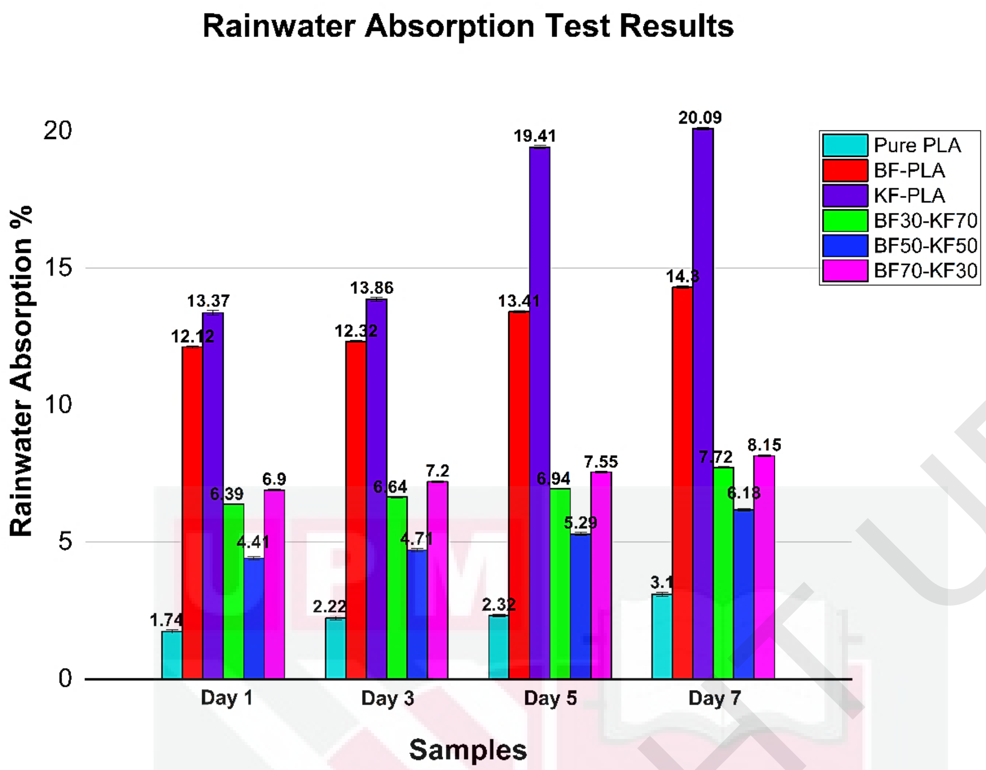

Figure 4.14 : Rainwater Absorption Test Results of BF-KF/PLA Hybrid Composites

4.5.3 Thickness Swelling

The thickness swelling behavior was examined to reveal the dimensional stability of the bamboo-kenaf/PLA hybrid composites in humid condition. Thickness swelling is often linked to the composite's ability to absorb moisture (Edhirej et al., 2017). The thickness swelling data of bamboo-kenaf/PLA hybrid composites after 0.5 hours and 2 hours of immersion are presented in **Table 4.5**. Thickness swelling percentages after 0.5 hour and 2 hours of immersion for the all the bamboo and kenaf fiber reinforced composites are higher compared to pure PLA sample due to the hygroscopic nature of the fibers (Chee et al., 2019). The hygroscopicity nature of natural fibers come from the presence of hydroxyl groups (-OH) which are polar and form bond with the water molecules (Akil et al., 2011). The presence of hydroxyl (–OH) groups in bamboo and kenaf fibers can cause fiber swelling and poor water resistance in hybrid composites. Consequently, water accumulation within the fiber cell wall and around the fiber-matrix interface increases the void content in fiber-based hybrid composites, leading to the swelling of both the fibers and the composites (Jawaid et al., 2022). The BF50-KF50 hybrid sample has seen the lowest swelling value among the fiber reinforced samples both after 0.5 hours and 2 hours. This lower swelling rate indicate the packed hybrid arrangement of the bamboo and kenaf fibers in the 50:50 blend. The packed arrangement of fibers limits the moisture absorption and thereby greater dimensional stability (Saw et al., 2014).

Table 4.5 : Thickness Swelling Test Results of BF-KF/PLA Hybrid Composites

Sample	Thickness Swelling %	
	0.5 hr	**2 hr**
Pure PLA	0.20 ± 0.03	0.65 ± 0.02
BF-PLA	0.59 ± 0.11	0.88 ± 0.09
KF-PLA	0.72 ± 0.23	1.76 ± 0.25
BF30-KF70	0.41 ± 0.07	0.74 ± 0.06
BF50-KF50	0.29 ± 0.17	0.67 ± 0.20
BF70-KF30	0.59 ± 0.05	0.78 ± 0.04

4.5.4 Water Solubility

The water solubility of a compound exhibits a direct correlation with the biodegradability characteristics of the corresponding material (Nazrin et al., 2021). Moisture absorption leads to the swelling of capillaries of fibers and the formation of micro-cracks in fiber-reinforced composites (Bachchan et al., 2022). These micro-cracks allow water molecules to diffuse into the fiber-matrix interfaces. As water penetrates the composite, it can leach water-soluble substances from the fibers, particularly at the fiber-matrix interface, leading to osmotic pressure buildup and eventual debonding between the fiber and matrix (Joseph et al., 2002). This degradation process is accelerated in high-humidity or high-temperature environments, where moisture-induced microcracks, swelling, and surface dissolution further compromise the composite's integrity. Therefore, the weight of the composites is reduced in the water solubility test. As demonstrated in **Figure 4.15**, the solubility of all the composites was affected by the bamboo and kenaf fiber content. Notably, the KF-PLA formulation exhibits the highest solubility rate, measured at 7.15% in 24 hours. This result correlates with the higher water absorption results for the KF-PLA sample. All the other composite samples follow the similar trend as observed in the water absorption test. Water solubility test revealed significant results that indicate enhanced biodegradability of the PLA composites. Both low and high solubility materials offer advantages for a range of applications. Materials with low solubility are particularly valuable in applications that require protection from moisture and prevention of water loss. Conversely, high solubility materials are suitable for enhanced biodegradable application (Cai et al., 2011; Lv et al., 2018; Preechawong et al., 2005).

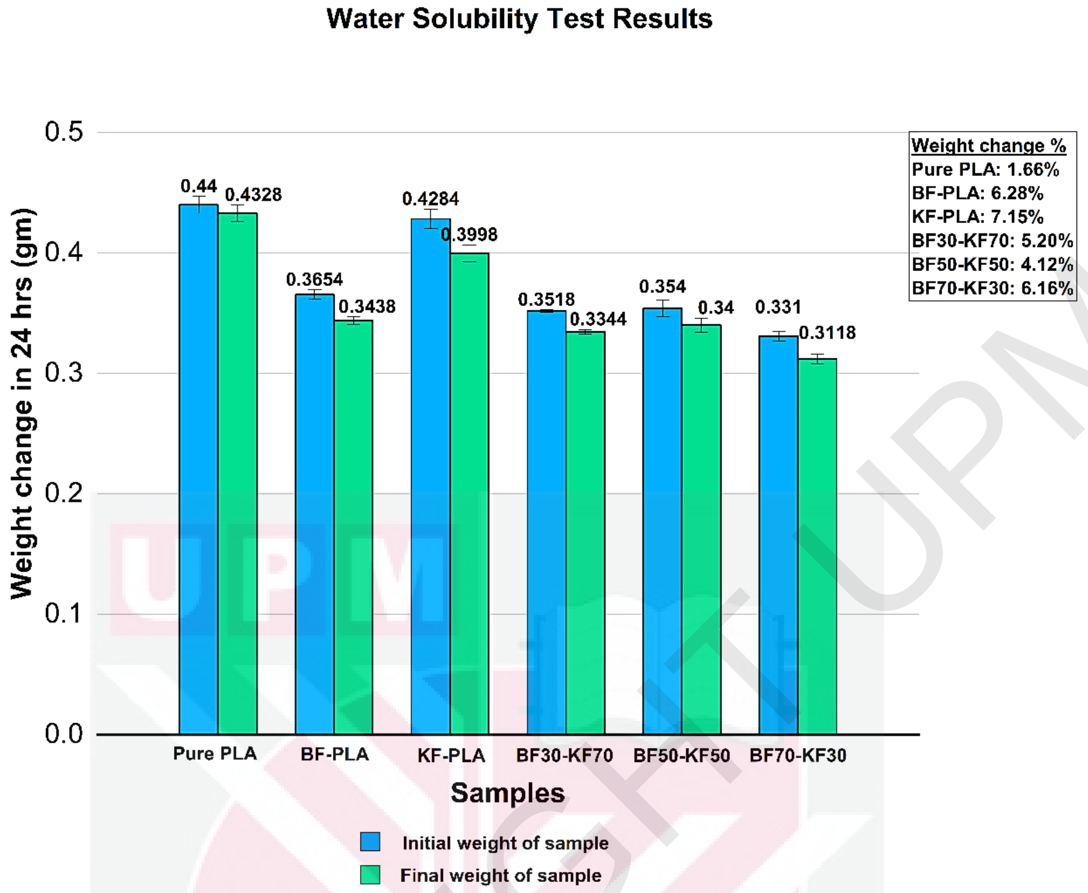

Figure 4.15 : Water Solubility Test Data of BF-KF/PLA Hybrid Composites

4.5.5 Water Vapor Permeability (WVP)

The WVP test determines the effectiveness of composite materials in preventing moisture transmission as barrier material (Chen et al., 2023). WVP is influenced by the free volume within the polymeric network and the interactions between polymer chains (Caicedo et al., 2022). When the composite is less dense, there is an increase in free volume and a decrease in direct interactions between the polymer chains. This less dense structure facilitates greater adsorption of water molecules on the surface and allows for easier penetration of water molecules through the polymer structure (higher diffusivity), thereby increasing the WVP (Loredo et al., 2016). Conversely, when the polymer network is denser, with less free volume and stronger interactions between polymer chains, the movement of water vapor molecules is restricted. This results in lower adsorption and penetration of water molecules, thus decreasing the WVP. The density values of the developed composites were found to be as follows: pure PLA (1.2034 g/cm^3), BF-PLA (1.1826 g/cm^3), KF-PLA (1.2479 g/cm^3), BF30-KF70 (1.2471 g/cm^3), BF50-KF50 (1.2392 g/cm^3) and BF70-KF30 (1.1505 g/cm^3). From **Table 4.6** it is observed that KF-PLA having the highest density among the samples exhibited the least WVP of 5.121845×10^{-10} g s^{-1}m^{-1}Pa^{-1}. Reducing the water vapor transmission remains a significant challenge in the development of eco-friendly packaging materials (Song et al., 2014). Therefore, KF-PLA composites with lower WVP are ideal for moisture-sensitive applications such as pharmaceutical packaging, where effective moisture barriers are crucial. On the other hand, the BF70-KF30 sample having the lowest density values recorded the highest WVP values of 1.894463×10^{-9} g s^{-1}m^{-1}Pa^{-1}. The observed WVP data indicate that composites with higher WVP, like BF70-KF30, are suitable for applications requiring breathability, such as packaging for fresh produce or breathable building membranes.

Table 4.6 : Water Vapor Permeability Data of BF-KF/PLA Hybrid Composites

Samples	WVP (g s^{-1}m^{-1}Pa^{-1})
Pure PLA	1.732164×10^{-9}
BF-PLA	1.787664×10^{-9}
KF-PLA	5.121845×10^{-10}
BF30-KF70	1.467824×10^{-9}
BF50-KF50	1.636563×10^{-9}
BF70-KF30	1.894463×10^{-9}

4.6 Surface Wettability Properties of BF-KF/PLA Hybrid Composites

4.6.1 Water Contact Angle

Water contact angle is an important quantitative measure used to assess the hydrophilicity or hydrophobicity of composite materials, providing insight into their surface wettability (Yorseng et al., 2020). A contact angle greater than 90° indicates a hydrophobic surface, which repels water and minimizes absorption, whereas a contact angle below 90° denotes a hydrophilic surface with higher wettability and improved self-cleaning capabilities (Ismail et al., 2022). The surface roughness of a composite material plays a key role in contact angle values. This relationship between roughness and wettability is explained by the Wenzel and Cassie-Baxter models (Kubiak et al., 2011). Increased surface roughness generally leads to a higher contact angle, as rough textures create air pockets under water droplets, reducing the contact area and enhancing hydrophobicity. As observed in **Figure 4.16**, the pure PLA exhibited a contact angle of 83.45°, reflecting its relatively hydrophobic nature with limited water absorption. However, the BF-PLA composite showed a lower contact angle of 78.38°, indicating increased surface roughness and improved wettability due to the exposure of hydrophilic groups in the bamboo fibers. The KF-PLA composite had an even lower contact angle of 73.09°, suggesting better hydrophilicity. The most hydrophilic configuration, the BF70-KF30 composite (70% bamboo fiber and 30% kenaf fiber), recorded the lowest contact angle of 50.78°. This result demonstrates that incorporating both bamboo and kenaf fibers in a hybrid configuration significantly enhances the hydrophilicity of the PLA matrix. The enhanced hydrophilicity observed in these fiber-reinforced composites is largely attributed to the alkali treatment of the fibers. Alkali treatment removes surface impurities such as waxes, lignin, and hemicellulose, increasing surface roughness and exposing more hydroxyl groups. Consequently, the surface energy of the fibers is enhanced, leading to better wettability and a reduced contact angle (Ariawan et al., 2017). These findings are significant for applications requiring moisture interaction and strong adhesion properties, such as outdoor environments. In such cases, hydrophilic surfaces are often preferred to ensure optimal adhesion for coatings such as varnish or lacquer (Awaja et al., 2009). Therefore, the incorporation of bamboo and kenaf fibers can broaden the applicability of PLA polymer.

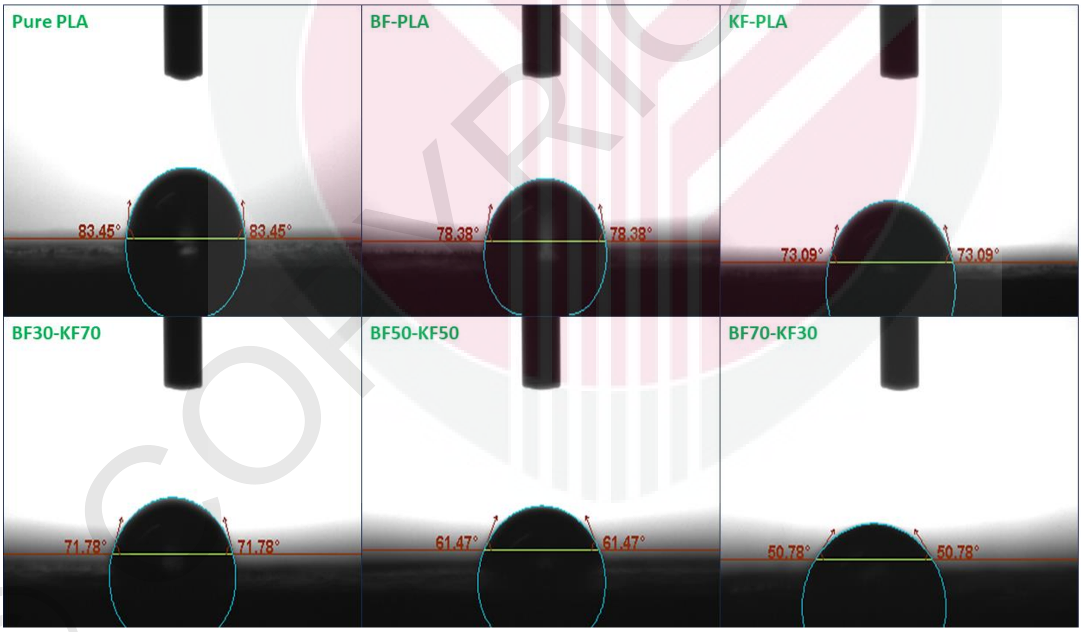

Figure 4.16 : Water Contact Angle Figures of BF-KF/PLA Hybrid Composites

4.7 Flammability Properties of BF-KF/PLA Hybrid Composites

4.7.1 UL-94 (Vertical) Test

The UL-94 vertical test signifies the ability of a material to extinguish itself during continuous combustion and widely accepted for plastic composites (Subasinghe et al., 2016). This test classifies materials into categories such as V-0, V-1, and V-2 based on its self-extinguishing properties with V-2 being the least flame retardant. The flammability properties of bamboo-kenaf fibers reinforced PLA composites are shown in **Table 4.7**. All the composite samples achieved V-2 rating in the UL-94 test as in case of all the specimens, the cotton used in the test was ignited (ASTM, 2019). PLA has been reported to ignite quickly and can drip while it melts (Li et al., 2018). This property of PLA potentially exacerbates the spread of a fire. When bamboo fibers were incorporated into PLA, the burning time after first ignition was reduced due to the char formation by bamboo fiber (Khan et al., 2023). According to Niu et al. (Niu et al., 2022b) higher bamboo fibers content result in pronounced wick effect and as a result poorer flame retardancy is observed. This justifies the higher total burning time for BF70-KF30 compared to BF50-KF50 composites. In case of KF-PLA, the burning time after first flame ignition was also reduced compared to pure PLA. The probable reason is an increase in viscosity and less heat was transmitted through convection method in the melt (Sypaseuth et al., 2017). Therefore, the BF30-KF70 sample has lower total burning time compared to BF70-KF30 sample. V-2 rating signifies that the BF-KF/PLA composites would provide adequate flame resistance under regular working condition highlighting the prospect of consumer products. However, in case of high temperature applications, the composites must be flame proofed with chemical treatments or coatings.

Table 4.7 : UL-94 Test Data of BF-KF/PLA Hybrid Composites

Sample	Specimen	t_1(s)	t_2(s)	t_2+t_3 (s)	Total burning time, $t_1 + t_2$ (s)	Total burning time for 5 specimens, t_1+t_2 (s)	Dripping / Cotton ignition	Burning or glowing up to holding clamp	Rating
Pure PLA	S1	23.2	20.1	20.1	43.3		Yes	No	
	S2	24.7	17.9	17.9	42.6		Yes	No	
	S3	6.2	25.8	25.8	32.0	213.6	Yes	No	V-2
	S4	26.3	29.0	29.0	55.3		Yes	No	
	S5	26.4	14.0	14.0	40.4		Yes	No	
BF-PLA	S1	5.6	22.4	22.4	28.0		Yes	No	
	S2	5.3	23.4	23.4	28.7		Yes	No	
	S3	5.5	16.1	16.1	21.6	125.3	Yes	No	V-2
	S4	15.4	6.7	6.7	22.1		Yes	No	
	S5	9.8	15.1	15.1	24.9		Yes	No	
KF-PLA	S1	0.5	41.7	41.7	42.2		Yes	No	
	S2	16.9	18.3	18.3	35.2		Yes	No	
	S3	14.7	8.0	8.0	22.7	170.0	Yes	No	V-2
	S4	9.2	13.4	13.4	24.4		Yes	No	
	S5	19.7	25.8	25.8	45.5		Yes	No	
BF30-KF70	S1	9.9	7.1	7.1	17.0		Yes	No	
	S2	12.8	12.6	12.6	25.4		Yes	No	
	S3	10.0	25.4	25.4	35.4	117.2	Yes	Yes	V-2
	S4	11.7	8.8	8.8	20.5		Yes	No	
	S5	10.8	8.1	8.1	18.9		Yes	No	
BF50-KF50	S1	11.8	18.9	18.9	30.7		Yes	No	
	S2	10.3	14.4	14.4	24.7		Yes	No	
	S3	7.4	18.9	18.9	26.3	213.6	Yes	No	V-2
	S4	13.7	15.1	15.1	28.8		Yes	No	
	S5	10.3	12.8	12.8	23.1		Yes	No	
BF70-KF30	S1	18.7	28.8	28.8	47.5		Yes	Yes	
	S2	21.6	12.8	12.8	34.4		Yes	No	
	S3	22.8	29.4	29.4	44.2	218.8	Yes	Yes	V-2
	S4	21.6	15.7	15.7	37.3		Yes	No	
	S5	55.4	0.0	0.0	55.4		Yes	Yes	

*t1= Burning time after 1st flame application, t2= Burning time after 2nd flame application and t2+t3= Burning plus glowing time after 2nd flame application

4.7.2 Limiting Oxygen Index (LOI) Test

The Limiting Oxygen Index (LOI) represents the minimum oxygen concentration in a flowing atmosphere required to sustain the downward burning of a plastic strip or fabric (Lyon et al., 2010). The LOI test provides precise, quantitative data on the oxygen concentration needed to maintain combustion, which is essential for assessing and enhancing the fire resistance and thermal stability of bamboo-kenaf/PLA hybrid composites. As presented in **Table 4.8**, the LOI value of Pure PLA, BF30-KF70 and BF70-KF30 are 19.03%, 19.81% and 19.20% respectively. These values classify the composites as combustible since they are below the oxygen content in air (21%) (Chee et al., 2020). However, the incorporation of bamboo fiber into PLA significantly improves flame resistance, evidenced by an LOI of 21.88% for BF-PLA sample. This suggests that bamboo fibers enhance resistance to burning due to their intrinsic properties, such as higher thermal stability and char formation during combustion (Liu et al., 2014). The increased formation of char decreases the release of flammable volatiles, thereby raising the oxygen concentration required to sustain combustion (Chiang & Ma, 2002). Similarly, the OI of 21.79% for kenaf fiber reinforced PLA (KF-PLA) shows improved flame resistance compared to Pure PLA. Kenaf fibers, known for their high cellulose content, likely enhanced the composite's ability to resist ignition and sustain combustion (Dorez et al., 2014). An equal blend of bamboo and kenaf fibers (BF50-KF50) results in a balanced improvement in flame resistance with an LOI of 21.10%. This indicates that the synergistic effect of both fibers enhances the composite's flame-retardant properties, possibly due to a balanced contribution of thermal stability and char formation from both fiber types. The data indicates that both bamboo and kenaf fibers enhance the flame resistance of PLA, but the effectiveness depends on their proportion. Common polymers like polypropylene (PP) and polyethylene (PE) have LOI values around 17-19% (Goodarzi et al., 2008; Xie et al., 2006). The LOI results for the bamboo-kenaf fiber reinforced PLA hybrid composites indicate higher flame resistance, making them suitable for eco-friendly plastic furniture and other consumer products. For such applications, materials need to balance flammability with sustainability and cost-effectiveness. For enhanced fire protection, further optimization through fire retardant coatings or treatments can be considered.

Table 4.8 : LOI Value of BF-KF/PLA Hybrid Composites

Sample	LOI
Pure PLA	19.03%
BF-PLA	21.88%
KF-PLA	21.79%
BF30-KF70	19.81%
BF50-KF50	21.10%
BF70-KF30	19.20%

4.8 Summary

The findings indicate that BF-KF/PLA hybrid composites offer significant potential for sustainable furniture applications. The reduced density with BF reinforcement makes the material lighter, while the inclusion of KF increases robustness, providing options for different furniture needs. The 50:50 bamboo-kenaf blend's low void content (0.27%) enhances structural integrity, and the high crystallinity of BF-PLA ensures stiffness and resistance to deformation. The highlighted summary of mechanical properties presented in **Table 4.9** shows BF-PLA's superior tensile and compressive strength makes it ideal for load-bearing furniture, while the 30BF-70KF hybrid's notable impact strength is suited for high-use parts. Excellent fiber-matrix adhesion further improves durability, and the high thermal stability (onset of degradation at 484°C) of BF30-KF70 ensures safety in heat-exposed conditions. Overall, these composites present an eco-friendly alternative to commercially available materials for furniture as highlighted in **Table 4.10** by balancing strength, flexibility, and thermal resistance.

Table 4.9 : Summary of the Mechanical Properties of BF-KF/PLA Hybrid Composites

Sample	Tensile Strength (MPa)	Flexural Strength (MPa)	Compressive Strength (MPa)	Impact Strength (Nm)	Hardness (HRS)
Pure PLA	24.51	71.09	128.09	5.6	47.18
BF-PLA	25.95	45.53	173.15	6.2	59.32
KF-PLA	8.45	27.46	134.52	6.3	69.2
BF30-KF70	9.35	25.74	169.03	6.6	49.26
BF50-KF50	17.82	33.18	112.35	6.3	55.8
BF70-KF30	6.94	21.56	89.48	6.4	29.6

Table 4.10 : Comparison of BF-KF/PLA Composites with Commercially Available Materials for Furniture Application

Criteria	This Study	SABIC	POLYGOOD	UKCOMPOSITE DECKING
Materiall	BF-KF/PLA	20% Talc-80% Polypropylene	100% Recycled polystyrene	55% Wood fibers – 45% Polyethylene
Tensile Strength	25.95 (BF-PLA)	25 MPa	22.6 MPa	1.3 MPa
Tensile Modulus	2199.15 MPa (BF-PLA)	2100 MPa	1877 MPa	
Flexural Strength	45.53 MPa (BF-PLA)	-	51.77 MPa	31.5 MPa
Flexural Modulus	2900.41 MPa (BF-PLA)	2650 MPa	-	4181 MPa
Impact Strength	6.6 Nm (BF30-KF70)	0.24 Nm	0.28 Nm	0.336 Nm
Application	Furniture	Furniture parts	Furniture	Fencing Rails
Reference	-	(SABIC, 2024)	(POLYGOOD, 2024)	(UKCOMPOSITE DECKING, 2024)

CHAPTER 5

CONCLUSIONS AND RECOMMENDATION FOR FUTURE RESEARCH

5.1 Conclusions

This study developed BF-KF/PLA hybrid composites as eco-friendly alternatives for synthetic plastic composites. Key findings include reduced density, void content and enhanced crystallinity with BF inclusion, superior tensile (25.95 MPa) and compressive (173.15 MPa) strengths in BF-PLA composites, improved impact strength (6.6 Nm) in BF30-KF70, and high hardness resistance (69.2 HRS) in KF-PLA composites. The fiber reinforced composites exhibited higher thermal degradation temperatures, with BF30-KF70 reaching 484°C. These physical, mechanical and thermal properties make the composites suitable for applications in eco-friendly furniture components. The use of renewable fibers with biodegradable PLA aligns with the growing demand for sustainable materials.

Results from the second objective shows that the incorporation of BF and KF increased moisture uptake, indicating enhanced biodegradability. KF-PLA outperformed the other composites in biodegradability, with water solubility tests confirming this. Thickness swelling tests showed that the 50:50 bamboo-kenaf blend (BF50-KF50) had good dimensional stability. Water vapor permeability (WVP) tests revealed that the densest composite, KF-PLA, had the lowest WVP, making it suitable for moisture-sensitive application of furniture. Surface wettability tests indicated increased hydrophilicity, particularly in BF50-KF50, making it ideal for strong adhesion application such as painting or coating. Flammability tests (UL-94) showed that all composites achieved a V-2 rating, with BF70-KF30 having the longest total burning time. Limiting oxygen index (LOI) values demonstrated improved flame resistance in BF-PLA (21.88%) and KF-PLA (21.79%) compared to pure PLA (19.03%), with BF50-KF50 achieving an LOI of 21.10%, indicating optimal flame resistance. These findings highlight the potential of BF-KF/PLA composites for eco-friendly furniture applications.

5.2 Recommendations for Future Research

1. Future research direction could explore the integration of nanomaterials such as carbon nanotubes or graphene nanoplates into BF-KF/PLA hybrid composites to enhance physical, mechanical and thermal and properties, paving the way for advanced industrial applications.
2. Future research should explore other possible ratio of bamboo and kenaf fibers and advanced surface treatments such as silane treatment etc. for specific advanced industrial applications.

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APPENDICES

Appendix A

A.1 Twin screw extruder

A.2 Hot press

A.3 Sputter coater machine

A.4 FESEM machine

A.5 FTIR instrument

A.6 Oven

A.7 Pulverizer

A.8 Crusher machine

A.9 Composite sheet cutter

A.10 Heavy duty blender

LIST OF PUBLICATIONS

Journal Articles

Khan, A., Sapuan, S. M., Siddiqui, V. U., Zainudin, E. S., Zuhri, M. Y. M., & Harussani, M. M. (2023). A review of recent developments in kenaf fiber/polylactic acid composites research. International Journal of Biological Macromolecules, 127119. https://doi.org/10.1016/j.ijbiomac.2023.127119
(Q1, Impact factor-8.2)

Khan, A., Sapuan, S. M., Yusuf, J., Siddiqui, V. U., Zainudin, E. S., Zuhri, M. Y. M., Baharuddin BH, Ansari MA & Rahman, A. A. A. (2023). An examination of cutting-edge developments in Bamboo-PLA composite research: A comprehensive review. Renewable and Sustainable Energy Reviews, 188, 113832. https://doi.org/10.1016/j.rser.2023.113832
(Q1, Impact factor-15.7)

Khan, A., Sapuan, S. M., Zainudin, E. S., Zuhri, M. Y. M. Hybridization and Its Transformative Role in Bamboo Fiber Reinforced Polymer Composites: A Review. Advanced Composites and Hybrid Materials https://doi.org/10.1007/s42114-024-00974-8
(Paper accepted in Q1 journal, Impact factor-23.2)

Khan, A., Sapuan, S. M., Zainudin, E. S. & Zuhri, M. Y. M. (2024). Physical, Mechanical and Thermal Properties of Novel Bamboo/Kenaf Fiber-Reinforced Polylactic Acid (PLA) Hybrid Composites. Composites Communications, 51, 102103 https://doi.org/10.1016/j.coco.2024.102103
(Q1, Impact factor-6.5)

Khan, A., Sapuan, S. M., Zainudin, E. S., Zuhri, M. Y. M. Water Absorption, Surface Wettability and Flammability Properties of Novel Bamboo-Kenaf Fiber Reinforced PLA Hybrid Composites.
(Paper submitted in Q1 journal)

Conference Proceedings

Khan, A., Sapuan, S. M., INTERNATIONAL CONFERENCE ON ADVANCED ENGINEERING MATERIALS AND COMPOSITES 2024, Title "Advancements in Bamboo-Kenaf Fibers Reinforced Hybrid Polymer Composites Research", e-ISBN: 978-629-97315-2-8.

Khan, A., Sapuan, S. M., INTERNATIONAL CONFERENCE ON ADVANCED ENGINEERING MATERIALS AND COMPOSITES 2024, Title "A Review on Recent Advancements in Textile Composites for Biomedical Applications", e-ISBN: 978-629-97315-2-8.

Book Chapters

Khan, A., Sapuan, S. M., Effect of Chemical Surface Modifications of Bamboo Fibre on Mechanical and Thermal Performance. In Bamboo-based Polymer Composites. Elsevier.
(Chapter submitted)