UNIVERSITI TEKNIKAL MALAYSIA MELAKA

DEVELOPMENT OF BIODEGRADABLE PACKAGING FROM CASSAVA STARCH AND CORN HUSK FIBRE: MECHANICAL PROPERTIES

NURAIN BINTI ZAMRI
B092010115

BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY WITH HONOURS

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Faculty of Industrial and Manufacturing Engineering Technology

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NURAIN BINTI ZAMRI

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Disahkan oleh:

Alamat Tetap:

A-1-19, Apartment Kenanga, Taman Bunga Raya, Bukit Beruntung, 48300 Rawang, Selangor.

Cop Rasmi: DR. RIDHWAN BIN JUMAIDIN
Timbalan Pengarah II
Pejabat Perancangan & Pembangunan Akademik
Pejabat Timbalan Naib Canselor (Akademik & Antarabangsa)
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I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology with Honours.

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DEDICATION

Alhamdulillah

Praise to Allah for the strength, guidance and knowledge that was given by Allah for me to complete this proposal report

&

To my beloved parents for every support and encouragement that was given to me

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To all people who support me throughout this journey

&

To Al-Quran, the greatest source of knowledge

ABSTRACT

The accumulation of non-biodegradable plastic waste has emerged as a major environmental issue, causing significant harm to plant life and wildlife worldwide. In response, it has become crucial to develop environmentally friendly products, and there have been significant advancements in material quality to address this problem. Extensive research has been conducted on biopolymers sourced from renewable resources, as they hold great promise as alternatives to petroleum-based polymers. These biopolymers offer the advantage of biodegradability and environmental sustainability, making them a viable solution in tackling this challenge. Corn husk fibre (CHF) is a potential natural fiber resource in Malaysia. Meanwhile, thermoplastic cassava starch (TPCS) is a biopolymer derived from starch, however it has poor mechanical properties. Hence, this study aims to develop a composite material by reinforcing biodegradable TPCS with CHF. The mechanical and thermal properties of the TPCS reinforced with CHF will be investigated, and subsequently, biodegradable packaging will be fabricated using CHF and cassava starch. The goal is to explore the potential of these materials for environmentally friendly packaging applications. To achieve the study aims, a composite material was developed by blending CHF, cassava starch, glycerol, and palm wax. The fabrication process involved hot compression molding at a temperature of 155 °C for 60 minutes. By incorporating palm wax and CHF, the limitations of cassava starch biopolymer were addressed. Various compositions of CHF ranging from 0 to 40 wt% were investigated in this study. The mechanical testing involved analysis for tensile and flexural properties, while thermal analysis comprised Thermogravimetric Analysis (TGA). Additionally, Scanning Electron Microscopy (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) were employed for further examination. The results of tensile and flexural properties of CHF/TPCS showed an increasing trend with the addition of CHF into TPCS. Tensile and flexural strength both showed maximum strength at 40 wt.% CHF which are 2.41MPa and 10.39MPa respectively. The tensile and flexural modulus also record the same trend where the highest value are 288.492MPa for tensile and 725.4808MPa for flexural at 40 wt.%. FTIR showed improvement in the bonding between the components of the composite where O-H bonding in the composite was indicated by the highest peak at 3200cm^{-1}. When the fiber content increased, SEM of the composite revealed more micro voids, matrix cracks, and fiber breakage which aligned with the tensile and flexural findings. As a result of TGA testing, it was observed that the addition of fiber content increased the characteristics in terms of thermal stability. TPCS/CHF composite showed promise as a viable alternative material for biodegradable products, including disposable packaging trays, offering increased functionality and utility.

ABSTRAK

Pengumpulan sisa plastik tidak terbiodegradasi telah muncul sebagai isu alam sekitar yang utama, menyebabkan kemudaratan besar kepada kehidupan tumbuhan dan hidupan liar di seluruh dunia. Sebagai tindak balas, menjadi penting untuk membangunkan produk mesra alam, dan terdapat kemajuan ketara dalam kualiti bahan untuk menangani masalah ini. Penyelidikan meluas telah dijalankan ke atas biopolimer yang diperoleh daripada sumber yang boleh diperbaharui, kerana ia mempunyai janji besar sebagai alternatif kepada polimer berasaskan petroleum. Biopolimer ini menawarkan kelebihan biodegradasi dan kemampanan alam sekitar, menjadikannya penyelesaian yang berdaya maju dalam menangani cabaran ini. Serat sekam jagung (CHF) merupakan sumber gentian semula jadi yang berpotensi di Malaysia. Sementara itu, kanji ubi kayu termoplastik (TPCS) adalah biopolimer yang diperoleh daripada kanji, namun ia mempunyai sifat mekanikal yang lemah. Oleh itu, kajian ini bertujuan untuk membangunkan bahan komposit dengan mengukuhkan TPCS terbiodegradasi dengan CHF. Sifat mekanikal dan haba TPCS yang diperkukuh dengan CHF akan disiasat, dan seterusnya, pembungkusan biodegradasi akan dibuat menggunakan CHF dan kanji ubi kayu. Matlamatnya adalah untuk meneroka potensi bahan ini untuk aplikasi pembungkusan mesra alam. Untuk mencapai matlamat kajian, bahan komposit telah dibangunkan dengan mengadun CHF, kanji ubi kayu, gliserol, dan lilin sawit. Proses fabrikasi melibatkan pengacuan mampatan panas pada suhu 155 °C selama 60 minit. Dengan menggabungkan lilin sawit dan CHF, batasan biopolimer kanji ubi kayu telah ditangani. Pelbagai komposisi CHF antara 0 hingga 40% berat telah disiasat dalam kajian ini. Ujian mekanikal melibatkan analisis untuk sifat tegangan dan lentur, manakala analisis haba terdiri daripada Analisis Termogravimetrik (TGA). Selain itu, Scanning Electron Microscopy (SEM) dan Fourier-Transform Infrared Spectroscopy (FTIR) telah digunakan untuk pemeriksaan lanjut. Keputusan sifat tegangan dan lentur CHF/TPCS menunjukkan trend yang meningkat dengan penambahan CHF ke dalam TPCS. Kekuatan tegangan dan lentur kedua-duanya menunjukkan kekuatan maksimum pada 40 wt.% CHF iaitu masing-masing 2.41MPa dan 10.39MPa. Modulus tegangan dan lentur juga mencatatkan aliran yang sama di mana nilai tertinggi ialah 288.492MPa untuk tegangan dan 725.4808MPa untuk lenturan pada 40 wt.%. FTIR menunjukkan peningkatan dalam ikatan antara komponen komposit di mana ikatan O-H dalam komposit ditunjukkan oleh puncak tertinggi pada 3200cm-1. Apabila kandungan gentian meningkat, SEM komposit mendedahkan lebih banyak lompang mikro, keretakan matriks, dan pecahan gentian yang sejajar dengan penemuan tegangan dan lentur. Hasil daripada ujian TGA, diperhatikan bahawa penambahan kandungan gentian meningkatkan ciri-ciri dari segi kestabilan terma. Komposit TPCS/CHF menunjukkan janji sebagai bahan alternatif yang berdaya maju untuk produk terbiodegradasi, termasuk dulang pembungkusan pakai buang, menawarkan peningkatan fungsi dan utiliti.

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LIST OF SYMBOLS AND ABBREVIATIONS

%	-	Percentage
°C	-	Degrees Celsius
µm	-	Micrometer
AR	-	Analytical Reagent
As	-	Arsenic
ASTM	-	American Society for Testing and Materials
BLF	-	Banana Leaf Fiber
C	-	Carbon
CCF	-	Cymbopogan Citratus Fiber
CF	-	Carbon Fiber
CFRC	-	Carbon Fibre Reinforced Composite
CHF	-	Corn husk fiber
Cl	-	Chloride
CMC	-	Ceramic Matrix Composite
CNFs	-	Cellulose Nanofibrils
CNTs	-	Carbon Nanotubes
Cu	-	Copper
DSC	-	Differential Scanning Calorimetry
DTG	-	Derivative Thermogravimetric
FTIR	-	Fourier Transform Infrared Spectroscopy
g	-	Gram
G.C	-	Gas Chromatography
g/mol	-	Grams per mole
GFRC	-	Glass Fibre Reinforced Composite
GPa	-	Gigapascal
H	-	Hydrogen
HCHO	-	Formaldehyde
JASCO	-	Japan Spectroscopic Corporation
KBr	-	Potassium Bromide

kV	-	KiloVolt
mA	-	Milliampere
mg	-	milligram
mm	-	Millimeter
MMC	-	Metal Matrix Composite
MPa	-	Megapascal
N/mm^2	-	Newton per square millimeter
NaOH	-	Sodium Hydroxide
NFRC	-	Natural Fibre Reinforced Composite
NH4	-	Ammonium
Ni	-	Ni
nm	-	Nanometer
O	-	Oxygen
OPS	-	Oil Palm Shell
Pb	-	Lead
PBAT	-	Poly (butylene adipate-co-terephthalate)
PLA	-	Polylactic Acid
PMC	-	Polymer Matrix Composite
PP	-	Polypropylene
RF	-	Roselle fiber
r-HDPE	-	Recycled high-density polyethylene
SEM	-	Scanning Electron Microscope
SO4	-	Sulfate
SPF	-	Sugar palm fiber
SPS	-	Sugar Pal Starch
Tc	-	Curing Temperature
Td	-	Exothermic Temperature
Tg	-	Glass Transition Temperature
TGA	-	Thermogravimetric Analysis
Tm	-	Melting Temperature
TPCS	-	Thermoplastic cassava starch

TPU	-	Thermoplastic polyurethane
UTM	-	Universal Testing Machine
Vf	-	Volume fraction
wt%	-	Weight percentage
XRD	-	X-ray Diffraction
Zn	-	Zinc
α	-	Alpha (Greek letter)
β	-	Beta (Greek letter)

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CHAPTER 1

INTRODUCTION

1.1 Background

The escalating environmental crisis resulting from the widespread use and improper disposal of plastic products has become a significant concern. Plastics, particularly those derived from petroleum, present significant challenges in terms of their non-biodegradability and negative effects on ecosystems and human health. Research estimates that approximately 8 million metric tons of plastic waste find their way into the oceans every year, posing a severe threat to marine life and ecosystems (Jambeck et al., 2015). The accumulation of plastic waste in landfills and oceans has led to ecological imbalances, harm to wildlife, and the contamination of water sources. Therefore, there is a growing urgency to develop sustainable alternatives to traditional plastics that can reduce environmental burdens and foster a circular economy (Jung et al., 2020).

Biodegradable materials, especially those derived from renewable resources, represent a promising solution to address the environmental challenges posed by conventional plastics. Biopolymers, such as starch-based materials, have garnered considerable attention due to their abundance, renewability, and biodegradability. Starch, derived from various sources including corn, tapioca, and potatoes, is a natural polymer with the potential to replace petroleum-based plastics in various applications (Marichelvamet al., 2019).

The use of starch-based biopolymers in packaging applications aligns with the growing demand for eco-friendly solutions. With the increasing awareness of plastic waste pollution and the need to transition to a circular economy, starch-based materials offer a viable

alternative that can address these concerns. Their biodegradability ensures that, at the end of their lifecycle, they can break down naturally, minimizing the accumulation of persistent waste in the environment (Geyer et a., 2017).

Natural fibers, on the other hand, have shown promise as reinforcing agents in biodegradable composites, offering the potential to enhance the mechanical and functional properties of biopolymers. One of the primary reasons for exploring natural fibers in packaging is their renewable nature. Unlike synthetic materials that rely on fossil fuel resources, natural fibers can be sustainably sourced from agricultural crops or forestry by-products (John et al., 2019). This renewable characteristic ensures a reduced reliance on finite resources and contributes to the overall sustainability of packaging production. Moreover, natural fibers possess inherent biodegradability, making them an attractive option for eco-friendly packaging (Reddy et al., 2020).

The corn industry generates a substantial quantity of agricultural waste in the form of corn husks, which are discarded after corn kernels have been harvested (Wang et al., 2017). Corn husks are an abundant resource, with millions of tons generated each year globally. This underutilized resource represents a valuable opportunity for potential applications in biodegradable packaging. The incorporation of corn husk fibers into the TPCS matrix offers the opportunity to enhance the mechanical performance, water resistance, and overall functionality of the resulting composite material (Luo et al., 2018).

1.2 Problem Statement

Plastic waste has become a significant environmental concern in recent years. The extensive use and improper disposal of plastics have led to detrimental effects on ecosystems and human health. As a result, there is a growing need for sustainable and eco-friendly alternatives to conventional plastics (Davis et al., 2019).

Exploration of biodegradable materials, especially biopolymers derived from renewable resources, represents one potential solution. Due to its renewability, and biodegradability, starch has received considerable attention as a natural polymer. Nevertheless, the use of starch alone in thermoplastic applications has limitations that inhibit its widespread adoption (Zhang et al., 2018). Li et al. (2019) note that thermoplastic cassava starch (TPCS) has a number of shortcomings, including low mechanical properties, brittleness, and poor resistance to moisture absorption, which limit its practical applications. These drawbacks have stimulated significant research interest in augmenting the properties of TPCS through the addition of reinforcing agents and additives.

In addition, the corn industry generates a substantial quantity of agricultural waste in the form of corn husks. Corn husks, which are discarded after corn kernels have been harvested, are a valuable yet mainly untapped resource for potential applications (Wang et al., 2017). Not only does the underutilization of corn husk fibre contribute to environmental issues regarding waste accumulation and disposal, but it also represents a missed opportunity to capitalise on its inherent mechanical and functional properties.

The primary purpose of this investigation is to investigate the use of corn husk fibre as a reinforcing material to improve the properties of TPCS. By incorporating corn husk fibre into the TPCS matrix, it is anticipated that the resultant composite material will enhance the mechanical and thermal performance, and overall functionality. This study seeks to

maximise the utilisation of corn husk fibre resources and contribute to the development of sustainable and environmentally friendly biodegradable materials.

1.3 Research Objective

The main objectives of this study are:

a) To prepare a composite material out of biodegradable thermoplastic cassava starch reinforce with corn husks fiber.

b) To investigate the mechanical properties, thermal properties, and the influence of fibre on a biodegradable thermoplastic cassava starch composite that is reinforced with corn husk fibres.

c) To fabricate biodegradable packaging by using cassava starch and fibre from corn husks.

1.4 Significance of Study

a) The study enhances understanding of biodegradable thermoplastic cassava starch composites reinforced with corn husk fiber.

b) The investigation introduces an innovative application of corn husk waste as a reinforcement material for biopolymer composites, offering potential solutions for waste management.

c) The development of biodegradable polymers with improved properties addresses environmental concerns and provides alternatives to natural fiber-based polymers.

1.5 Scopes and Limitation of Study

The justification of this study are as follows:

i. The thermoplastic cassava starch mixture was generated by mixing food grade cassava starch and glycerol as plasticizer in the correct formulation proportion (100:30).

ii. Palm wax is added to a combination of cassava starch and glycerol at the required formulation proportion (10:90).

iii. The percentage of corn husk fibre used is 0%, 10% 20%, 30%, 40%, and 50%.

iv. A hot press machine at 155°C made the thermoplastic starch composite with palm wax reinforcement and corn husk fibre.

v. The mechanical testing that will be use are tensile test and flexural test. While thermal testing is Thermogravimetric Analysis (TGA). And other testing is Scanning Electron Microscope (SEM) and Fourier Transform Infrared Spectroscopy (FT-IR).

1.6 Structure of Thesis

This thesis is organised according to the format specified by Universiti Teknologi Malaysia Melaka (UTeM), which is based on this study's publication. This report is divided into four sections: introduction, literature review, methodology and preliminary results. The following are the structure's specifics:

Chapter 1

This chapter describes the objective of the research and highlights the problem that prompted the study. In this chapter, the significance and scope of the study and activity were elaborated.

Chapter 2

This chapter justifies the total literature review conducted by previous research that is relevant to the topic of this thesis. In addition, this chapter describes the research gaps identified through a review of prior research.

Chapter 3

This chapter describes the methodology used in this study for the preparation of materials, testing procedure, and data acquisition.

Chapter 4

This chapter provided a comprehensive explanation of the mechanical and thermal result underlying the thermoplastic cassava starch reinforced by corn husk fiber composite. Additionally, the chapter presented a summary of the evaluated testing results and discussion.

Chapter 5

This chapter provide conclusion achieved from this study as well as recommendation to improve better result. In this chapter potential of the product also been recorded.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter provides an extensive review of the relevant literature related to composite materials, natural fibers, corn husk, starch, polymers, and the effects of natural fibers on mechanical, thermal, and other analysis. A comprehensive understanding of these topics is crucial for the subsequent investigation and analysis presented in this thesis. The growing concern for environmental sustainability has spurred the search for alternative packaging materials that are biodegradable and eco-friendly. Utilising natural fibres for packaging to their full potential, however, presents several challenges. Optimising fibre processing methods, improving compatibility with other biodegradable materials, ensuring adequate barrier properties, and maintaining mechanical strength over the course of the product's lifecycle are some of these challenges. This study aims to contribute to the development of sustainable packaging solutions that satisfy the needs of both industry and the environment by examining the properties, processing techniques, and performance of natural fiber-based composites. The results of this study will open up new possibilities for natural fiber-based packaging materials and pave the way for a more environmentally friendly and sustainable future for the packaging sector.

2.2 Composites

A composite is a mixture of two or more phases, one of which is the matrix stage and the other the reinforcement stage on a tiny scale, intended to have superior mechanical

performance and physical and chemical properties than the constituent materials, which cannot be achieved independently (Kumar et al., 2021). Composites are made of two main components which are fiber or filament reinforcement and matrix. Figure 2.1 shows the illustration of composite components.

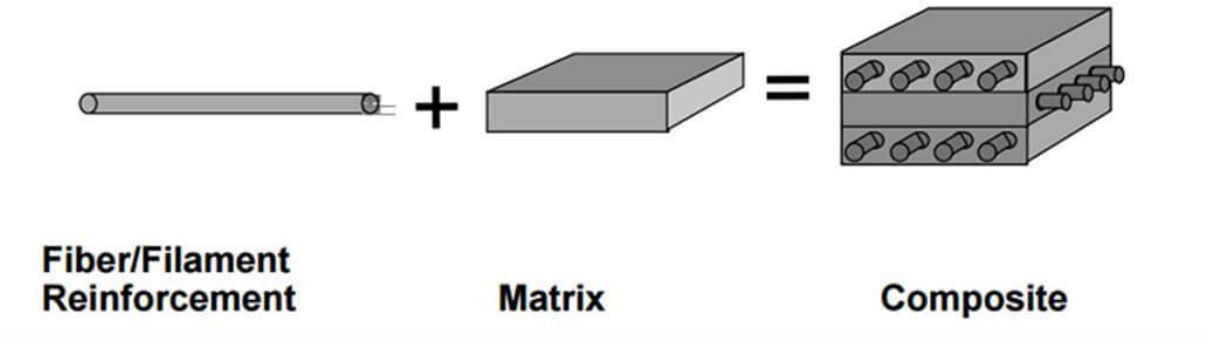


Figure 2.1 Formation of Composite (Vedith et al., 2022).

Reinforcing material provides strength and stiffness, while matrix material holds reinforcing material in position and transfers stresses between reinforcing material (Hull et al., 2019).

Composites offer a high strength-to-weight ratio, making them well-suited for weight-saving applications in industries such as aerospace and automotive (Zhang et al., 2021). Composites are typically more durable than traditional materials and require less maintenance over their lifespan. They are also highly resistant to environmental degradation and can withstand exposure to harsh chemicals and UV radiation. Moreover, the use of composites can lead to reduced manufacturing costs and improved production efficiency (Askeland et al., 2019).

2.2.1 Classification of Composite Materials

Composites are a type of material that is created by combining two or more distinct materials, each of which possesses its own set of features, in a manner that results in an

improvement in the composite's overall performance. There are many different kinds of composites, but the one that is used the most frequently is called a Polymer Matrix Composite (PMC). This type of composite comprises a polymer matrix strengthened with fibers or particles and can be classified into two types: thermosetting and thermoplastic (Ashby & M. F., 2013). Metal Matrix Composites (MMCs) and Ceramic Matrix Composites (CMCs) can be distinguished by the type of reinforced material used in the matrix. The matrix reinforced material can be metal or ceramic (Gupta et al., 2015; Singh et al., 2015).

On the other hand, Carbon Fibre Reinforced Composites (CFRCs), Glass Fibre Reinforced Composites (GFRCs), and Natural Fibre Reinforced Composites (NFRCs) are distinguished from one another by the reinforcing material that is utilised, specifically carbon fibres, glass fibres, and natural fibres (Hearle et al., 2013; Kumar & K. S., 2015; Thakur et al., 2014). There are numerous kinds of composite materials, each of which has a specific purpose that it serves. The qualities and applications that are necessary will guide the selection of composite materials.

2.2.2 Natural Fibre Reinforced Composited (NFRCs)

Natural Fiber Reinforced Composites (NFRCs) are a class of composites that utilize plant-based fibers as a reinforcing material in a polymer matrix. NFRCs have gained some popularity due to their potential to replace traditional synthetic fibre reinforced composites which are proven to be more sustainable and environmentally friendly (Thakur et al., 2014).

It has been demonstrated that NFRCs possess outstanding mechanical properties, such as high strength and stiffness, making them suitable for use in a variety of industries, including the automotive, construction, and packaging sectors. The mechanical properties of NFRCs are determined by a number of variables, including the type of fibre, fibre content,

fibre orientation, and matrix material. Jute, hemp, sisal, flax, and kenaf are among the most common natural fibres utilised in NFRCs. These fibres are inexpensive, broadly accessible, and possess superior mechanical properties (Bhatnagar et al., 2021). The fact that NFRCs are renewable and biodegradable makes this form of composite a viable alternative to more sustainable composites. Kumar et al., (2019) report that the use of natural fibres in composites helps to reduce reliance on nonrenewable resources.

However, there are challenges of using natural fibers as reinforcing material such as the inconsistencies of the mechanical properties and more prone to absorb moisture over time which can affect the composites (López-Cervantes et al., 2020).

2.2.3 Application of Composite

Composites have a wide range of applications due to its unique properties. Numerous applications, including those in the aerospace, automotive, construction, marine, and biomedical industries, have shown these materials to have enormous potential. A report from MarketsandMarkets (2021), transportation sectors is the largest consumer of composites, accounting for most of the market share due to the increasing demand for lightweight and fuel-efficient vehicles. The aerospace and defense sector are also expected to witness significant growth in the coming years due to the increasing use of composites in aircraft and defense applications.

The advantages and disadvantages of composites vary depending on what type and material of the composites itself. For example, carbon fiber reinforced composites have high strength, stiffness, and lightweight, making them suitable for aerospace applications, but they are brittle and can be expensive to produce (Todoroki & A., 2022). On the other hand, glass fiber reinforced composites have lower strength and stiffness than carbon fiber

reinforced composites, but they are more impact-resistant and cost-effective (Zhang et al., 2022). Similarly, natural fiber reinforced composites have the advantage of being renewable, biodegradable, and lightweight, but they have lower mechanical properties compared to synthetic fiber reinforced composites (Bhatnagar et al., 2021). Ceramic matrix composites have high temperature resistance and excellent wear properties, but they can be brittle and difficult to process (Rana et al., 2020).

2.2.4 Polymer Composite

Polymer composite is a composite where the matrix material is a polymer and the reinforcement is made of fibers, particles, or flakes to enhance the properties of the matrix alone (Mishra et al., 2019). Polymer composites have been used to develop packaging materials with enhanced mechanical strength, thermal stability, and barrier properties. Researchers have developed a polymer composite film containing cellulose nanocrystals and chitosan, which exhibited excellent mechanical properties and high oxygen barrier properties (Cazón et al., 2021). Nevertheless, the characteristics of polymer composites can be customized to fulfill the specific demands of diverse packaging applications.

2.3 Polymer

Polymer is a large molecule composed of repeating structural units, also known as monomers. Polymers are typically synthetic, and their properties and behavior are determined by the chemical composition of the monomers and the length and arrangement of the repeating units (Callister et al., 2018). Polymer materials, which can be derived from natural sources or synthesized, are extensively employed in diverse industries including

packaging, textiles, construction, and electronics, owing to their distinct characteristics and attributes. Polyethylene, polypropylene, polystyrene, and polyvinyl chloride are among the widely recognized synthetic polymers commonly used in various applications. Natural polymers such as DNA, silk, and cellulose are also widely used (Jia et al., 2022; Martínez-Abad et al., 2021; Pittenger et al., 2020).

Polymers are present in our daily lives in various forms and applications. Some common examples of polymers are plastics, rubber, fibers, adhesives, coatings, and films. Plastics are used in packaging, such as bottles, containers, and bags, as well as in household items like toys, furniture, and appliances. Rubber is used in tires, seals, and hoses, while fibers such as nylon and polyester are used in clothing, carpets, and ropes. Adhesives and coatings are used to bind and protect surfaces, while films are used in food packaging and window tinting. Overall, polymers play a significant role in our daily lives and have revolutionized the modern world with their versatile properties and applications (Sarwar et al., 2021).

2.4 Natural wax

Natural waxes have gained significant attention as potential reinforcements in composite materials. They offer unique properties and can enhance the performance of the composites in various applications. Researchers have conducted several studies investigating the use of natural waxes in composite materials.

Several studies have explored the impact of wax on natural polymers. Analysis using fourier transform infrared spectroscopy (FTIR) revealed the presence of interactions between the starch matrix and wax, facilitated by the formation of hydrogen bonding networks. These interactions resulted in improved water barrier properties, mechanical

strength, and unique characteristics, making them suitable for applications in edible food coatings and food packaging materials (Khazandi et al., 2015; Kamaruddin et al.,2020).

2.4.1 Palm wax

Palm wax is obtained from the fruit of *Elaeis guineensis*, a prominent crop in Malaysia, Indonesia, and Thailand, through the extraction of palm oil (Sahid et al., 2020). Palm wax, characterized by its high content of long-chain saturated fatty acids such as palmitic acid ($C_{16:0}$) and oleic acid ($C_{18:1}$), exhibits excellent hydrophobic properties. With a melting temperature range of approximately 58 – 60 ℃, it is well-suited for applications involving thermosensitive active compounds (Arcan & Yemenicioğlu, 2013). Due to its significant saturation, palm wax is less susceptible to conventional autooxidation that primarily targets double bonds, resulting in enhanced oxidative stability over an extended duration (Garrison & Dayan, 2011). As a result, palm wax is considered a cost-effective and environmentally friendly substitute for hydrophobic substances derived from fossil sources.

2.5 Fibre

There has been an uptick in interest in fibre-reinforced composites as a result of their high strength and stiffness, in addition to their low weight and competitive pricing. Natural fibres are one form of fibre that have been investigated at length for its potential use in composite applications. Natural fibres come from a wide variety of plant sources and offer a number of benefits, including the ability to be renewed, to be biodegradable, and to be inexpensive. On the other hand, synthetic fibres are man-made fibres that are not found in

nature but are generated through the process of chemical synthesis. Natural fibres cannot be synthesised. (Aprianingish et al., 2019).

2.5.1 Natural Fibre

Natural fibers can be categorized into three main groups: plant fibers, animal fibers, and mineral fibers (Thakur et al., 2014). A variety of natural fibres may be used as polymer reinforcements.

In natural fibers there are cellulose, and hemicellulose are both polysaccharides that make up the cell walls of plants. Hemicellulose is a branched polymer made up of various sugar units, including xylose, arabinose, and mannose, linked together by various glycosidic bonds, as opposed to cellulose, which is a linear polymer made up of glucose units connected by β (1→4) glycosidic bonds. Because cellulose and hemicellulose both give plant cell walls strength and rigidity, they are frequently used as sources of natural fibres for industrial applications (Gírio et al., 2010). Figure 2.2 and 2.3 shows chemical structure of cellulose and hemicellulose.

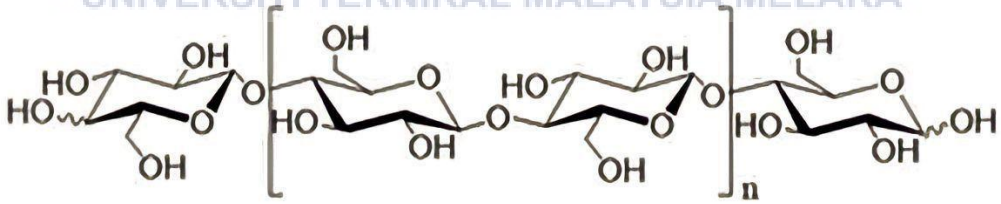


Figure 2.2 Chemical Structure of Cellulose (Istasse et al., 2021).

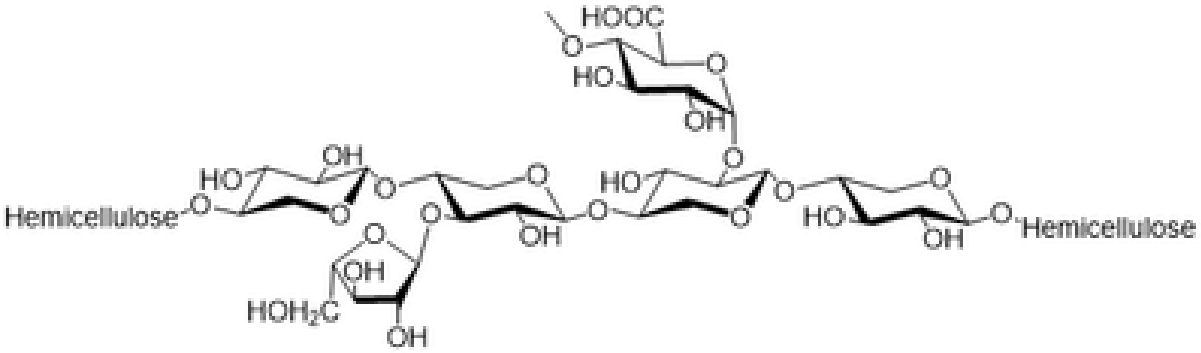


Figure 2.3 Chemical Structure of Hemicellulose (Istasse et al., 2021).

Research has been done on the possibility of using natural fibres as reinforcement in composite applications. For example, Raj et al., (2021) conducted research on the use of pineapple leaf fibres as reinforcement in a polyurethane matrix. They discovered that the addition of fibres resulted in a considerable improvement in the mechanical properties of the composite material. The composite's tensile strength, flexural strength, and impact strength all saw considerable improvements as a direct result of the incorporation of pineapple leaf fibres into the polyurethane matrix. Researchers believe that the increased tensile strength and stiffness of the pineapple leaf fibres, which enabled efficient stress transfer between the fibres and the matrix, were responsible for this improvement.In a similar vein, Similarly, Venkateshwaran et al., (2021) conducted a study on the utilization of banana stem fibers as a reinforcing agent in a polyester matrix, revealing substantial enhancements in the tensile and flexural properties of the composite. It should be noted that natural fibers exhibit considerable variation in terms of their quality, mechanical properties, and chemical composition (Singh et al., 2020), thereby influencing the ultimate characteristics of the composite material. This is the primary disadvantage of natural fibres.

According to Thakur et al., (2014), natural fibres have a low resistance to moisture as well as environmental deterioration, which can have a negative impact on their long-term

durability as well as their mechanical qualities. Synthetic fibres, on the other hand, demonstrate remarkable resistance to moisture, chemicals, and environmental degradation. This makes synthetic fibres more suitable for demanding applications that require high durability and reliability. Examples of synthetic fibres are glass fibres and carbon fibres.

2.5.2 Synthetic Fibre

Synthetic fibers are produced by chemically synthesizing polymers derived from petrochemicals. The process involves polymerization, spinning, drawing, and finishing. A study by Tang et al. (2021) investigated the mechanical properties of poly (lactic acid) (PLA) composites reinforced with carbon fiber, basalt fiber, and glass fiber. Figure 2.4 shows fiber sheets of basalt fiber sheet, carbon fiber sheet and E-glass fiber sheet.

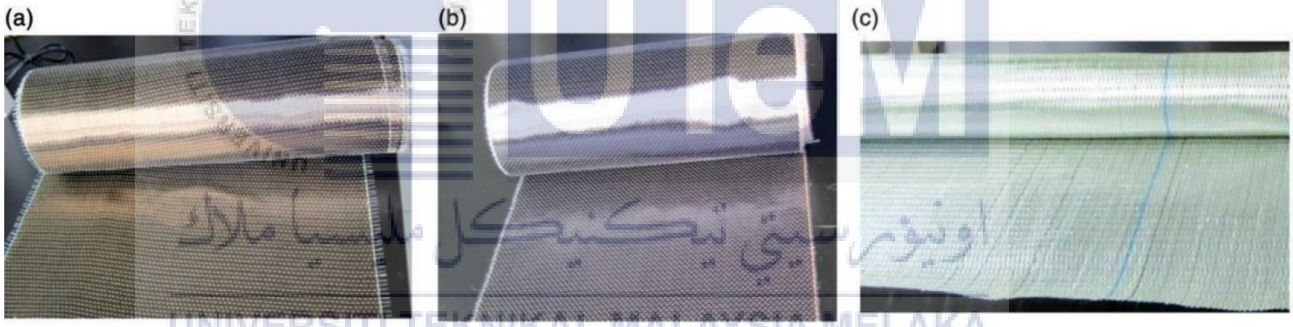


Figure 2.4 The unidirectional fiber sheets: (a) basalt fiber sheet; (b) carbon fiber sheet; (c) E glass fiber sheet (Wang et al., 2015).

The study discovered that the composite with carbon fibre reinforcement had the highest tensile strength, while the composite with basalt fibre reinforcement had the highest tensile modulus. Additionally, research has focused on combining synthetic fibres with natural fibres to obtain enhanced composite properties. Ashori et al., (2020) investigated the effects of incorporating carbon nanotubes (CNTs) into a kenaf fibre and polypropylene composite. The addition of CNTs enhanced the composite's mechanical properties, with a significant increase in both tensile strength and modulus, according to the study.

2.6 Corn Husk

Corn, also known as maize, is a type of cereal grain that is widely cultivated for its edible seeds. It is a staple food in many parts of the world and is used for a variety of purposes, including as animal feed, in the production of biofuels, and as a raw material for various industrial products. The plant itself has a distinctive leaf structure with large flat leaves arranged alternately on the stem and can grow up to 3 meters in height. Since maize can be grown in a wide range of climates and soil types, it is a crop that is very adaptable and whose cultivation has had a significant impact on the agricultural practices and economies of many parts of the world (Kadam et al., 2021).

2.6.1 Anatomy of Corn

Corn includes several parts such as the kernel or corn seed, which is the edible part of the corn. The cob, which is the part of the corn where the kernels are attached, is covered by the husk, which is composed of several layers of tough, fibrous tissue. The stalk of the corn plant supports the cob and leaves and is made up of nodes, internodes, and vascular tissue (Mukherjee et al., 2010). The corn husk refers to the corn ear's protective outer layer, also known as the husk or corn husk leaves. It is the leafy portion that encircles the corn cob and acts as a barrier during the corn plant's growth and development.

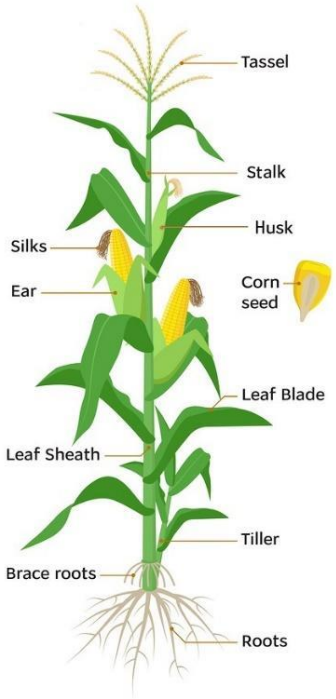


Figure 2.5 Anatomy of corn husk (Singh et al., 2020).

Figure 2.6 Corn Husk (Kumar et al., 2020).

The fibrous nature of corn husk lends itself to various industrial applications. It possesses attributes such as renewability, abundance, and biodegradability, making it an eco-friendly alternative to synthetic materials. The fibrous structure of corn husk provides strength, flexibility, and potential reinforcement capabilities, making it suitable for

incorporation into composite materials. Moreover, corn husk contains cellulose, hemicellulose, lignin, and other natural compounds that contribute to its unique properties. (Singh et al., 2020).

2.6.2 Corn Husk Fibre

Corn husk fibre is a natural fibre that is derived from the outer layer of corn kernels (Liang et al., 2021). It is a waste product from corn processing, making it a sustainable and eco-friendly material (Kumar et al., 2020). Corn husk fibre is rich in cellulose and hemicellulose, which make it suitable for use in the production of composites (Raj et al., 2020). The fibre can be extracted from corn husk through various methods, including chemical, mechanical, and enzymatic processes (Liang et al., 2021).

Corn husk fibre can be extracted using various methods. In chemical methods, lignin and other non-cellulosic components are removed using solvents, leaving only pure cellulose fibres. The corn husk is physically broken down to release the fibres using mechanical methods like ball milling and ultrasonic treatment. The non-cellulosic components are selectively broken down by enzymes in enzymatic methods, leaving only the cellulose fibres (Kumar et al., 2020).

2.6.3 Corn Husk Fibre Composite

Corn husk fibre has demonstrated potential as a reinforcing agent in composite materials. Composite materials consist of two or more materials that have been combined to produce a material with superior properties compared to its constituent parts. (Gupta et al., 2019).

According to Kumar et al. (2020), researchers reinforced polyethylene with corn husk fibre to produce composites. They discovered that the composite's tensile strength and Young's modulus increased in proportion to the fibre content. This suggests that the addition of maize husk fibre enhanced the mechanical properties of the composite, most likely due to the cellulose and hemicellulose present in the fibre, which provide strength and rigidity.

A study by Sarawati et al., (2021) use High Density Polyethylene (HDPE) and Polyolefin (POE) as matrices and corn husk fibre shows as the amount of corn husk fibre increased, the tensile strength and elongation at break decreased while the tensile modulus increased. The strength is slightly increased when compared to when there are no corn husk fibre fillers added, but as the amount of corn husk fibres increased the strength of composite is decreased. The result of the studies shows in Figure 2.5.

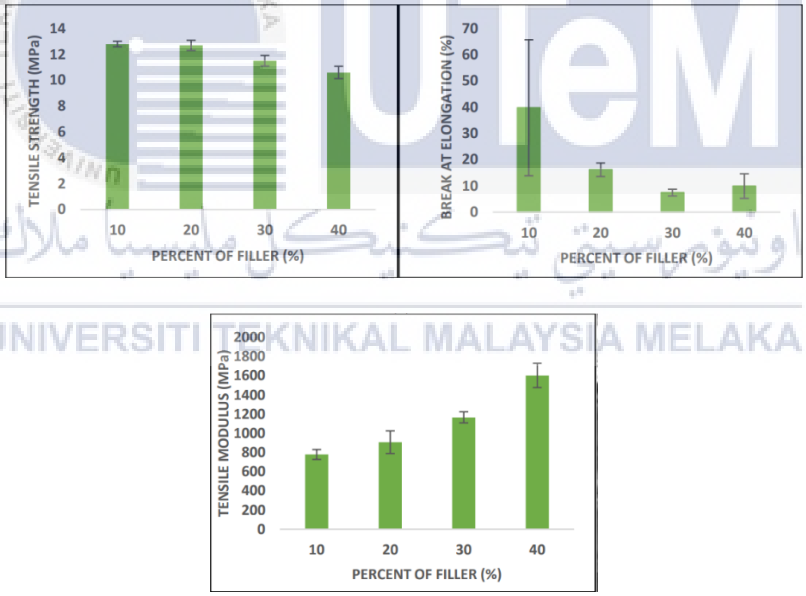


Figure 2.7 Mechanical Properties of Corn Husk Fibre Composites (a) Tensile Strength (b) Elongation at Break (c) Tensile Modulus (Sarawati et al., 2021).

On the other hand, Raj et al. (2020) created composites by adding corn husk fibre to polypropylene as reinforcement. However, they discovered that the tensile strength and Young's modulus of the composite decreased as the fibre content rose. This could result in

stress concentrations and eventual failure of the composite because of poor interfacial adhesion between the fibre and matrix. It's important to keep in mind that the use of different matrices (polyethylene vs. polypropylene) could also be a factor in the disparate outcomes of these two studies.

2.6.4 Application of Corn Husk Fibre and Corn Husk Fibre Composite

Corn husk is a waste product generated during the processing of corn. Raw corn husk has been utilized as a natural and sustainable material in different industries, such as papermaking, bioplastics, and textiles. corn husk can be used as a low-cost alternative to wood pulp for papermaking and reported that the paper made from corn husk had similar characteristics to conventional paper, with comparable tensile strength and burst strength (Yadav et al., 2019).

Raw corn husk has been traditionally used for medicinal purposes due to its antibacterial and anti-inflammatory properties. These antioxidants can be extracted from corn husks and used in the food and pharmaceutical industries. A study conducted by Sun et al. (2021) investigated the potential of corn husks as a source of natural antioxidants. The researchers extracted antioxidants from corn husks using a variety of methods, including ethanol extraction, microwave-assisted extraction, and ultrasound-assisted extraction. They found that all three extraction methods were effective in extracting antioxidants from corn husks, with ethanol extraction being the most efficient. The extracted antioxidants were found to have strong antioxidant activity, with the potential to be used as natural antioxidants in the food and pharmaceutical industries.

In addition to being a potential source of natural antioxidants, maize fibre can also be used as a reinforcement material in composite materials. Corn fibre is a natural fibre that can be combined with a polymer matrix to produce a composite material with improved

mechanical properties. Kumar et al. (2020) examined the static and dynamic mechanical properties of maize husk fiber-reinforced polyethylene composites. Researchers discovered that the tensile strength and Young's modulus of the composite increased as the maize husk fibre content increased. This indicates that maize husk fibres can be utilised to enhance the mechanical properties of polyethylene composites.

In a separate study, Raj et al. (2020) analysed the mechanical properties and water absorption behaviour of polypropylene composites reinforced with maize husk fibre. Researchers discovered that the tensile strength and Young's modulus of the composite decreased as the corn husk fibre content increased. However, the incorporation of maize husk fibre enhanced the composite's impact resistance and decreased its water absorption. This indicates that corn husk fibres can be used to enhance the properties of polypropylene composites.

2.7 Starch

Starch is a carbohydrate polymer that is commonly found in plants and serves as a source of energy for many organisms. It is produced through the process of photosynthesis in plants, where carbon dioxide and water are converted into glucose, which is then polymerized to form starch. Starch is widely used in various industries such as food, paper, textiles, and pharmaceuticals due to its unique properties and low cost (Yu et al., 2018).

Starch has gained much attention as a biodegradable and renewable material for producing packaging due to its abundance, low cost, and biocompatibility. A study by Zuo et al. (2020) reported the successful preparation of biodegradable packaging film by blending potato starch with polylactic acid (PLA) and nano-cellulose. The resulting composite showed enhanced mechanical properties, good water resistance, and biodegradability.

Amylose and amylopectin are the principal forms of starch. Amylose is a linear polymer of glucose units connected via α-(1→4) glycosidic bonds, whereas amylopectin is a branched polymer of glucose units connected via α-(1→4) and α-(1→6) glycosidic bonds (Bertoft, 2017).

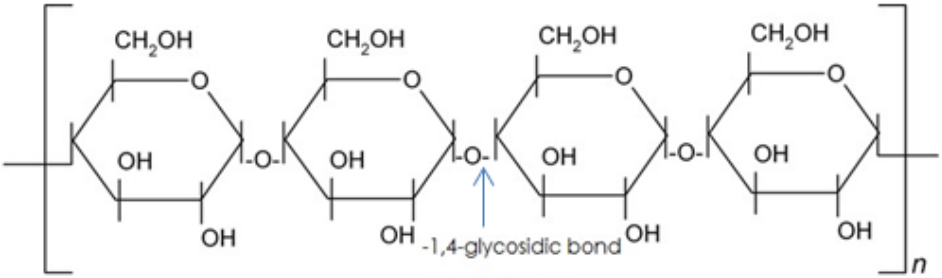


Figure 2.8 Chemical Structure of Amylose (Asharudin et al., 2018)

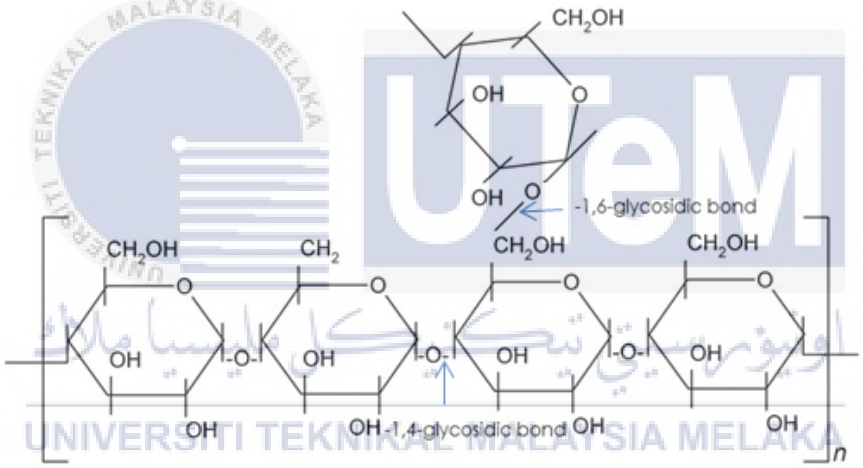


Figure 2.9 Chemical Structure of Amylopectin (Asharudin et al., 2018).

According to Bertoft (2017), amylose content in plant starch ranges from 15% to 30% generally, but some varieties can contain up to 80% amylose. Amylopectin on the other hand constitutes around 70% to 80% of the total starch content in a plant. A study by Zhang et al. (2017) shows that amylopectin content has a significant effect on starch properties. The study shows the higher amylopectin content can lead to higher viscosity, final viscosity, and starch gelatinization properties. Another study by Lie et al. (2019) found that the

amylopectin content of potato starch affected its retrogradation behavior, with higher amylopectin content leading to a slower retrogradation rate. The percentage of amylose and amylopectin content in various starches is shown in Table.

Table 2.1 Amylose and Amylopectin Concentration of Various Starch Sources (Marichelvam et al., 2019).

Source	Amylose Content (%)	Amylopectin Content (%)
Arrowroot	20.5	79.5
Banana	17	83
Cassava	18.6	81.4
Corn	28	72
Potato	17.8	82.2
Rice	35	65
Tapioca	16.7	83.3
Wheat	20	80

2.7.1 Thermoplastic Starch

Thermoplastic starch (TPS) has gained increasing attention in recent years due to its biodegradability, renewability, and potential to replace conventional plastics. Thermoplastic starch can be extracted and plasticized from various starch sources, including corn, potato, tapioca, and wheat (Bertoft & E., 2017).

Adding plasticizer such as glycerol, sorbitol, or polyethylene glycol to starch granules allows for the application of mechanical and thermal energy during the production of TPS. Plasticization is crucial as it turns TPS into a thermoplastic that can be shaped into various forms (Rachtanapun P. et al., 2020). Plasticizers play an essential part in the

manufacturing of thermoplastic starch because they improve starch behaviour. Specifically, they do this by reducing the number of hydrogen bonds that exist within polymer chains while simultaneously increasing free volume. Additionally, this fosters molecular chain mobility and increases flexibility and processability (Diyana et al., 2021).

A study by Saari et al. (2019) investigated the effect of incorporating cellulose nanofibrils (CNFs) into TPS films on their mechanical properties. The results showed that the addition of CNFs improved the tensile strength and Young's modulus of the TPS films. In another study by Li et al. (2020), TPS was blended with polylactic acid (PLA) and glycerol to improve its mechanical and barrier properties. The addition of PLA and glycerol increased the tensile strength, elongation at break, and water vapor barrier properties of the TPS film. The study also found that the TPS/PLA/glycerol blend had good biodegradability.

2.7.2 Thermoplastic Cassava Starch

Thermoplastic cassava starch (TPCS) has gained attention as a potential alternative to synthetic thermoplastics due to its biodegradability and renewability (Sudaryanto et al., 2019). TPCS is derived from cassava starch, which is extracted from the roots of the cassava plant and can be used as a matrix in composite materials. The use of TPCS in composites has been found to improve their mechanical properties.

Thermoplastic cassava starch is produced by modifying the starch molecules through physical, chemical, or enzymatic processes to enhance its thermoplasticity and mechanical properties. Modified cassava starch offers versatility in its processing, as it can be utilized in conventional plastic manufacturing methods like extrusion and injection molding,

enabling the production of diverse items including packaging materials, disposable utensils, and even toys (Fakhruddin et al., 2021).

A study by Oktaviana et al. (2021) investigated the effect of TPCS on the mechanical properties of polylactic acid (PLA) biocomposites. The addition of TPCS to the PLA biocomposites resulted in an increase in tensile strength and modulus, as well as an improvement in the impact strength of the composites. The researchers attributed these improvements to the good adhesion between TPCS and PLA, as well as the formation of hydrogen bonds between the two materials.

Similarly, another study by Chen et al. (2018) reported that the addition of TPCS to poly (butylene adipate-co-terephthalate) (PBAT) composites resulted in an increase in tensile strength, modulus, flexural strength, and elongation at break. The researchers attributed these improvements to the good compatibility and interfacial adhesion between TPCS and PBAT.

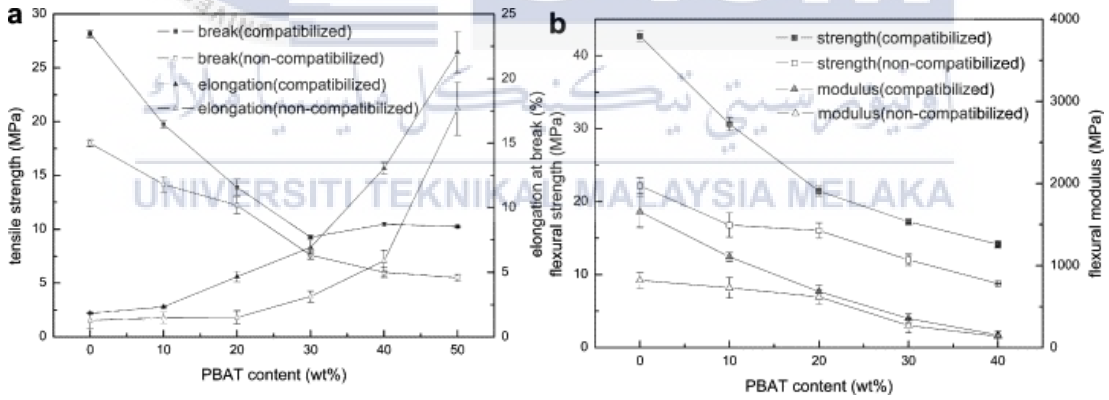


Figure 2.10 Mechanical Properties of TPCS with PBAT (a) tensile strength and elongation at break (b) flexural strength and flexural modulus.

According to a study by Nazrin et al. (2020), adding TPS to PLA100 reduced its tensile strength by 60% with just a 20% addition of TPS. The chemical incompatibility of PLA100 and TPS is well known, and TPS may have made the PLA100 blend more brittle.

The values kept dropping as TPS was added for the elongation at break, which is similar. Nevertheless, the Young's modulus seemed to be enhanced by the increasing addition of TPS. Figure shows mech properties.

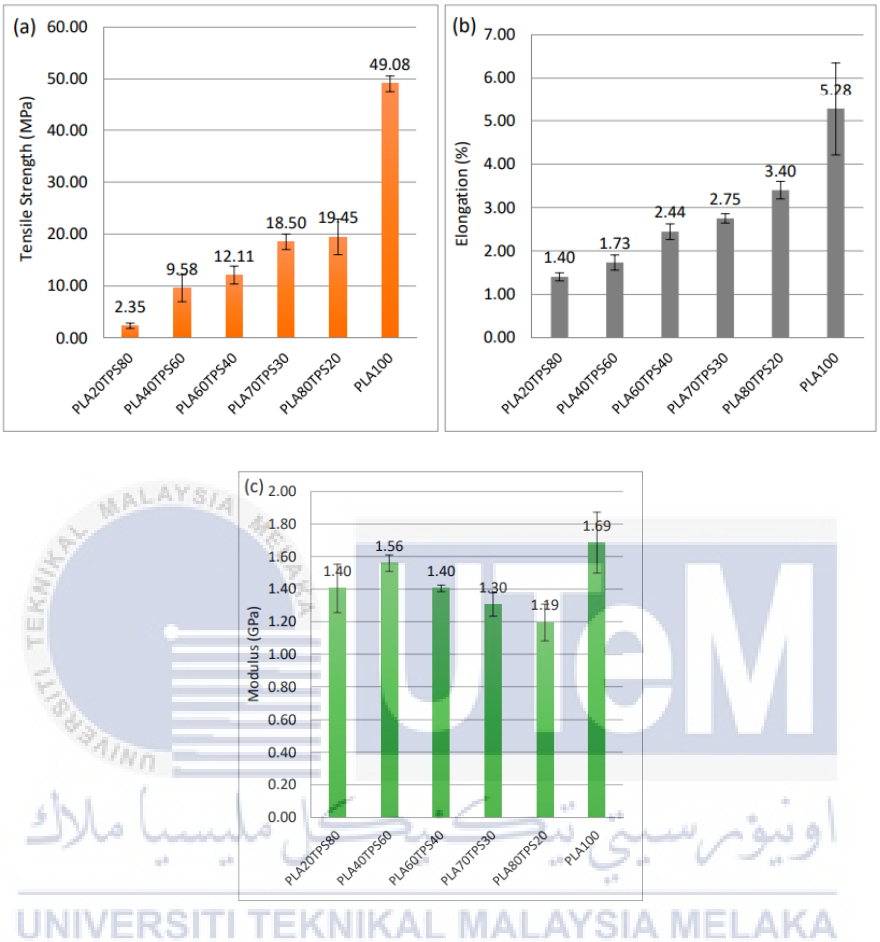


Figure 2.11 (a) Tensile strength, (b) elongation at break, (c) Young's modulus of PLA100 and PLA/TPS blend bionanocomposites (Nazrin et al., 2020).

Figure 2.12 depicts the SEM images of the fractured tensile surfaces of PLA100 and the PLA/TPS blend bionanocomposite. In general, the SEM images demonstrated that by combining glycerol, sorbitol, and water as plasticisers, good dispersion was attained.

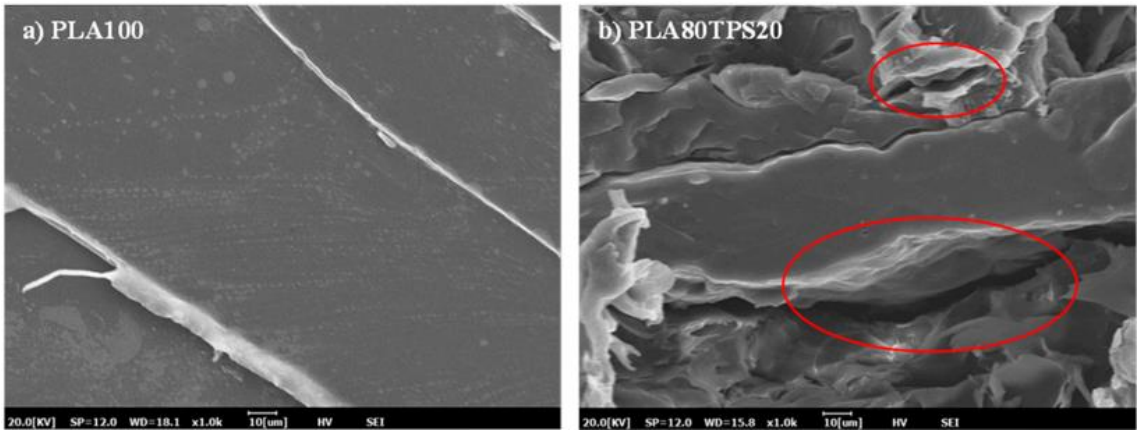


Figure 2.12 SEM images of tensile fractured surfaces for (a) PLA100, (b) PLA80TPS20.

One of the advantages of thermoplastic cassava starch is its biodegradability, which makes it a sustainable alternative to conventional plastics. When disposed of in the environment, thermoplastic cassava starch can be broken down by microorganisms into natural compounds, such as carbon dioxide and water. However, the rate of degradation depends on various factors, such as the composition of the bioplastic, the conditions of the disposal site, and the presence of microorganisms (Moura et al., 2021).

Overall, these studies suggest that TPCS has potential as a reinforcing agent in composite materials. Its good adhesion and compatibility with other materials can lead to improvements in the mechanical properties of the composites. Further research is needed to explore the full potential of TPCS in composite materials and to optimize its use in various applications.

2.7.3 Thermoplastic Starch Composite

Table 2.2 Potential applications of cassava starch composite.

Type of Starch	Type of Filler	Potential Application	Ref.
Cassava	Kraft	Biodegradable tray with chitosan coating	Campos et al., 2018
Cassava	Orange, sugarcane	Biodegradable tray, packaging material	Ferreira et al., 2020
Cassava	Cogon grass	Biodegradable material	Ilyas et al., 2019
Cassava	Sugar palm	Biodegradable polymer	Campos et al., 2018
Cassava	Grape stalks	Food packaging plastic	Engel et al., 2019
Cassava	Cassava bagasse	Food packaging plastic	Paula et al., 2019

2.8 Effect of Natural Fibre on Mechanical Analysis

Understanding the mechanical characteristics of the composite material will aid in determining the proper application and the resistance of the composites in particular applications.

2.8.1 Tensile Test

Tensile testing can help evaluate the effectiveness of natural fibers as reinforcement in composites by providing information on the tensile strength, Young's modulus, and other mechanical properties of the composite material. By comparing the mechanical properties of a composite material with different natural fiber content or processing methods, researchers can optimize the composite formulation to achieve the desired properties.

Widiastuti et al. (2021) conducted a study to examine the impact of fiber loading on the tensile strength and modulus of elasticity in a composite material. The average values of

tensile strength and modulus were obtained from five specimens per treatment, with a standard deviation ranging from 1.05 to 1.52 N/mm^2 and 26.7 to 53.1 N/mm^2, respectively. The findings indicated that as the fiber loading increased, the tensile strength of the composite decreased. The composite without bamboo fiber exhibited the highest tensile strength of 8,298 N/mm^2, while the r-HDPE composite with 30% bamboo fiber demonstrated the lowest tensile strength of 4,880 N/mm^2. In contrast, as the fraction of fibre in the r-HDPE matrix increased, so did the elasticity modulus. This suggests that a higher proportion of bamboo fibre results in a composite that is both more rigid and brittle. According to the article, the potential causes for the low tensile strength of this r-HDPE/bamboo composite include the dearth of adhesives, the use of randomly oriented bamboo fibres, and incompatible barrel temperatures.

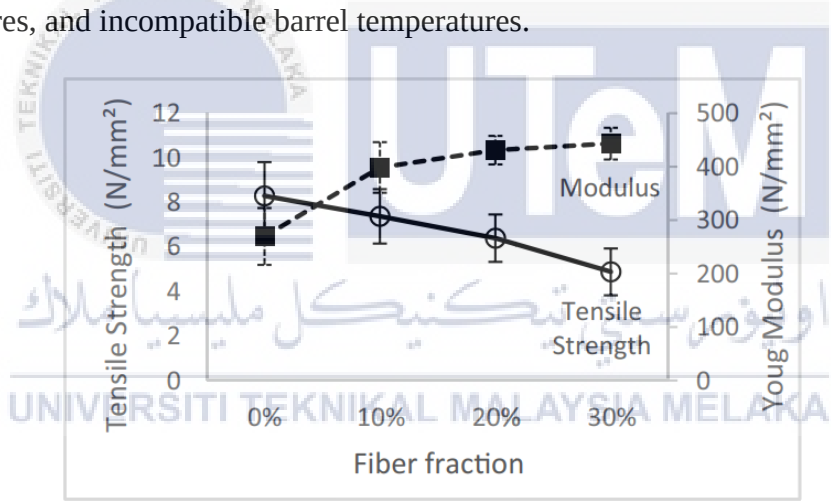


Figure 2.13 Tensile strength and Young modulus of bamboo composite (Widiastuti et al., 2021).

Similarly, to a study by Ku et al. (2011) that investigates the effect of hemp fiber content and anisotropy on the tensile properties of nonwoven mats made from polypropylene (PP) and hemp fibers. The nonwoven mats are hot pressed to create composite materials. The tensile strength of composites with fibers perpendicular to the load direction tends to decrease with increasing hemp fiber content, up to 34% at 70% hemp content, while

composites with fibers parallel to the load direction show an increasing trend. The tensile strength of composites with fibers in the parallel direction is 20-40% higher than those with fibers in the perpendicular direction. The presence of fibers oriented perpendicular to the load direction in the composite matrix does not contribute as load-bearing elements and can introduce defects and potential failure. On the other hand, specimens obtained from composite sheets parallel to the carding direction demonstrate improved tensile properties. The Young's modulus of composite materials tends to increase with the increase in the amount of fiber content. It reaches its maximum value at 50% hemp fibre loading and then decreases marginally when the loading reaches 70%. At a hemp fibre loading of 50%, the Young's modulus is nearly 1.5 times that of unadulterated PP without any fibre content.

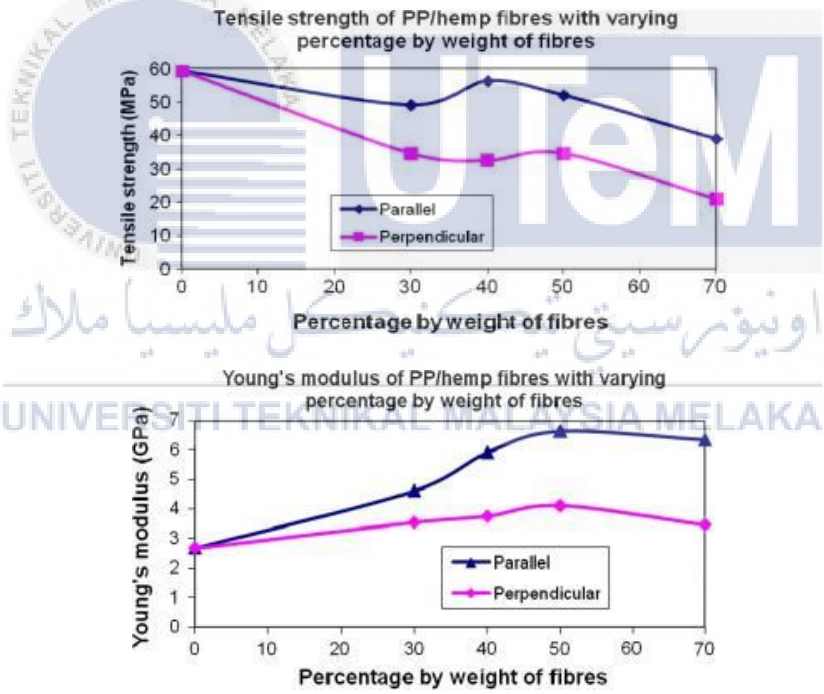


Figure 2.14 Mechanical properties of polypropylene with varying percentage by weight of fibres (a) Tensile strength (b) Young's modulus (Ku et al., 2011).

Mohamed et al. (2018) investigated the effects of fibre diameters and contents on the mechanical and physical properties of Mengkuang reinforced thermoplastic natural rubber

composites. The results demonstrated that the fibre size had a significant effect on the tensile strength, tensile strain at break, and tensile modulus of the composite material. For instance, at a fibre content of 10%, the tensile strength of the 125 µm, 250 µm, and 500 µm fibre sizes was 5%, 22.5 %, and 17.5% lower, respectively, than at a fibre content of 20%. This was due to the volume taken up by the presence of fibre at a low fibre concentration. Maximum tensile strength was demonstrated by a fibre content increase of up to 20% for a fibre size of 250 µm. The stronger surface interaction between the fibre and matrix contributed to less fibre agglomeration at this size and fibre concentration. Tensile strength decreased with the addition of up to 30% fibre content for particle sizes 125 µm, 250 µm, and 500 µm. In addition, the results demonstrated that, as the fibre size increased, the tensile strain at failure decreased for 10% fibre content. This was attributed to the weak internal interaction of larger fibres and their inability to withstand the load conveyed from the matrix.

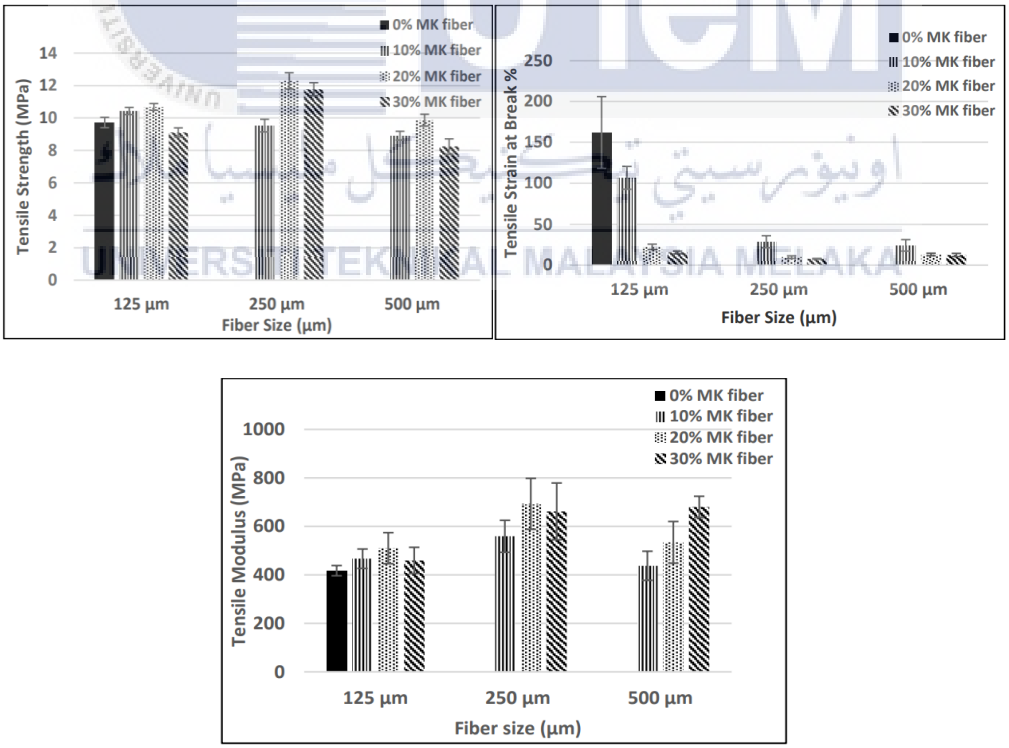


Figure 2.15 Effect of fiber size and fiber content (%) of mengkuang reinforced (a) Tensile strength, (b) Tensile strain at break, (c) Tensile modulus (Mohamed et al., 2018).

2.8.2 Flexural Test

Flexural strength, which evaluates a material's resistance to deformation under applied load, quantifies its capacity to withstand bending stress before failure occurs. Numerous factors, such as composition, microstructure, and processing conditions, contribute to the flexural strength of a material (Wang et al., 2021).

The flexural strength values increased steadily until they reached 20% fibre volume fraction (Vf). Beyond 20% Vf, the flexural strength increased abruptly, but then decreased drastically at 30% Vf because the fibre content was too high for the matrix to adequately fill the surrounding fibres. Maximum flexural strength was determined to be 33.45 MPa for a 10% Vf composite, 38.14 MPa for 15% Vf, and 75.29 MPa with a fibre length of 150 mm for a 25% Vf composite. The length and composition of the fibres affected the maximal flexural strength, and the study found that longer fibres carried more weight than shorter ones. The flexural modulus values also increased with increasing volume fraction, reaching a maximum of 15.99 GPa at 25% Vf and 150 mm fibre length, before decreasing to 6.72 GPa at 30% Vf. The study revealed that the strength and modulus of the isophthalic polyester resin-reinforced composite increased steadily with chopped snake grass fibres up to 25% Vf, whereas higher fibre loading increased the probability of microcrack initiation. The composite's strength was dependent on a number of variables, including the fiber's strength and modulus, fibre length, fibre and matrix density, fibre volume fraction, fibre weight content, and fibre orientation. In general, fabrication of the composite should take into account the fibre volume fraction and orientation across various fibre lengths (Sathishkumar et al., 2012).

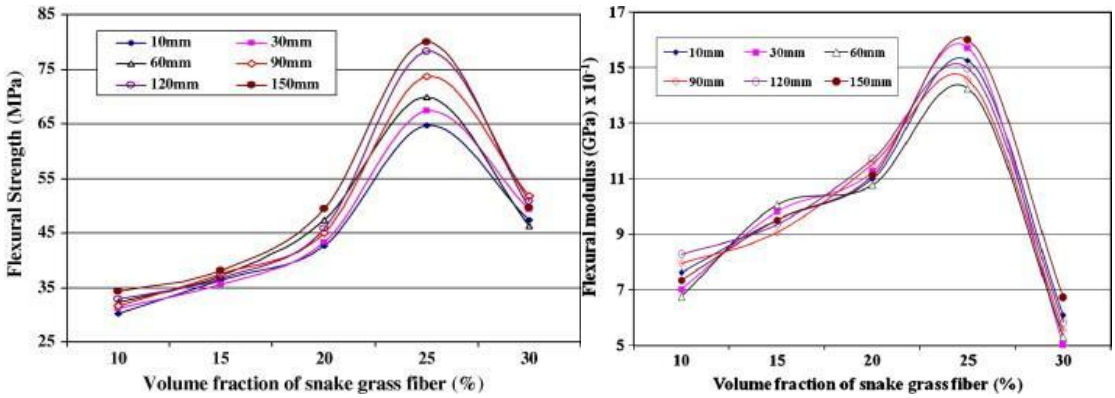


Figure 2.16 Effect when volume fraction of snake grass fiber with different fiber lengths (a) Flexural strength, (b) Flexural modulus (Sathishkumar et al., 2012).

The study by Elanchezhian et al. (2018) measured the flexural properties, including flexural strength and flexural modulus, of hybrid polypropylene composites reinforced with raw coir and jute fibers. The flexural strength increased as the fiber loading increased, likely due to the better entanglement of the polymer chain with the filler, which improves the filler matrix adhesion as the filler content increases. Similarly, the flexural modulus also increased as the fiber loading increased because both coir and jute are high modulus materials, meaning that higher fiber concentration requires higher stress for the same deformation. Therefore, incorporating rigid coir and jute fibers into the soft polypropylene matrix results in an increase in the flexural modulus.

Radzi et al. (2018) conducted research on the flexural strength and modulus of hybrid roselle (RF)/ sugar palm fibre (SPF)/ thermoplastic polyurethane (TPU) hybrid composites. Their findings showed that the strength and modulus of the hybrid composites decreased when the fraction of SPF increased. Both the strength and the modulus values were best displayed by the RS-1 hybrid composite. It is possible that the same elements that boost tensile strength are also responsible for the rise in flexural strength. However, non-uniform

interfacial bonding, poor distribution of fibres between RF and the SPF and matrix, and voids can all contribute to a loss in strength and modulus.

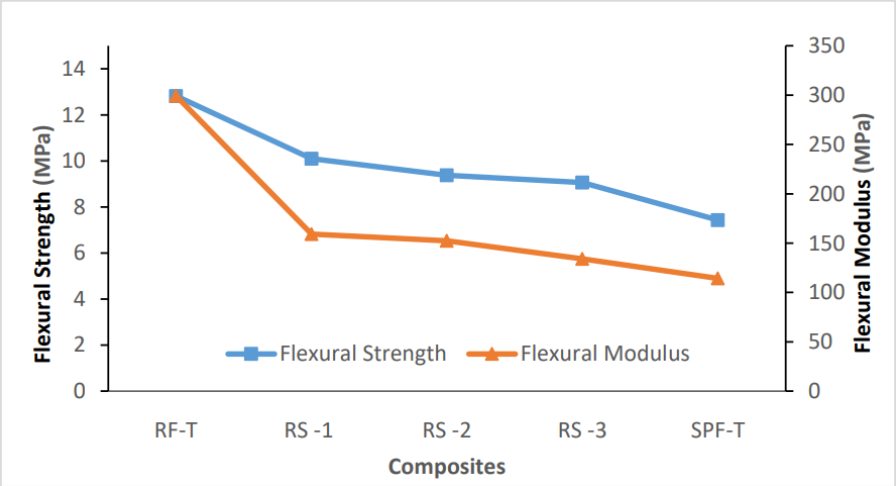


Figure 2.17 Flexural strength and modulus of RF/SPF/TPU hybrid composites (Radzi et al., 2018).

A reduction in flexural strength has also been documented by other authors, and they attribute it to a lack of wettability between the fibres and the matrix. In addition, the flexural strength and modulus of the single fibre SPF/TPU composites declined more than those of the RF/TPU composites, which is indicative of the SPF/TPU composites' lacklustre performance in the flexural strength department. It is possible that the aggregation of fibres will lead to weaknesses in the interface between the fibres and the matrix, which will ultimately result in a reduction in the flexural strength.

2.9 Effect of Natural Fibre on Thermal Analysis

Any scientific or technological description of a material in which temperature is changed as an experimental parameter is referred to as thermal analysis. But this term has

long been restricted to particular methods involving thermogravimetric and caorimetric effects (Nurazzi et al., 2021).

2.9.1 Thermogravimetric Analysis (TGA)

Thermal analysis is a test used to assess how a material will change chemically, physically, and structurally as a result of a temperature change. In general, the majority of chemical reactions, physical properties, and structural transformations are influenced by temperature, which is a fundamental state variable (Nurazzi et al., 2021).

In a study conducted by Widiastuti et al. (2022), the thermal performance of bamboo fibre immersed for 6 hours in a 10% NaOH solution was examined. Below 200°C, between 200 and 490°C, and above 490°C are depicted as temperature regions on the graph. Below 200 degrees Celsius, the composite's weight reduction is relatively stable. In the temperature range of 200–490°C, however, the weight loss differs depending on the fibre loading. At 490°C, the materials devoid of bamboo fibre lost the most weight (94.88%), while the composite with the maximum fibre loading lost the least (80.38%). The temperature of the residual material was higher than 490℃. The temperature at which a substance begins to degrade is indicative of its thermal stability.

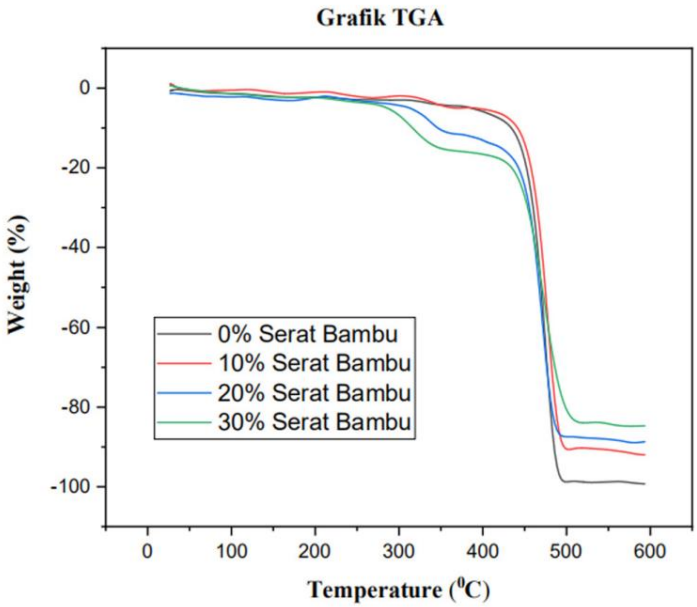


Figure 2.18 Graph of TGA (Widiastuti et al., 2022).

Fique fibers are heated at 10°C/min rate. The curve shows three regions of weight loss for fique fibers, each corresponding to a different temperature range. The first region (around 60-100 °C) corresponds to the evaporation of water that is present on the surface of the fibers. The second and third regions (250-350 °C and 350-600 °C, respectively) correspond to the decomposition of the fiber constituents. The DTG curve shows the rate of weight loss as a function of temperature. It is used to identify the temperature at which the highest rate of weight loss occurs, which is known as T_{max}. The first peak in the DTG curve for Fique fibers corresponds to the T_{max} of hemicellulose, which is a type of carbohydrate that is present in the fibers. The second peak corresponds to the T_{max} of α-cellulose, which is another type of carbohydrate that is also present in the fibers.

The residual weight of the fique fibers is the weight of the remaining material after it has been heated to a specific temperature. In this case, the residual weight is measured at 600 °C and is equal to 15.7%. This indicates that the Fique fibers have good thermal stability

and can withstand high temperatures without significant weight loss or degradation (Hidalgo-Salazar et al., 2018).

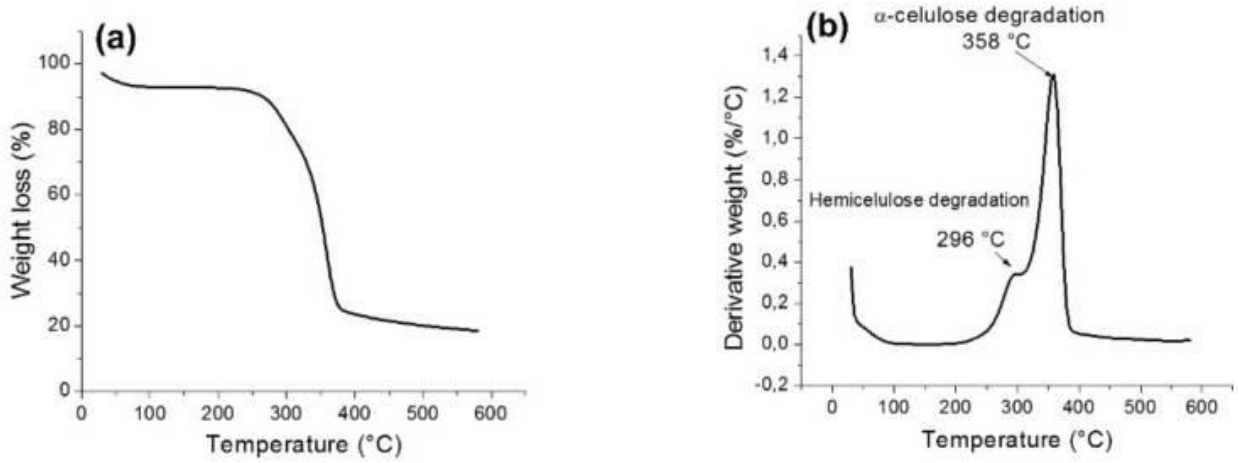


Figure 2.19 (a) Thermogravimetric, (b) Derivative Thermogravimetric (Hidalgo-Salazar et al., 2018).

2.10 Effect of Natural Fibre on Other Analysis

2.10.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a technique used to visualize the microstructure of materials at high magnification by using a focused beam of electrons. As the electron beam scans the sample's surface, different signals, including secondary electrons, backscattered electrons, and X-rays, are produced because of the interaction between the electrons and the sample. An image of the sample's surface is produced using the signals that are discovered (Li et al., 2021).

Rosamah et al., (2016) studied the hybrid bio composite's tensile fracture surface was identified using SEM. Figure 2.22 depicts SEM images of the fracture surface of a natural fiber-reinforced polyester composite containing nanoparticles of oil palm shell (OPS). Surprisingly, the nanoparticles were not visible, indicating effective integration with

the matrix. In the absence of fillers, weak adhesion caused fiber detachment and void formation (Figure 2.22a). Adding 1% OPS nanoparticles improved surface smoothness but still exhibited poor adhesion (Figure 2.22b). Increasing nanoparticle loading to 3% enhanced adhesion and reduced voids (Figure 2.22c), while 5% loading led to more fiber detachment and voids (Figure 2.22d). These findings suggest the influence of OPS nanoparticle content on fiber wetting and adhesion.

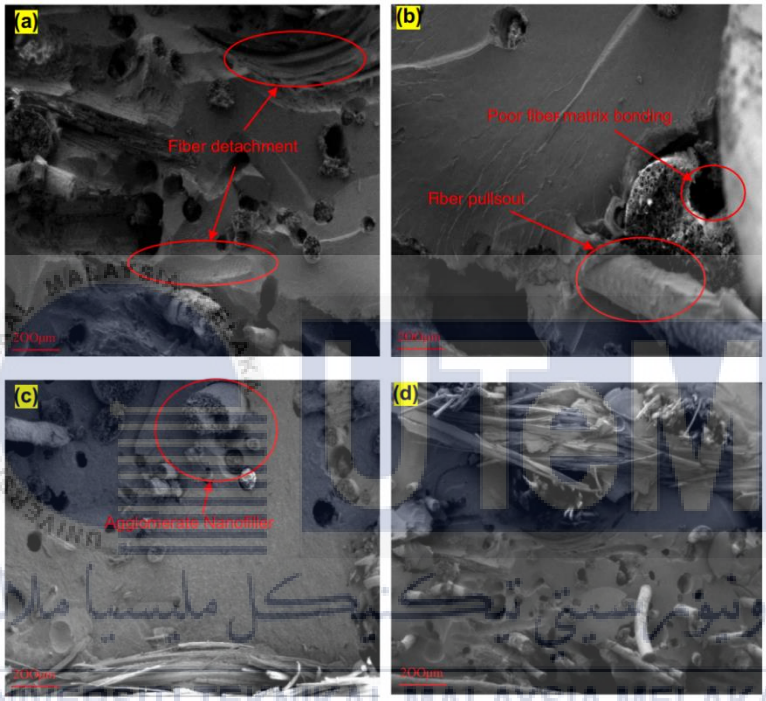


Figure 2.20 SEM images of natural fiber-reinforced hybrid composite with the incorporation of OPS nanoparticles. (a) Control; (b) 1 wt.% OPS nanoparticles (c) 3 wt.% OPS nanoparticles; (d) 5 wt.% OPS nanoparticles (Rosamah et al., 2016).

The study conducted by Sivakumar et al. (2022) investigated the influence of banana leaf fiber (BLF) on the mechanical and thermal properties of thermoplastic cassava starch (TPCS). Figure 2.23 displays the SEM image of TCPS reinforced with 10% to 80% BLF. At 10% BLF, the matrix and fiber exhibited strong adhesion, resulting in a homogeneous surface (Figure 2.23a). Signs of fiber breakage were observed in Figures 2.23b and 2.23c, with TCPS covering the fiber surface. This suggests the presence of strong hydrogen

intermolecular interactions between TCPS and the filament. As the fibre content increased to 40 percent, the composite structure changed significantly. Figures 2.23f to 2.23h show broken fibers and a porous surface. Similar observations were made in Figure 2.23g, which depicted TPCS with 10% BLF and a porous surface appearance. The presence of TPS on the surface of banana fibers was clearly observed, as evidenced by the visible fiber shattering. The increase in tensile strength further supported TPS as a suitable matrix for natural fibers.

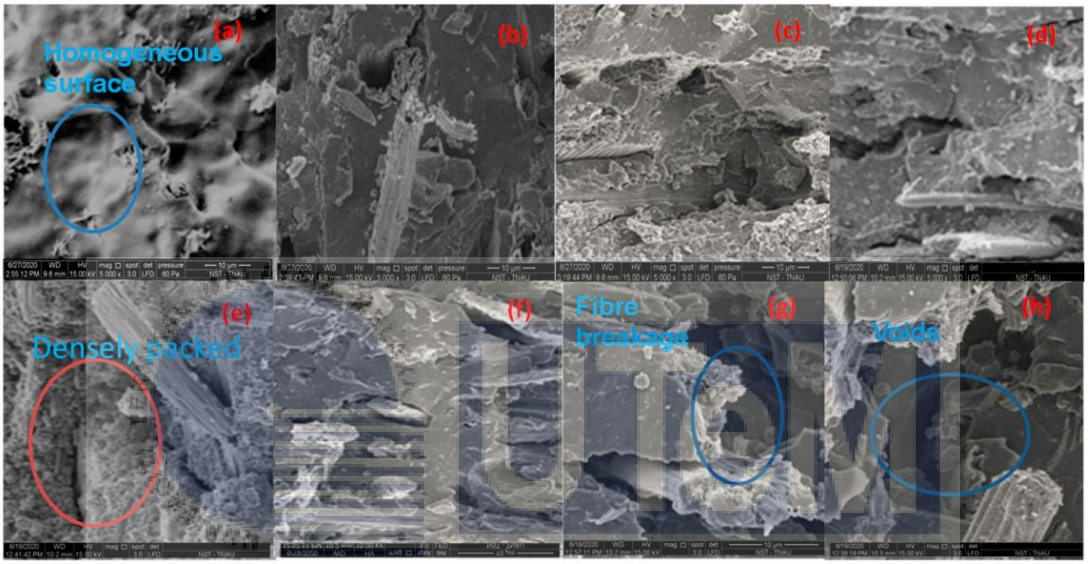


Figure 2.21 SEM image of TPCS with (a) 10% BLF (b) 20% BLF (c) 30% BLF (d) 40% BLF (e) 50% BLF (f) 60% BLF (g) 70% BLF (h) 80% BLF (Sivakumar et al., 2022).

2.10.2 Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR analysis is a method for examining a substance's chemical make-up and molecular structure. It measures the interaction between the infrared radiation and the sample, giving important details about the functional groups that are present in the substance. In order to measure the sample's absorption and transmission of light at various wavelengths, infrared light is used in the technique. Specific functional groups can be identified by contrasting the obtained spectrum with reference spectra (Smith et al.,2021).

Figure 2.23 depicts the FT-IR spectra of native sugar pal starch (SPS), agar, and thermoplastic SPS composites. Native SPS and agar exhibited similar spectra, with broad bands indicating hydrogen-bonded hydroxyl groups (OH) and characteristic peaks for various functional groups. In the SPS spectrum, peaks related to C=O bond stretching, and anhydro-glucose ring were observed. Agar showed peaks associated with 3,6-anhydro-galactose. The interaction between components in polymer blends was determined based on the shift in peak positions, indicating the strength of interaction. The spectrum of thermoplastic SPS showed lower wavenumbers for OH stretching and bending compared to native SPS, indicating enhanced hydrogen bonding with glycerol. The addition of agar did not significantly affect the IR peak positions, suggesting compatibility and increase intermolecular hydrogen bonding between agar and SPS (Jumaidin et al., 2016).

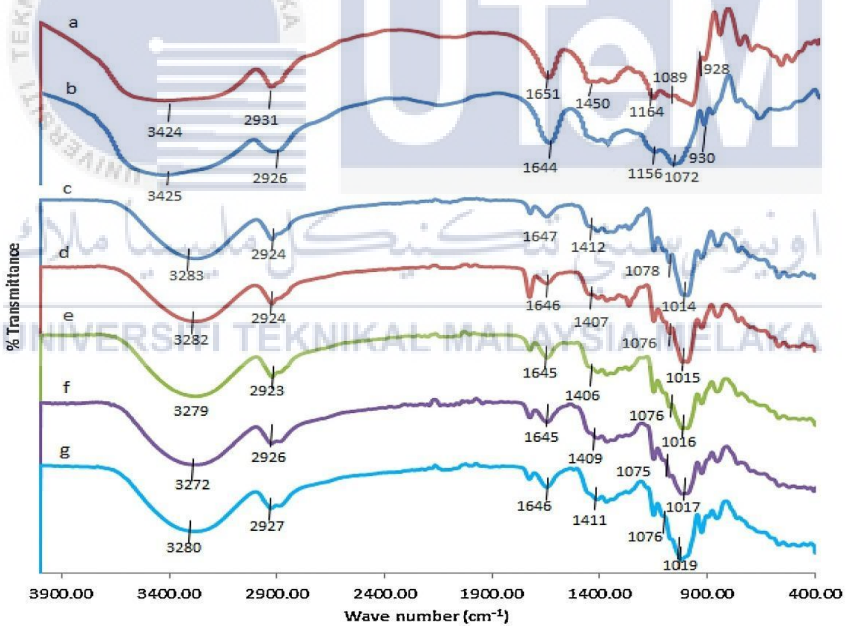


Figure 2.22 FT-IR spectra of (a) native SPS and (b) native agar (c) neat SPS matrix (d) 10% agar (e) 20% agar (f) 30% agar (g) 40% agar (Jumaidin et al., 2016).

Similarly, to a study by Sivakumar et al. (2022), the researchers investigated the impact of incorporating banana leaf (BL) fiber into TPCS composites with varying fiber

content ranging from 0% to 80%. In general, all the TPCS composite spectra showed the same band structure. The findings indicate that the incorporation of banana leaf fiber into the composites did not have any discernible effect on the TPCS matrix. Figure 2.24 depicts the data collected.

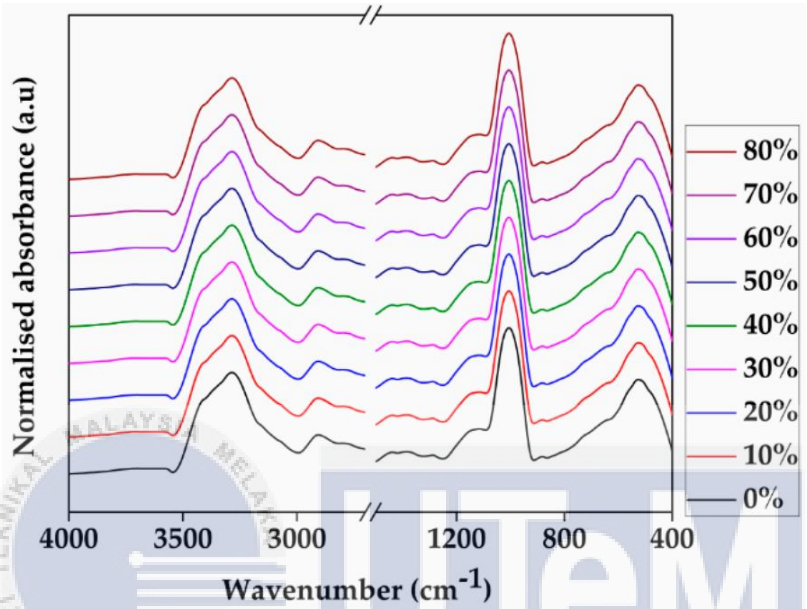


Figure 2.23 FT–IR spectra of TPCS with Banana Leaf Fibre (Sivakumar et al., 2022).

2.11 Summary

A thorough analysis of studies on thermoplastic starch, cassava starch composites, corn husk fibre, and composites made of corn husk fibre reveals several important conclusions:

i. The utilization of natural fibers in composites provides environmental benefits by repurposing waste from materials like corn husks, converting them into valuable products.

ii. Thermoplastic starch, as a biopolymer, possesses fascinating properties. Various modifications of thermoplastic starch, including the addition of fillers or fibers, have been found to enhance its characteristics.

iii. Corn husk fiber demonstrates promising capabilities as a reinforcement material in polymer composites, showing the ability to enhance the mechanical properties of the polymer matrix when incorporated into the composite structure.

Therefore, despite numerous studies conducted on thermoplastic starch, it has been observed that no research has been carried out to investigate the integration of corn husk into thermoplastic cassava starch/palm wax composites.

2.12 Literature Review Critique

This thesis explores the cost-effective and environmentally friendly integration of CHF into composite materials, particularly when combined with thermoplastic starch. Previous studies consistently show improved mechanical and thermal properties at an optimal fiber content. The effectiveness of CHF in composite production is underscored by these enhancements. In conclusion, this study advances our understanding of comprehensive analysis, specific data inclusion, and transparent methodology, bringing us closer to achieving our objectives in future applications.

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter will provide a comprehensive overview of the raw materials used, the fabrication process employed for thermoplastic cassava starch (TPCS) and corn husk fiber (CHF), as well as the planned testing procedures to assess the material's properties. Figure 3.1 presents a visual representation of the research approach adopted in this study, depicted in the form of a flowchart.

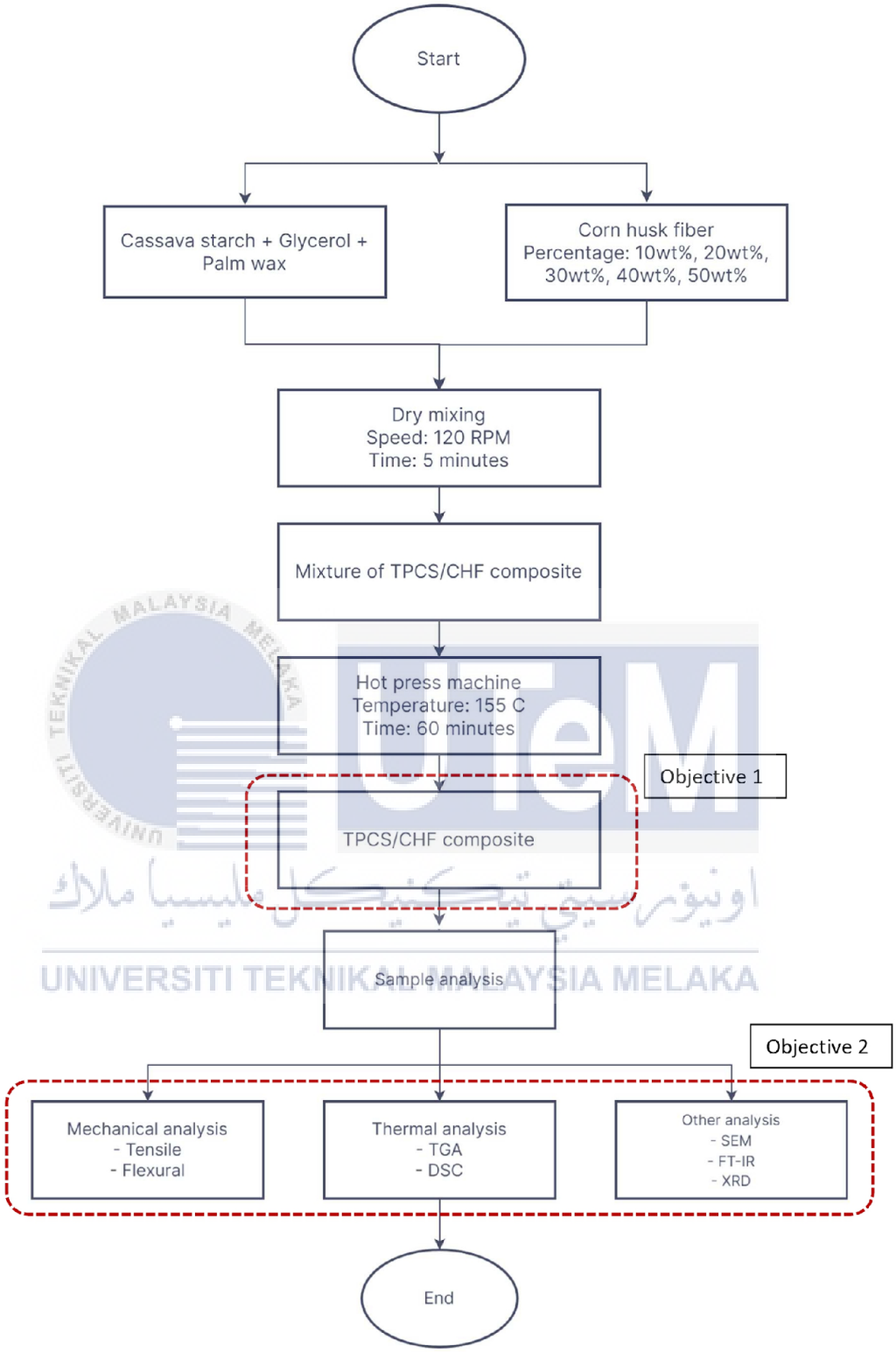


Figure 3.1 Flow of research methodology.

3.2 Materials

3.2.1 Corn husk fibre

Corn husks will be collected from one of the vendors at Wednesday Pasar Malam, Durian Tunggal, Melaka. There are few steps require in order to obtain the corn husk fiber. Firstly, the husks of the corn will be carefully removed from the corn. Next, the husks will be separated and placed in a container or basin filled with water. It will be ensured that the husks are fully submerged, and they will be left to soak for two weeks. After two weeks, the husks will be taken out of the water, and any excess moisture will be gently squeezed out. The rehydrated husks will be carefully torn or cut into thin strips or strands using sharp scissors or a knife. The tearing or cutting process will be conducted slowly and evenly to achieve a consistent thickness of the resulting fiber. The collected fibers will be sun-dried for a few hours and then dried at 80°C for another 5 hours to remove all moisture. Finally, they will be ground into short fibers.

3.2.2 Cassava starch

For this study, the main source of primary data was cassava starch obtained from Antik Sempurna Sdn. Bhd. in Malaysia. Cassava starch, similar to other starches, is a white powder.

Figure 3.2 Cassava starch.

3.2.3 Glycerol

A plasticizer, glycerol (C3H8O3), obtained from QReC (Asia) Sdn Bhd, was used in this study to enhance the flexibility and properties of the product. The glycerol from QReC Chemical was 99.5% AR grade and had a molar weight of 92.10 g/mol. Glycerol is a clear, viscous liquid with moderate viscosity. Table 3.1 displays the chemical composition of glycerol from QReC Chemical.

Figure 3.3 QReC Glycerol 99.5% Grade aAR.

Table 3.1 Chemical Composition of Glycerol from QReC Chemical.

Chemical Composition	Chemical Formula	Percentage (%)
Assay (Acidimetric)		Min 99.5
Insoluble in water		Passes test
Acidity / Alkalinity		Passes test
Halogen Compounds	Cl	Max 0.003
Chloride	Cl-	Max 0.001
Sulphates	SO4	Max 0.001
Ammonium	NH4	Max 0.0015
Arsenic	As	Max 0.0001
Copper	Cu	Max 0.001
Heavy Metal	Pb	Max 0.0005
Iron	Fe	Max 0.0005
Lead	Pb	Max 0.001
Nickel	Ni	Max 0.0005
Zinc	Zn	Max 0.001
Aldehydes	HCHO	Max 0.0005
1,2,4-butanorial	G.C	Max 0.2
Sulphated Ash		Max 0.01

3.2.4 Palm wax

Palm wax serves as a component that contributes to the overall properties of the composite material. Palm wax composition is palmitic acid, C16:0 = 59.09% and oleic acid, C18:1 = 39.55%.

Figure 3.4 Palm wax.

3.3 Preparation of samples

3.3.1 Preparation of Thermoplastic Cassava Starch with Palm Wax

The production process of the thermoplastic cassava starch with palm wax involved several sequential steps to achieve a homogeneous mixture with desirable properties. A weight of 100 grams of cassava starch was carefully measured using a digital scale, followed by 30 grams of glycerol supplied by QReC (Asia) Sdn Bhd, to maintain a fixed ratio of 100:30 between the starch and glycerol.

Thorough mixing of the cassava starch and glycerol was carried out in a suitable container to achieve a uniform blend. The mixture was stirred carefully with a spatula and by hand to ensure proper integration. Subsequently, the resulting blend was weighed again to determine its mass. The expected weight of the mixture, after incorporating the glycerol, was approximately 58.2 grams, suitable for fitting the mold size. To further enhance the properties of the blend, 10% of palm wax (5.82 grams) was added as a plasticizer.

To ensure complete homogeneity, the mixture underwent a thorough blending process using a dry blender set at 1200 rpm. The blending continued until all the ingredients were fully integrated. The blended mixture was then poured into a mold placed on a Mylar sheet to prevent sticking. The mold with the mixture was subjected to hot pressing at a temperature of 155°C for one hour. Afterward, the mold was allowed to cool for a period of 20 minutes. This cooling process facilitated the solidification and stabilization of the blend, preserving its integrity and shape. Finally, the solidified mixture was carefully removed from the mold using a spatula, completing the production process of the cassava starch-glycerol blend.

Figure 3.5 Fabrication of thermoplastic cassava starch with palm wax.

Figure 3.6 Thermoplastic Cassava Starch with Palm Wax Sample.

3.3.2 Preparation of Thermoplastic Cassava Starch Reinforced with Corn Husk Fibre

The production of the thermoplastic cassava starch composite with corn husk fiber involved the combination of thermoplastic cassava starch and corn husk fiber. The composition of the mixture is determined by the specified fiber percentages, including 0%, 10%, 20%, 30%, and 40%. The proportions of the mixture will be calculated based on the corresponding fiber percentage. For instance, when incorporating 40% corn husk fiber, it will necessitate 60% thermoplastic cassava starch, equivalent to 23.28g of corn husk fiber and 34.92g of thermoplastic cassava starch. Subsequently, the thoroughly mixed and blended mixture is placed into a mold and subjected to pressing in a hot press machine at a temperature of 155°C for a duration of 60 minutes. This procedure replicated for each of the remaining fiber percentages.

Table 3.2 Mixture weight of preparation of Thermoplastic Cassava Starch Reinforced with Corn Husk Fibre

CHF (%)	Cassava Starch (g)	Glycerol (g)	Fiber (g)	Palm Wax (g)
0	36.66	15.72	0	5.82
10	32.59	13.96	5.82	5.82
20	28.52	12.22	11.64	5.82
30	24.44	10.48	17.46	5.82
40	20.37	8.73	23.28	5.82

Figure 3.7 Fabrication of thermoplastic cassava starch reinforced with corn husk fiber.

3.4 Characterisation of samples

3.4.1 Mechanical testing

The mechanical testing of the composite material samples was conducted in this project. The objective of the study was to assess the durability of the samples through mechanical testing. Tensile and flexural tests were involved to analyze the material characteristics and properties.

3.4.1.1 Tensile testing

The tensile strength, tensile strain, and tensile modulus were determined through tensile testing. This test measured the force required to fracture the specimen and its length. Following the guidelines outlined in the ASTM D638 standard, a total of five (5) samples were chosen for analysis. These samples underwent thorough examination using an INSTRON 5969 Universal Testing Machine (UTM) featuring a crosshead speed of 5 mm/min and a 50 kN load cell. The test was conducted at a temperature of 24.0 °C and a relative humidity of 40% - 50%. The results of the tensile properties were determined by calculating the mean of the collected data.

Figure 3.8 Universal Testing Machine (UTM).

3.4.1.2 Flexural testing

Flexural tests, following the ASTM D790 standard, were conducted at a relative humidity of 50 ± 5% and a temperature of 23 ± 1 °C. A set of five (5) samples, measuring 130 mm in length, 13 mm in width, and 3 mm in thickness, were fabricated for the experiment. The mechanical testing was conducted using an INSTRON 5969 Universal Testing Machine (UTM) with a crosshead speed of 2 mm/min and a 50 kN load cell.

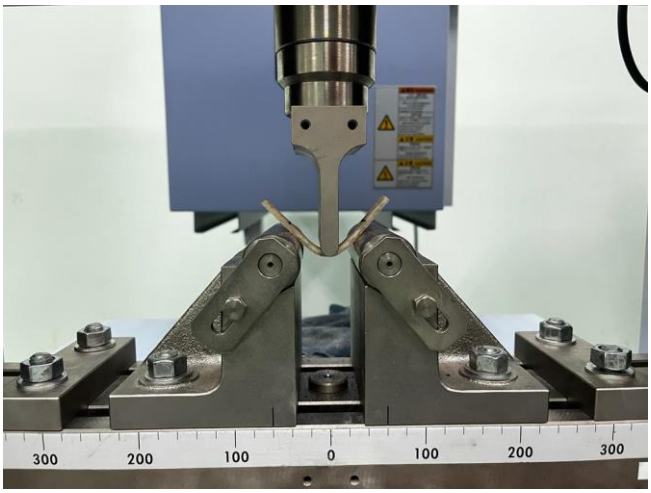


Figure 3.9 Flexural testing on Universal Testing Machine (UTM)

3.4.2 Thermal testing

3.4.2.1 Thermogravimetric analysis (TGA)

The thermal decomposition of the composites was analyzed using thermogravimetric analysis (TGA), as indicated by the weight loss observed with increasing temperature. The TGA study was conducted in aluminum pans, with a heating rate of 10°C per minute, covering a temperature range from 25 to 900°C, under a nitrogen environment with dynamic pressure. The obtained TGA data were utilized to derive the values of thermogravimetric analysis (DTG), highlighting the variations in thermal behavior.

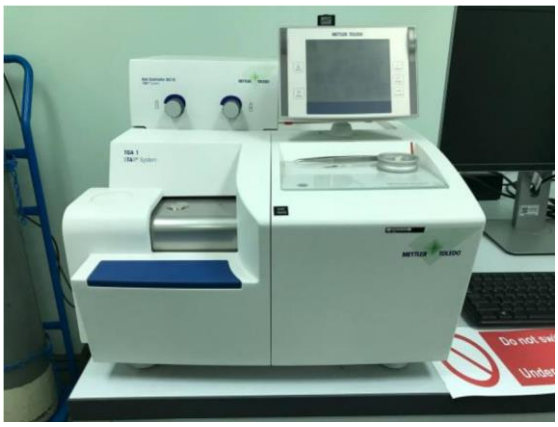


Figure 3.10 Thermogravimetric analysis (TGA) machine.

3.4.3 Other testing

3.4.3.1 Scanning electron microscope (SEM)

The morphological characteristics of fractured tensile samples were investigated using a scanning electron microscope (SEM), specifically the Zeiss Evo 18 Research model from Jena, Germany, with an acceleration voltage of 10 kV. The samples were prepared by resizing them to comparable dimensions, and their surfaces were coated with a layer of gold prior to examination. After conducting the tensile tests, the specimens were carefully stored in zip-locked containers for subsequent characterization using SEM.

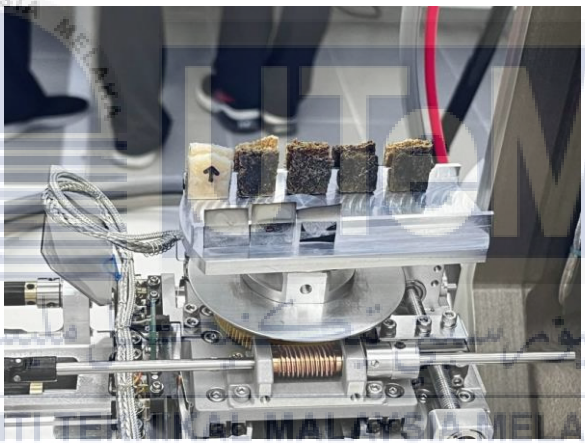


Figure 3.11 Scanning Electron Microscope (SEM)

3.4.3.2 Fourier transform infrared spectroscopy (FT-IR)

Fourier transform infrared spectroscopy (FTIR) was used to examine thermoplastic starch, palm wax, and corn husk to identify their functional groups. The JASCO FTIR-6100 Spectrometer (Japan) was employed to obtain the spectra of each material. The FT-IR spectra of the samples were examined in the range between 4000 cm-1 and 5000 cm-1. For the measurements, the samples were turned into a powder and weighed at 2 mg. Subsequently,

the samples were mixed with potassium bromide (KBr) and compressed into 1 mm thick discs for spectroscopic measurements.

Figure 3.12 Fourier transform infrared spectroscopy (FT-IR) machine.

3.5 Fabrication of the packaging

The production of the biodegradable packaging tray, made from a composite of corn husk fibers, involved utilizing hot-pressing equipment. The tray was manufactured at a temperature of 155°C for 60 minutes, followed by a cooling period of 20 minutes. The manufacturing process was similar to that of material testing samples. To initiate the process, cassava starch, palm wax, glycerol, and corn husk fiber were physically combined. The mixtures were then blended using a blender until a homogeneous consistency was achieved. For this particular production, fibers with a 30% ratio were used throughout the manufacturing procedure.

CHAPTER 4

RESULT & DISCUSSION

4.1 Introduction

This section presents the findings derived from sample production and the results of various tests conducted in this study. The focus is on mechanical testing, including the Tensile Test and the Flexural Test, as well as thermal testing, including Thermogravimetric Analysis (TGA). Additional tests involve Scanning Electron Microscopy (SEM), and Fourier-Transform Infrared Spectroscopy (FT-IR). The objective of mechanical testing is to assess the material's characteristics and structure, while thermal testing evaluates its performance at different temperatures. Other tests aim to analyse the surface topography and composition.

4.2 Mechanical testing

4.2.1 Tensile testing

Tensile testing can help evaluate the effectiveness of natural fibers as reinforcement in composites by providing information on the tensile strength, tensile modulus, and tensile strain to achieve the composite formulation desired. The test measured the force required to fracture the specimen and its length. In accordance with the ASTM D638 standard guidelines, a total of five (5) samples were chosen for analysis and were machine-cut to dimensions of 10mm (L) by 13mm (W) by 3mm (T). These samples underwent a comprehensive examination using an INSTRON 5969 Universal Testing Machine (UTM)

with a crosshead speed of 5 mm/min and a 50 kN load cell. The test was conducted at a temperature of 24.0 °C and a relative humidity of 40% - 50%. The tensile strength and modulus of the composites increased until the maximum amount of fiber percentage at 40%. This enhancement underscores the effectiveness of the reinforcement materials.

Figure 4.1 shows the tensile strength of TPCS/CHF composites for fiber percentages of 0%, 10%, 20%, 30% and 40% respectively. The tensile strength for fiber percentage of 0% is 0.44MPa increasing until 40% at 2.41MPa. The results indicate a significant increase in tensile strength with the addition of CHF percentage to 40%. The increase in tensile strength with the addition of CHF percentage aligns with the previous study observed in natural fiber reinforced polymer composites, where the tensile strengths increase with fiber content, up to a maximum or optimum value (Ku et al., 2011). This is consistent with the idea that at optimum percentage of fiber, the tensile strength of a composite increases with the increase in fiber volume content (Huang et al., 2021).

The increasing tensile strength of CHF and TPCS can be due to the reinforcement effect where the addition of CHF to a polymer matrix provides reinforcement, leading to an increase in tensile strength. This reinforcement effect is attributed to the load-bearing capability of the CHF because reinforcement can provide strength and stiffness to the composite in one direction as reinforcement carries the load along the length of the CHF which contribute to the overall strength of the composite material (Rios-Soberanis, 2020; Huang et al., 2021).

In this study, the maximum or optimum load of CHF content is unable to be obtained due to a substantial crack occurring in the last sample at 40%, further CHF addition is not feasible for the next sample, making it unattainable to achieve the 50% sample. This explains

that the mechanical properties of the composite may have been compromised, leading to a loss of structural integrity (Bijelic-Donova et al., 2022). This could be due to factors such as inadequate bonding between the CHF and the matrix, uneven distribution of the fibers, or limitations in the load-bearing capability of the CHF at higher concentrations (Nasmi et al., 2020).

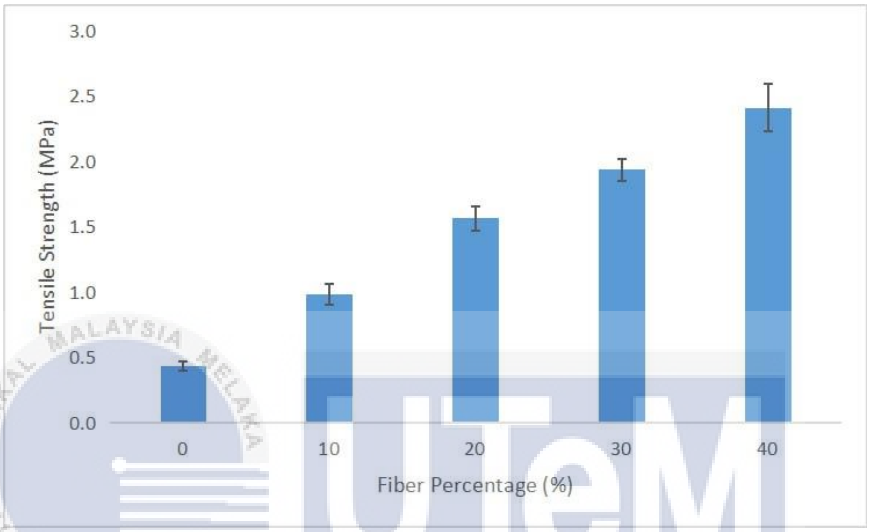


Figure 4.1 Tensile strength result of TPCS/CHF

Figure 4.2 shows the tensile modulus of CHF/TPCS with a consistent increasing pattern as the CHF content increases. The initial slope and linear region of the stress-strain curve were used to derive the tensile modulus. According to Jones et al., (2019), the tensile modulus, also known as Young's modulus, is a measure of a material's stiffness or resistance to deformation under tensile (pulling) forces. The tensile modulus exhibited a consistent pattern, showing an increase from 0% to 40% CHF loading. A higher tensile modulus indicates a stiffer material, which is consistent with the general understanding of material behavior. The inclusion of CHF was shown to increase the tensile modulus of 0% from 15.01MPa to 280.87MPa at 40% CHF content. Result indicates that the higher fiber content, the greater tensile modulus obtained. This behavior may be the result of fiber mass fraction

which indicates a positive correlation between fiber content and tensile modulus (Aguado et al., 2023). According to Ku et al. (2011), this reaction happened because the presence of fibers in the composite enhances its overall structural integrity by acting as reinforcement. The fibers distribute and bear the applied load, thereby strengthening the material and increasing its resistance to deformation and failure.

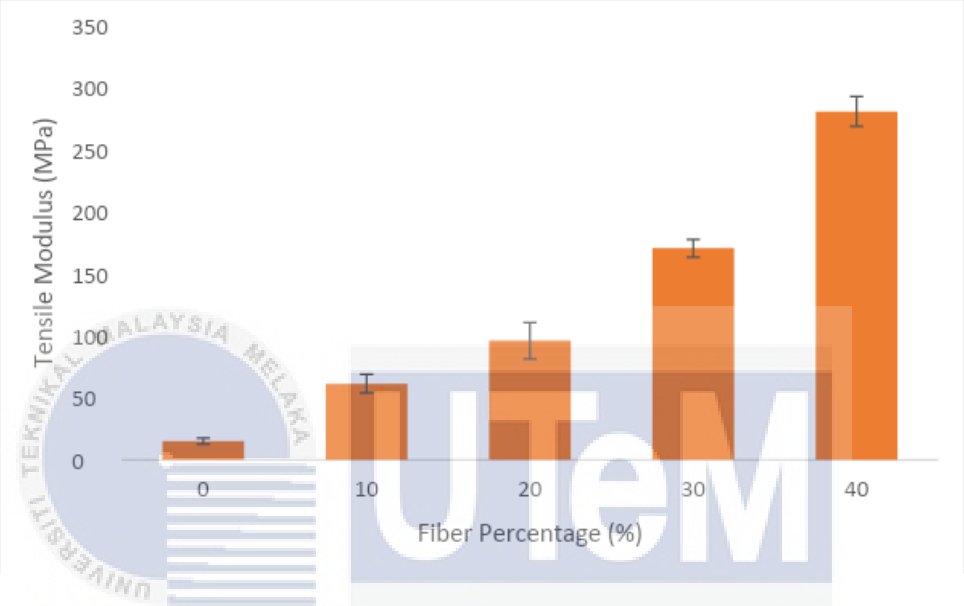


Figure 4.2 Tensile modulus result of TPCS/CHF

The elongation break results achieved from CHF/TPCS are shown in Figure 4.3. This reveals an increasing trend at 0% to 10% CHF percentage and decreasing from 10% to 40% at 5.56% to 0.59% respectively. CHF/TPCS become stiffer and less ductile as the percentage of natural fiber increases. This is due to the fact that CHF are stiffer and less ductile, which can lead to a decrease in the overall ductility of the composite material (Ku et al., 2011). SEM images as shown in Figure… of composites with 40% content of CHF show more brittle fracture surfaces with less fiber pull-out, indicating decrease in tensile elongation. Conversely, CHF/TPCS with lower natural fiber content may show more ductile fracture surface with more fiber pull-out, indicating higher tensile elongation (Pickering et al., 2015).

This observation is consistent with the findings of Sani et al. (2020), where the elongation at break of CHF composites increased at 25-30% fiber content, and subsequently decreased with a further increase in fiber content. These collective findings underscore the inverse relationship between the percentage of natural fiber and the elongation at break in composite materials, highlighting the influence of fiber content on the mechanical properties of the composites.

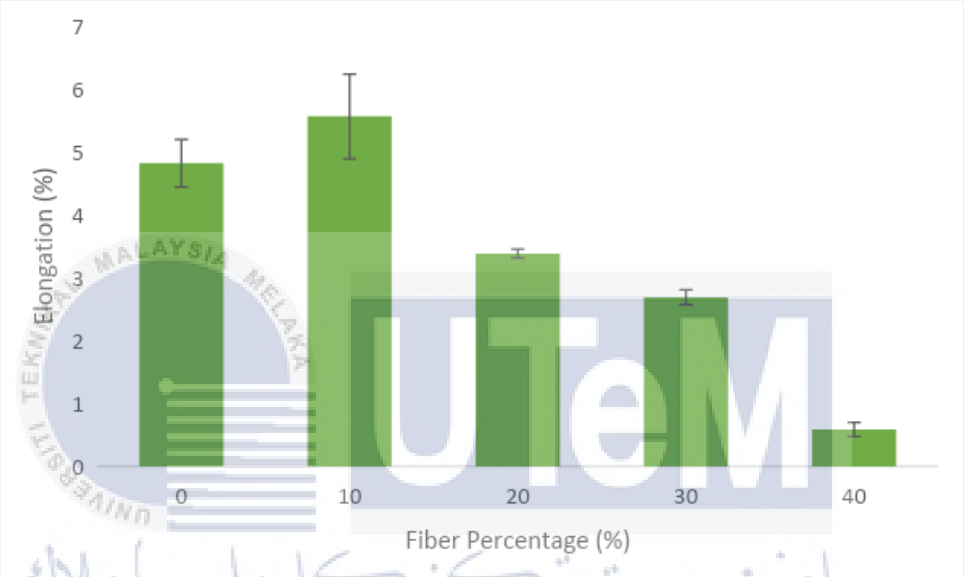


Figure 4.3 Tensile elongation result of TPCS/CHF

Table 4.1 Summary of the analysis of variance (ANOVA) of flexural properties

Variables	df	Tensile strength	Tensile Strain	Tensile Modulus
Mixtures	4	0.00*	0.00*	0.00*

4.2.2 Flexural testing

The flexural test determines a material's ability to withstand bending and stiffness by measuring the force required to bend a plastic beam. The test is widely used to evaluate the

bending behavior of materials and their mechanical performance. By utilizing the third point load test, this experiment was conducted. Compared to traditional synthetic fiber composites, natural fiber composites often exhibit different flexural properties due to the unique characteristics of the natural fibers (Elfaleh et al., 2023). Flexural testing plays a crucial role in understanding how natural fibers affect the bending and stiffness characteristics of composite materials, providing essential data for the development and optimization of these sustainable and eco-friendly composites (Santos et al., 2019).

The flexural strength of CHF/TPCS are presented in Figure 4.4. This shows that the values of flexural strength increase as the CHF content increases where the highest flexural strength obtained is 10.39MPa at 40% fiber percentage. This indicates that the best amount of CHF required for reinforcing is 40%wt. Similar factors that were mentioned in the tensile test could also be attributed to the improvement of the flexural strength properties of the composite. The increase is mostly due to stronger interfacial bonding between the fiber and TPCS. Enhanced bonding can lead to better stress transfer between the fiber and the matrix (Pickering et al., 2016). According to Hafila et al., (2022), the higher the flexural strength of CHF/TPCS indicates the degree of cross-linking between the molecular chain. However, a slight decrease of flexural strength occurred at 30%wt. which might be attributed to the difficulty in achieving uniform fiber dispersion. This can create stress concentration points and weaken the composite (Jumaidin et al., 2021). Another factor that might contribute to the decreasing flexural strength at 30%wt is errors in preparing samples that can affect the fiber orientation, fiber dispersion and fiber-matrix interaction of CHF/TPCS (Morrell, 2007).

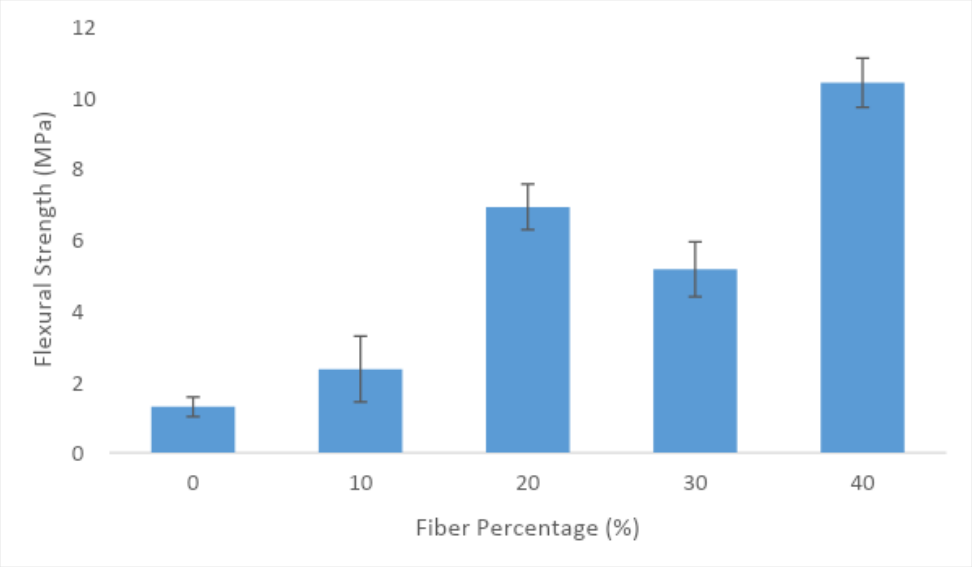


Figure 4.4 Flexural strength result of TPCS/CHF

Figure 4.5 displays the flexural modulus of CHF/TPCS. Generally, the outcome of this flexural modulus exhibited similar trends with tensile modulus. The trend shows that the flexural modulus increased from 0 to 40 wt.% of fiber content. The increase in flexural modulus might be due to the improvement of the fiber and matrix adhesion (Bhatnagar et al., 2015). A higher stress was required for the same deformation when a higher amount of fiber was added, thus the incorporation of the fiber into the matrix resulted in an increase in the flexural modulus (Elanchezhian et al., 2018). A similar trend was found in a study by Shinde et al., (2014) where the properties of coir fiber composite had increased up to 60 wt.% fiber content. However, the flexural modulus started to decrease as higher fiber content was added. In this study of CHF/TPCS higher load of fiber was not able to be obtained due to substantial crack occurring in the last sample at 40 wt.% fiber content. This might be attributable to the insufficient matrix to cover all the surfaces of the fiber.

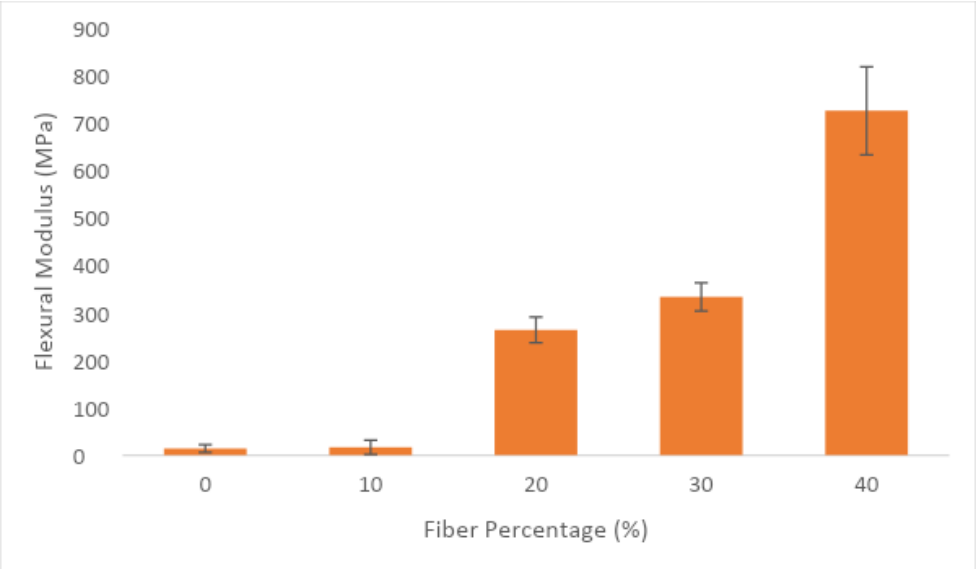


Figure 4.5 Flexural modulus result of TPCS/CHF

Table 4.2 Summary of the analysis of variance (ANOVA) of flexural properties

Variables	df	Flexural Strength	Flexural Modulus
Mixtures	4	0.00*	0.00*

4.3 Thermal testing

4.3.1 Thermogravimetric analysis (TGA)

Thermogravimetric Analysis (TGA) involves subjecting a sample to a controlled temperature programme and measuring the sample's weight change as a function of temperature or time. TGA is notably useful for determining a material's thermal degradation properties, including its onset temperature, rate of degradation, and the amount of weight loss. In literature, Yalzir et al., (2014) noted that the cellulose in corn husk fibers was significantly more thermally stable, with a maximum degradation temperature of 338.9 °C. Khalaf et al., (2021) observed that thermal conductivity increased with decreasing porosity

and rising density, identifying porosity as the most influential factor on this thermal parameter.

The thermal deterioration and stability of a blend comprising palm wax, cassava starch, and CHF were evaluated by depicting TGA curves, representing the percentage (%) of sample weight loss against temperature (°C), as depicted in Figure 4.6. Figure 4.6 illustrates that the composite exhibiting the minimal content of corn husk fibers underwent the most substantial weight reduction. Shahzad, (2013) posits that the thermal disintegration of natural fibers, composed of hemicellulose, lignin, pectin, and glycosidic cellulose connections, is the underlying cause of thermal degradation at the specified temperature. Mass diminution in natural fibers results from the breakdown of the three principal components: hemicellulose, cellulose, and lignin (Sapuan et al., 2017).

Observations indicate a decrease in sample weight with escalating temperature. The initial weight loss, influenced by the primary thermal event occurring below 100°C, suggests the elimination of water molecules through evaporation (Hafila et al., 2022a; Rajak et al., 2019). Ibrahim et al. (2020) attribute weight loss in a prior study to fructose fragment evaporation and water particle evaporation, aligning with the findings of Zhang et al. (2018) on the characteristics of gelatine films following beeswax addition.

The biopolymer's secondary thermal deterioration, within the temperature range of 200°C to 320°C, is associated with volatile matter, bound water loss, and plasticizer disintegration (Oluwasina et al., 2019). The most significant mass loss is a consequence of depolymerization and breakdown of the carbon chain within the starch matrix, the primary component of the composite (Ibrahim et al., 2020). Nurazzi et al. (2021) contend that the most substantial weight loss occurs between 278°C to 306°C due to the thermal degradation of hemicellulose, lignin, pectin, and glycosidic linkages of cellulose in natural fibers. The

subsequent stage of weight loss may be linked to the breakdown of glycerol, wax, and starch, attributed to hydrogen bonds formed by electrostatic and Van de Waals forces, as well as steric repulsions (Zhang et al., 2018). As previously articulated by Razali et al. (2015), the thermal degradation of roselles fiber results from the thermochemical shift in the fiber's hemicellulose content caused by cellular breakdown as the temperature rises.

The conclusive phase of thermal degradation, arising from the disintegration of amylose and amylopectin glycosidic connections within the wax and starch polymer, was observed to manifest between 340°C to 380°C, as reported by Zhang et al. (2018).

In an investigation conducted by Sapuan et al. (2017), the third stage of a preceding study on environmentally sustainable composites crafted from sugar palm trees focused on the decomposition of lignin. Lignin, recognized for its elevated resistance and its role in conferring rigidity to plant materials, proves to be the most resistant among hemicellulose and cellulose during the decomposition process.

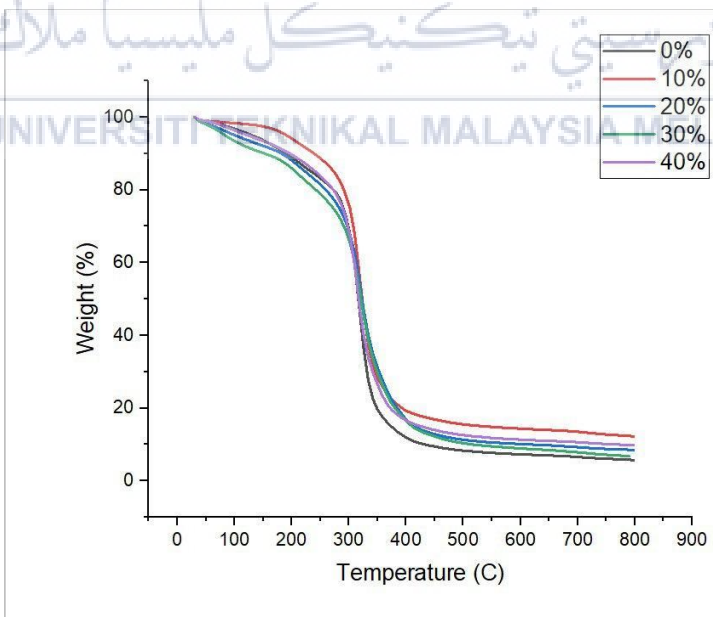


Figure 4.6 TGA curves of TPCS/CHF composite as weight loss (%) versus temperature (°C)

Figure 4.7 illustrates DTG curves delineating degradation characteristics. The most significant loss in derivative weight was observed in instances with 0% fiber loadings. As posited by Diyana et al. (2021), the incorporation of fiber into the starch matrix enhances heat stability due to the matrix's robust adherence to the fiber, resulting in reduced weight loss in the samples. In a prior examination of the impact of fiber surface treatment on their properties, Zhou (2022) asserted that the degradation rate signifies the breakdown of fiber cellulose and hemicellulose or the loosening of fibers within the composite.

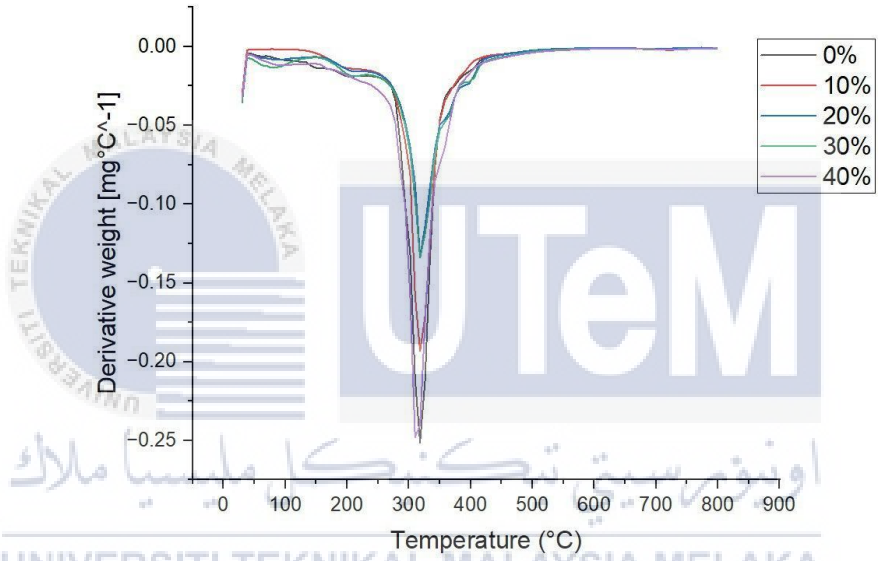


Figure 4.7 DTG curves of TPCS/CHF composite as Derivative weight loss (%) versus temperature (°C).

4.4 Other testing

4.4.1 Scanning electron microscope (SEM)

Scanning Electron Microscope (SEM) is a high-resolution electron microscopy technique that provides more information about nanomaterials by probing samples on a very

fine scale with a high electron beam (Parisa, 2018). This study aims to investigate the morphology and failure mechanism of TPCS and corn husk fibre.

Figure 4.9 presents the SEM micrograph of the tensile-tested fracture surface for pure TPCS. From the result, it can be seen that the sample has no fiber. A remarkable finding was that a part of the mixture between cassava starch and palm wax had formed a granular structure like a glossy shape. This finding is in concurrence with the research by Halifa et al. (2022), where a similar structure formation of palm wax/TPCS was observed. Furthermore, the existence of voids found on the 0%wt. TPCS/CHF composites indicated the interaction between starch/palm wax that was relatively low ascribed to the addition of high filler amount and the inability of proper hydrophilic starch mixing with hydrophobic wax matrix (Filho et al., 2020). This was due to discontinuity in the matrix and it started to agglomerate at higher filler content (Filho et al., 2020).

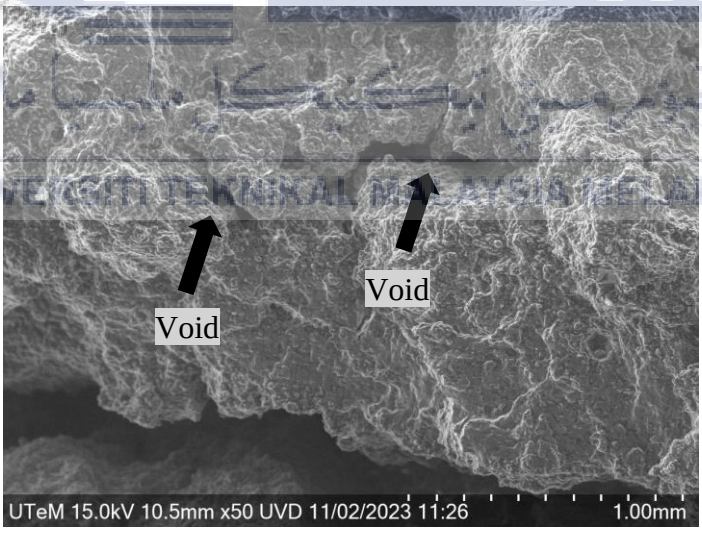


Figure 4.8 SEM images of fracture surfaces of pure TPCS

Figure 4.9 (a, b and c) shows the SEM fractographs of the tensile-tested fracture surface for 10% CHF contained in the composite specimen. The presence of cracks can be

seen in Figure 9 (c) indicating insufficient mixing between the fiber and matrix. The result did not show evidence of an excellent adhesive structure. It showed most of the fibers and the matrix were separated, indicating a weak interfacial bonding between the corn husk fiber and the matrix.

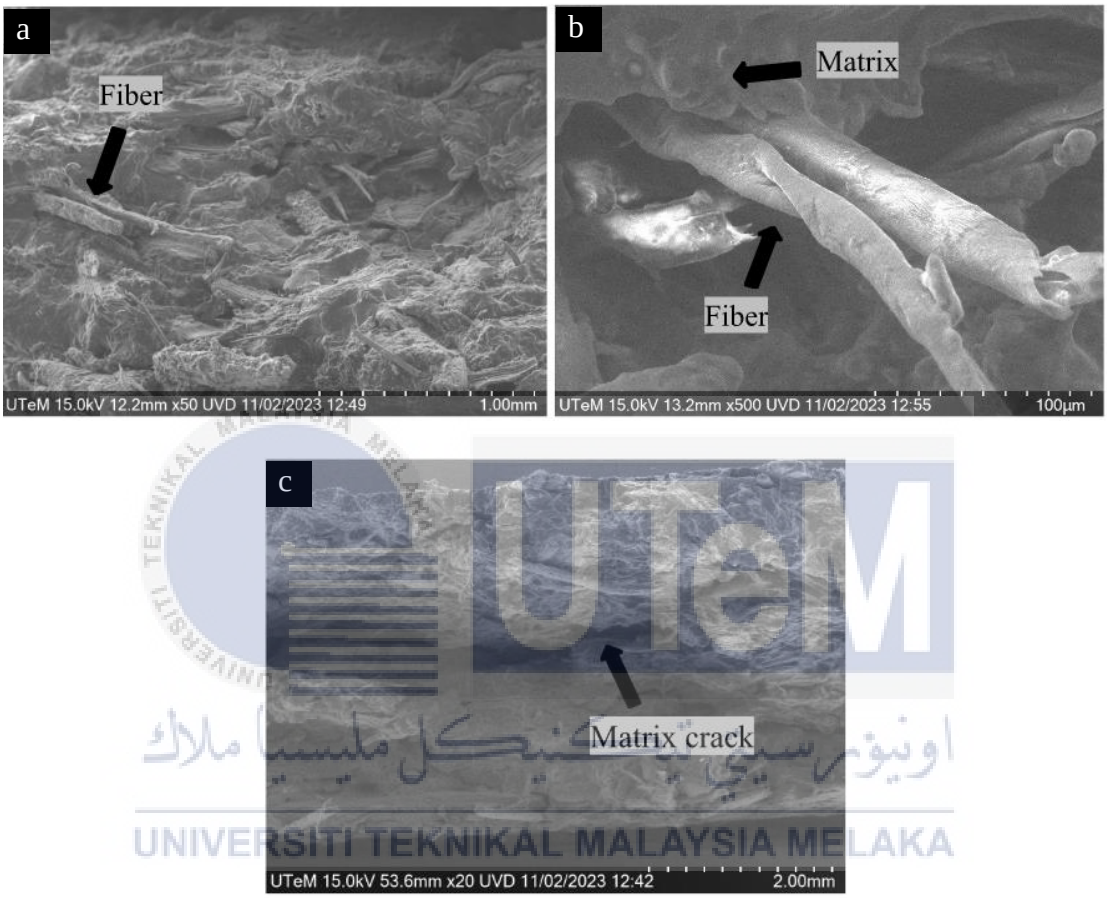


Figure 4.9 SEM images of fracture surfaces of 10 %wt. TPCS/CHF with different magnification (a) x50 (b) x500 (c) x20

Figure 4.10 (a, b and c) presents the fiber breakage in all composites from 20% to 40% content fiber resulting from the tensile fracture, owing to effective stress transfer from the TPCS matrix to CHF, which contributed to the reinforcing effect of the composites. This discovery was in line with the results of tensile tests, which demonstrated increased tensile modulus and strength, as presented in the tensile strength result. Other researchers have reported similar observations in this area on the development of thermoplastic starch where

the tensile strength increases as the stress transfer gets more effective. The figure also illustrates the presence of micro voids in the samples. The emergence of voids upon the addition of CHF signifies a weakened interfacial bonding between the CHF and matrix. This can be attributed to the high amount of filler and the challenge of hydrophilic starch forming a proper bond with hydrophobic wax (Halifa et al., 2022).

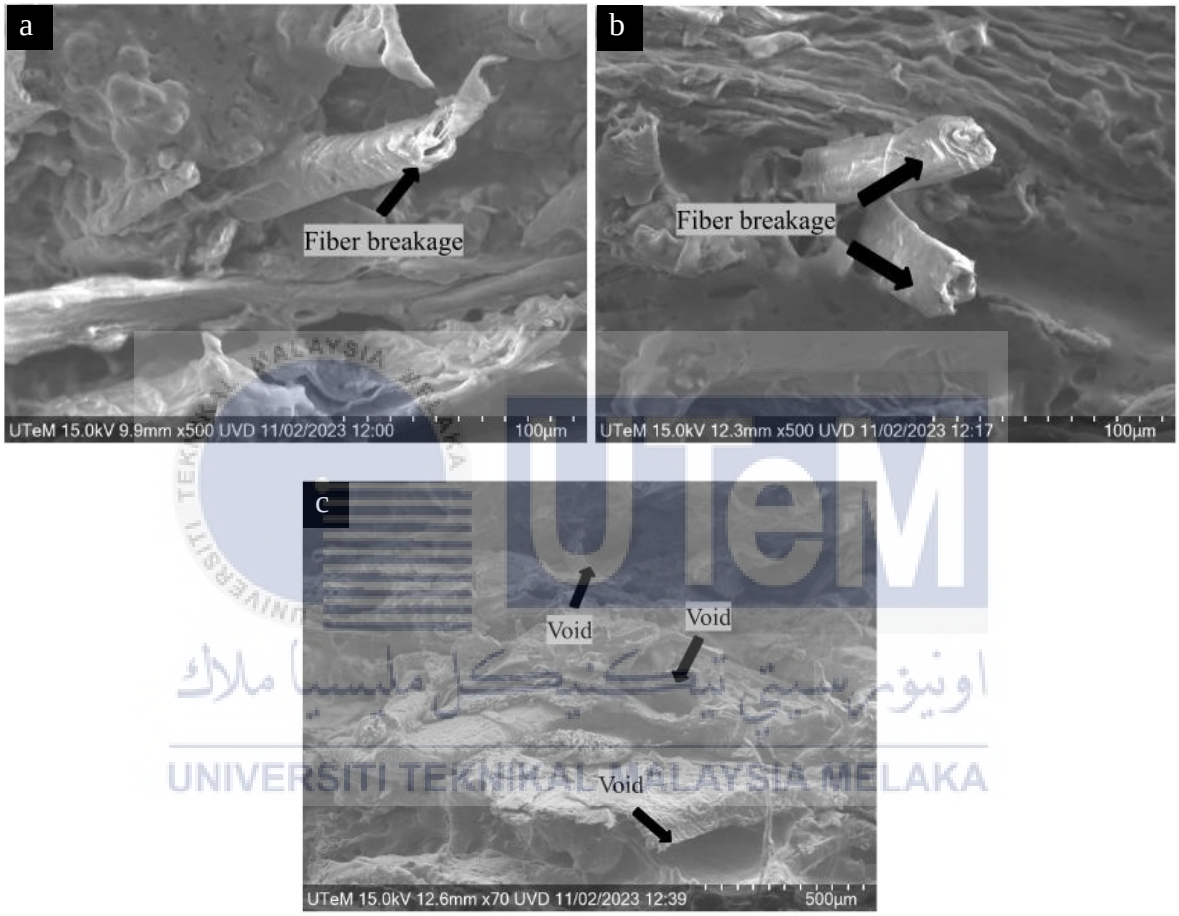


Figure 4.10 SEM images of fracture surfaces TPCS/CHF with different fiber content (a) 20%wt. (b) 30%wt. (c) 40%wt.

4.4.2 Fourier transform infrared spectroscopy (FTIR)

The FTIR test aimed to assess the presence of functional groups in corn husk fibers and to examine the functional group and chemical properties within the composite material

of fiber and starch. The Nicolet 6700 AEM IR spectrometer was utilized to obtain the material's spectrum.

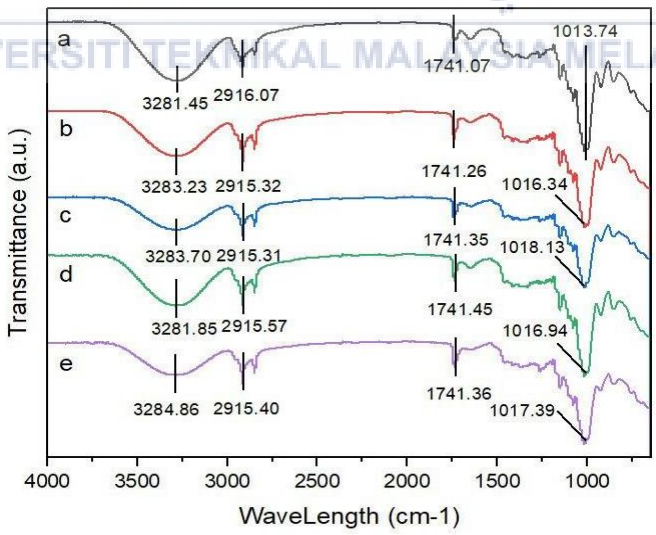
In Figure 4.9, the FTIR spectra of TPCS reinforced with CHF composites (ranging from 0% to 40%) exhibit similar band patterns for fiber contents of 0%, 10%, 20%, 30%, and 40%. These patterns suggest that the combination of cassava starch, glycerol, palm wax, and corn husk fiber have no discernible effect on chemical composition. This outcome is likely due to the cellulose structure predominant in both starch and fibers (AL-Hassan, 2017). Similar observations were reported by Bangar et al., (2021) in prior studies on related composite materials, affirming that FT-IR spectra display consistent peaks for samples derived from biological resources.

In Figure 4.9, the presence of hydroxyl groups (O-H) in TPCS/CHF from 0% to 40% is evident around 3200 cm^{-1}, originating primarily from cassava starch and corn husk fiber. The existence of O-H groups in starch and fiber is supported by prominent peaks at 3200 cm^{-1} to 3500 cm^{-1}, as noted by Hafila et al. (2022a) and Sapuan et al. (2017). This suggests that starches are highly responsive to water molecules due to their substantial hydroxyl group content.

Examining Figure 4.9, the wavenumber of O-H bands at 10% fiber content slightly increases from 3281.45 cm^{-1} to 3283.23 cm^{-1}. Although the O-H bands exhibit minor changes with increasing fiber content, Mehyar et al. (2012) observed similar variations in previous research on thermoplastic pea starch composites. This alteration in the spectrum and frequency of the OH band is attributed to the plasticizing process, indicating that glycerol influences the network of intermolecular hydrogen bonds between starch molecules. Consistent findings were also reported by Jumaidin (2017), affirming that spectra band peaks

are influenced by the interaction of intermolecular hydrogen bonding in related investigations.

The band at approximately 2916 cm^{-1} is ascribed to C–H stretching from CH_2 or CH_3 conjugated bending vibrations, a feature present in all composite samples. Consistent with Barkoula et al. (2008), this band aligns with the cellulose and hemicellulose constituents of natural fibers. The sensitivity of starch to water molecules, as noted by Ilyas et al. (2018b) and Sahari et al. (2013), is attributed to the presence of hydroxyl groups, with their stretching influenced by hydrogen bonding between molecules. According to Dang et al. (2021), the peak around 2916 cm^{-1} corresponds to C–H stretching (-CH_2) of the anhydro-glucose ring (Zullo et al., 2009), while the peak at around 3277 cm^{-1} corresponds to the firmly bound water within the starch structure (Y. Zhang et al., 2006) due to its hygroscopic nature. According to Tkachenko et al., (2022), the band at around 1741 cm^{-1} may correspond to stretching vibrations of carbonyl groups (C=O). However, when the fiber content increased, there were no significant changes to indicate the increasing carbonyl group in the composite.



CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Many studies aim to create eco-friendly and durable materials, emphasizing the need for new biodegradable products to protect the environment. This chapter details the development of a material from cassava starch, focusing on its main purpose and results. The study primarily concentrates on improving the properties of the reinforced TPCS/CHF composite through various tests, including mechanical and thermal evaluations. The research has three main goals, and the following section outlines these objectives along with the findings.

Objective 1: To prepare a composite material out of biodegradable thermoplastic cassava starch reinforced with corn husks fiber.

The biodegradable thermoplastic composite made of cassava starch and corn husk fibre has been successfully prepared by using dry mixing and hot-pressed method. The TPCS reinforced corn husk fibre composite was prepared at different fibre content of 0%wt., 10%wt., 20%wt., 30%wt., and 40%wt.

Objective 2: To investigate the mechanical properties, thermal properties, and the influence of fibre on a biodegradable thermoplastic cassava starch composite that is reinforced with corn husk fibres.

In examining the mechanical aspects, the tensile and flexural properties of the TPCS composite reinforced with corn husk fiber were investigated. The composite attained

maximum strengths of 2.41 MPa for tensile and 10.39 MPa for flexural at a 40% fiber content. The tensile and flexural characteristics of the TPCS/CHF composite displayed a consistent trend. The introduction of fiber led to increased tensile and flexural strength, along with modulus. However, the variation in corn husk fiber sizes resulted in a significant decrease in tensile elongation, plummeting from 5.56% to 0.59%.

In terms of thermal stability, the characteristics of the TPCS/CHF composites were analyzed using thermogravimetric analysis (TGA). Generally, incorporating fiber into cassava starch improved the thermal stability of the composites in comparison to using cassava starch alone. Consequently, the thermal stability of the fiber increased, reaching its maximum stability with a larger fiber content of 40%. The TPCS/CHF composites, with 40% fiber content, achieved the most stable thermal condition, marked by the highest onset temperature of 318 °C.

Additionally, the characterization of TPCS reinforced with corn husk fiber was analyzed through SEM and FTIR testing. The SEM micrograph revealed an increase in micro voids and fiber breakage with higher corn husk fiber content. Moreover, FTIR demonstrated an enhancement in the bonding between the components in the composite.

Objective 3: To fabricate biodegradable packaging by using cassava starch and fibre from corn husks.

The creation of a biodegradable packing tray, composed of corn husk fibers and cassava starch, was successfully accomplished using hot-pressing equipment at 155°C for 60 minutes, followed by 20 minutes of cooling. The manufacturing process for this item resembled that of crafting material testing samples, and it exhibits significant potential as an alternative to synthetic plastic. A survey was conducted to collect data on the market

feasibility of this product, assessing its potential for widespread commercialization within the industry.

5.2 Project Potential

Project has been developed as a packaging product; a process akin to producing material testing samples was employed. The procedure initiated with the blending of cassava starch, palm wax, glycerol, and CHF until a smooth and well-blended mixture was achieved, containing 20 wt.% CHF. This blend was then poured into a mold lined with mylar film and subjected to a hot press machine set at 155°C for an hour, followed by a 20-minute cooling period. With improved manufacturing processes, this product could find application in everyday life as eco-friendly food packaging, potentially substituting synthetic plastics.

Figure 5.1 depicts a conceivable version of the final product—a thermoplastic tray made from a combination of TPCS reinforced with CHF. This versatile product is suitable for storing food, essentials, or stationary items and offers the added advantage of being cost-effective, utilizing minimal raw materials. Through a survey of end users, such as store owners, it was determined that this product holds potential for commercialization in the industry. A preliminary cost estimate in Table 5.1 demonstrates its competitiveness with other non-biodegradable bioplastics. Through a survey of end users, such as store owners, it was determined that this product holds potential for commercialization in the industry.

Table 5.1 Total cost of raw material for one tray

Material	Weight (g)	Price per gram (RM)	Price per tray
Cassava starch	40	0.003	0.12
Glycerol	18	0.0026	0.047
Palm wax	9	0.0026	0.023
Corn husk fiber	17	0	0
Total cost			**0.19**

Figure 5.1 Product made of TPCS/CHF

COMMERCIALIZATION OF SURVEY ON PACKAGING TRAY OF BIODEGRADABLE CORN HUSK FIBER COMPOSITE

NAME/NAMA: MOHD FAIZ ZAKARIA
COMPANY NAME/NAMA SYARIKAT:

1. If this product is marketed, are you willing to purchase this product?
   Kalau produk ini didapati di pasaran, adakah anda akan membelinya?
   ☑Yes/Ya ☐No/Tidak
2. How much are you willing to pay for this product?
   Berapakah harga yang anda sudi bayar untuk produk ini?
   ☑RM 2.00
   ☐RM 3.00
   ☐RM 4.00
   ☐RM 5.00
3. Do you think packaging companies would be willing to buy this product?
   Adakah anda fikir Syarikat pembungkusan bersedia untuk membeli produk ini?
   ☑Yes/Ya ☐No/Tidak

Both statements are true and correct,
Maklumat diatas adalah tepat dan benar,

COMMERCIALIZATION OF SURVEY ON PACKAGING TRAY OF BIODEGRADABLE CORN HUSK FIBER COMPOSITE

NAME/NAMA: MOHD FAIZAL
COMPANY NAME/NAMA SYARIKAT: BJK

1. If this product is marketed, are you willing to purchase this product?
   Kalau produk ini didapati di pasaran, adakah anda akan membelinya?
   ☑Yes/Ya ☐No/Tidak
2. How much are you willing to pay for this product?
   Berapakah harga yang anda sudi bayar untuk produk ini?
   ☑RM 2.00
   ☐RM 3.00
   ☐RM 4.00
   ☐RM 5.00
3. Do you think packaging companies would be willing to buy this product?
   Adakah anda fikir Syarikat pembungkusan bersedia untuk membeli produk ini?
   ☑Yes/Ya ☐No/Tidak

Both statements are true and correct,
Maklumat diatas adalah tepat dan benar,

Figure 5.2 Survey on food packaging potential user

Figure 5.3 Picture with food packaging potential user

5.3 Recommendation

Recommendations for future work and potential for progress are described as follows to further and enhance the production of biodegradable plastic in natural fibre studies that reinforce matrix material:

i. Explore the impact of various coatings, including epoxies, oil, polyester, and their combinations, on the TPCS/CHF composite.

ii. Enhance the potential application of this material by developing antimicrobial properties in the composites.

iii. Develop coatings for the composites to further decrease the hydrophilicity of this material.

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APPENDICES

APPENDIX A GANTT CHART: PSM 1

TASK / PLANNING	WEEK													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
PSM 1: PROJECT BRIEFING														
CHAPTER 2: LITERATURE REVIEW														
Literature Survey														
Report Writing														
CHAPTER 3: METHODOLOGY														
Preparation of Composites														
Report Writing														
CHAPTER 4: PRELIMINARY RESULT & DISCUSSION														
Previous Study Finding														
Report Writing														
CHAPTER 1: INTRODUCTION														
Report Writing														
OTHERS PREPARATION														
Format Thesis														
Gantt Chart														
Slide Presentation Preparation														
Presentation														

MID SEM BREAK

Submit Final PSM 1 Report														

APPENDIX B GANTT CHART: PSM 2

TASK / PLANNING	WEEK													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Receive Previous Project									MID SEM BREAK					
Supervisor Meeting Discussion														
MECHANICAL ANALYSIS														
Tensile Testing														
Flexural Testing														
THERMAL ANALYSIS														
Thermogravimetric Analysis (TGA)														
OTHER TESTING														
Fourier Transform Infrared Spectroscopy (FTIR)														
Scanning Electron Microscope (SEM)														
PRODUCT FABRICATION														
Production of Packaging Tray														
OTHERS PREPARATION														
Report Writing														
Format Thesis														
Slide Presentation Preparation														
Presentation														
Submit Final PSM 2 Report														

Milestone for Gantt Chart PSM 1:

Description	Point	Week
Completition of Introduction	M1	13
Completition of Literature Review	M2	8
Completition of Methodology	M3	11
Completition of Preliminary Result	M4	13
Submission of Final Proposal Report	M5	14

Milestone for Gantt Chart PSM 2:

Description	Point	Week
Completition of Mechanical Testing	M1	4
Completition of Thermal Testing	M2	6
Completition of Other Testing	M3	8
Completition of Product Fabrication	M4	11
Submission of Final Report	M5	14