EFFECT OF THERMAL DEGRADATION ON THE MECHANICAL BEHAVIOUR OF GREEN BIOCOMPOSITES PACKAGING MATERIAL

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This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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DECLARATION

I hereby, declared this report entitled “Effect of thermal degradation on the mechanical behaviour of green biocomposite packaging material” is the result of my own research except as cited in references.

Signature : ……………………………………

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Date : 14/7/2024

APPROVAL

This report is submitted to the Faculty of Industrial and Manufacturing Technology and Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for the degree of Bachelor Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

………………………………………………………………

(Dr. Chang Siang Yee)

ABSTRAK

Biokomposit ialah penyelesaian yang sempurna untuk menghasilkan bahan pembungkusan komersial kerana biodegradasi dan kemampanannya. Walau bagaimanapun, apabila biokomposit terdedah kepada haba ia boleh merendahkan ikatan antara gentian semula jadi dan matriks polimer, mengurangkan kekukuhan dan kekuatan keseluruhan biokomposit. Kajian ini menyiasat kesan degradasi haba ke atas sifat mekanikal dan kehabluran komposit yang terdiri daripada Asid Polilaktik (PLA) dan Polybutylene Succinate (PBS), diperkukuh dengan gentian daun nanas (PALF). PALF sedang menjalani rawatan permukaan gentian yang merupakan rawatan alkali. Serat daun nanas telah dirawat dengan 5% natrium hidroksida dan menjalani analisis FTIR untuk membandingkan komposisi kimia PALF sebelum dan selepas rawatan. Proses fabrikasi gentian daun nanas bertetulang PLA/PBS dilakukan dengan proses mencampurkan leburan dan sampel dimampatkan ke dalam dimensi tertentu yang diperlukan dalam ASTM D790 untuk ujian lentur. Selepas itu, sampel dimasukkan ke dalam ketuhar pada suhu 45°C, 55°C, dan 60°C untuk tempoh 3, 7, dan 9 hari. Kehabluran diperiksa melalui XRD pada sudut 3°-90°. Keputusan menunjukkan kemerosotan peningkatan kehabluran pada suhu 60°C (3,7,9 hari) satu puncak tajam diperhatikan pada 2θ= 16.2°. Sudut mewakili PLA dalam biokomposit. Ini menunjukkan bahawa kehabluran meningkat apabila suhu dan tempoh meningkat. Kehabluran dipertingkatkan dengan masa yang lebih lama dan suhu yang lebih tinggi, yang membolehkan penjajaran rantai yang lebih meluas. Kekuatan fleksibel polistirena EPS adalah antara 0.0750-3.00 MPA dan modulus flexural ialah 0.00628-0.0342 GPa. Julat kekuatan lenturan berdasarkan kajian adalah dari 0.393 -8.039 MPa dan modulus flexural antara 0.122 - 0.48 GPa. Ini menunjukkan bahawa biokomposit mempunyai modulus dan kekuatan flexural yang tinggi berbanding dengan bahan polistirena EPS, menjadikan sampel itu sesuai untuk bahan pembungkusan di Abu Dhabi.

ABSTRACT

Biocomposite is the perfect solution to produce commercial packaging material because of its biodegradability and sustainability. However, when the biocomposite isexposed to heat it can degrade the bond between natural fibers and polymer matrix, reducing the biocomposite total stiffness and strength. This study investigated the effect of thermal degradation on the mechanical properties and crystallinity of thecomposite composed of Polylactic Acid (PLA) and Polybutylene Succinate (PBS), reinforced with pineapple leaf fiber (PALF). The PALF was undergoing fiber surface treatment which was an alkaline treatment. Pineapple leaf fiber was treated with 5% sodium hydroxide and underwent FTIR analysis to compare the chemical composition of PALF before and after treatment. The fabrication process of PLA/PBS reinforced pineapple leaf fiber was done by melt-mixing process and the sample was compressed into a specific dimension required in ASTM D790 for flexural test. After that, the sample was placed in the oven at 45°C, 55°C, and 60°C to determine the heat exposure effect on the flexural properties of the biocomposite for a duration of 3, 7, and 9 days. The crystallinity was checked through XRD at angles of 3°-90°. The result showed crystallinity increase degradation at a temperature of 60°C (3,7,9 days) one sharp peak was observed at 2θ= 16.2°. The angle represents the PLA in the biocomposite. This showed that crystallinity increased when temperature and duration increased. The crystallinity is improved by longer times and higher temperatures, which enable more extensive chain alignment. The flexural test showed a decrease in flexural strength and an increase in flexural modulus during exposure to a temperature of 45°C and both flexural strength and flexural modulus were decreased during exposure to a temperature of 55°C and 60°C. The interaction between the natural fiber filler and the polymer matrix affects the biocomposites' thermal stability causing the decrease in flexural strength and modulus. The flexural strength of EPS polystyrene ranged between 0.0750-3.00 MPA and the flexural modulus was 0.00628-0.0342. The range of flexural strength based on the study was from 0.393- 8.039 MPa and flexural modulus between 0.122 - 0.48 GPa. This showed that biocomposite has high flexural modulus and strength compared to EPS polystyrene material, making that sample suitable for packaging material in Abu Dhabi.

DEDICATION

This work is dedicated to my wonderful parents, Encik Ahmad Shukri bin Mat Jusoh and Puan Ramidah binti Abu Bakar who have never failed to give me financial and moral support. They give all their support to me and believe that I can do my project

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LIST OF ABBREVIATIONS

FMCG - Fast-moving consumer goods

FTIR - Fast-moving consumer goods

KOH - Pottasium Hydroxide

LiOH - Lithium Hydroxide

LLPDPE - Low-density polyethylene

NFRPC - Natural fiber-reinforced polymer composite

NFC - Natural fiber composites
NaOH - Sodium Hydroxide

PLA - PolyLactic Acid

PBS - Polybutylene Succinate

PP - Polypropylene

PVC - Polyvinyl chloride

SEM - Scanning Electron Microscopy

TS - Tensile Strength

TPALF - Treated pineapple leaf fibre

UPALF - Untreated pineapple leaf fiber

XRD - X-ray Diffraction

YM - Young’s Modulus

LIST OF SYMBOLS

mm	-	milimetre
%	-	Percent
g/cm^3	-	Grams per centimetre cube
wt. %	-	Weight percent
MPa	-	Mega Pascal
GPa	-	Giga Pascal
°C	-	Degree Celsius
phr	-	Part per hundred resin
mm/min.	-	Millimetre per minute
rpm	-	Revolution per minute
kN	-	Kilo newton

CHAPTER 1

INTRODUCTION

1.1 Research Background

Petroleum-based polymer is widely used in this world as material for packaging such as polystyrene. The growth of plastic is mainly owing to its excellent characteristics and processing possibilities. However, challenges like as the rising cost of petrochemicals, their decreasing availability, and the environmental persistence of these materials after their useful lifespan are posing serious problems for the packaging business (Nurul Fazita et al., 2016). Sustainable packaging solutions are rising as environmental challenges become more widely recognized. Because they are composed of renewable resources and have the potential to lessen the environmental effect of packaging materials, biocomposite are a promising alternative.

Biocomposite are made from natural fibers or particles that are extracted from plant resources mixed with a matrix. Pineapple, kenaf, wood, and bamboo are examples of plant resources. The matrix can be Polylactic Acid (PLA) and Polybutylene Succinate (PBS). Biocomposite is suitable for eco-friendly packaging because of its biodegradability and sustainability. It offers advantages in terms of sustainability and biodegradability but thereare some challenges related to the mechanical properties, moisture, and durability. Biocomposite materials are not a problem-free substitute, and they have some drawbacks such as poor moisture resistance (hydrophilicity), fiber/matrix incompatibility, supply logistic issues, low thermal stability, flammability, poor electrical properties (Andrew & Dhakal, 2022). To overcome this problem, treatment can be done tothe natural fibre. One of the treatments that can be done to the natural fiber is alkaline treatment. The mechanical properties such as tensile strength, impact strength, flexural strength, and elastic modulus of the fiber were all raised by alkali treatment (Mohammed et al., 2023).

It is also important to note that thermal degradation is the process where the action of heat on the material or the product causes a loss of physical and mechanical properties. This process results in the material or product losing its physical and mechanical qualities. A material's qualities can change over time because of environmental conditions including temperature, pressure, and humidity; this process is known as degradation (Lila et al., 2019). Besides that, thermal degradation also impacts the crystallinity of the biocompositethat may result. The thermal degradation process is very important in analyzing the feasibility of biocomposite material that can be used for packaging applications. This study is to investigate the effect of thermal degradation of the PLA/PBS reinforced with thepineapple leaf when it is exposed to certain temperatures to produce strong, temperature-resistant packing materials that meet both environmental and performance criteria.

1.2 Problem Statement

Petroleum-based polymers are widely used throughout the world. An increasingly pressing issue on a global scale is the proper disposal of plastic trash. Waste from packaging continues to be a major environmental issue, and there is rising demand from customers all over the world (Stark & Matuana, 2021).

Suitable packaging is the most important thing to make sure that all the items delivered are safe during transportation. That is possible that different products will need different degrees of weather resistance, depending on their susceptibilities and the kind of environmental circumstances they might be exposed to while being transported or stored. Biocomposite seems to be the perfect solution to produce commercial packaging material produced from natural fiber and polymer. However, when the biocomposite is exposed to heat or a certain temperature it can degrade the connection between natural fibers and polymer matrix, lowering the overall strength and stiffness of the biocomposite. This project was collaborated with an institution in Abu Dhabi and aims to utilize the outcome of the project there. The weather in Abu Dhabi was hot compared to Malaysia. The temperature at Abu Dhabi was between 38°C and 45 °C.

1.3 Objectives

The objectives are as follows:

- To investigate the effect of thermal degradation on the crystallinity of PLA/PBS composites reinforced pineapple leaf fiber using XRD and FTIR analysis.

- To investigate the effect of thermal degradation on the flexural properties of the PLA/PBS composites reinforced with pineapple leaf fiber using flexural test and SEM analysis.

1.4 Scopes of the Research

The scopes of research are as follows:

The materials used to produce biocomposite are Polylactic Acid (PLA), Polybutylene Succinate (PBS), and Pineapple Leaf Fiber (PALF). The pineapple leaf fiber underwent fiber treatment which is an alkaline treatment with 5% NaOH. After that, PLA/PBS/PALF composites were produced using a Brabender internal mixer and compression molding. This composite composition is based on the optimum findings of previous studies withinthe research team. After the fabrication process, the sample underwent a thermal degradation test. The sample was tested in the humidity chamber at 45, 55, and 60 (°C) and for 3, 5, and 7 days at 60% humidity. After the degradation test sample underwent mechanical testing which was the flexural test (ASTM D790) with a strain rate of 2mm/min. The sample underwent crystallinity analysis by using an X-ray diffraction.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview

This chapter covers rigid and flexible packaging, reinforcement of biocomposite with natural fiber, processing method that can be used to fabricate biocomposites. It also covers surface treatment that can be done on natural fiber. The effect of fiber loading, fiber treatment and fibers size on mechanical properties of biocomposite are also reviewed. Lastly, the effect of thermal degradation of biocomposites when subjected to biocomposite such as on thermal properties, mechanical properties, and crystallinity are also included in fulfillment the objective of the study.

2.2 Packaging

The science, art, and technology of enclosing or safeguarding goods for use, sale, distribution, and storage is known as packaging. In the commercial world, packaging is crucial because it enables the flow of goods from manufacturing facilities to clients and endusers (Ojha et al., 2015).

Packages are boxes, containers, or any covering that can be removed or left on that is meant to protect, keep things in use for a long time, or make selling them easier. Packaging is an intricate system of technology and economics that protects a certain substance from being made to being consumed. This makes it easier to identify, market, and negotiate that substance (Sastre et al,.2022).

Transportation's demands drove the evolution of packaging, bridging the gap between production and consumption. Today, packaging embodies the entire product journey, from factory floor to store shelf. The packaging sector has a substantial impact on the environment and cost efficiencies across supply chains, including packaging design creation, material procurement, and end-of-life treatment (Morashti et al,. (Basfar et al., 2023)2022). Packaging can be categorized into two types which are flexible packaging and rigid packaging.

2.2.1 Flexible packaging

Among the many forms of packaging popular in Malaysia, flexible packaging stands out. Plastic has been a popular packaging material because of its low cost, durability, and excellent barrier qualities. (Sook et al., 2020). Flexible plastic has many useful uses, such as packaging that helps the goods inside last longer and be easier to handle (for example, packing food and hygiene products) (Ahamed et al., 2021) Food, chemicals, medications, beverages, and electronics are among the many products that use flexible packaging in storage and packaging. Flexible packaging pack categories include various forms of bags, pouches, films, sachets, and squeezable containers (Farrukh et al., 2022).

Flexible packaging makes up around half of all plastics found in household trash in industrialized nations (Horodytska et al., 2018). Common monolayer plastics used in packaging include polypropylene (PP), polyvinyl chloride (PVC), and low-density polyethylene (LLPE). (Veksha et al., 2020).

2.2.2 Rigid packaging

Rigid packaging refers to packaging that utilizes sturdier and more durable materials compared to flexible packaging. Examples of stiff packing materials encompass glass, durable polymers, cardboard, metal, and other similar substances. Typically, inflexible packaging materials are pricier than their flexible counterparts and tend to have considerably larger carbon footprints compared to flexible packaging (Dube., 2020).

When it comes to security, rigid packaging is better than flexible packaging. On the other hand, rigid packaging is worse for the earth than flexible packaging. The materials used to make polymers and packing, as well as how they are disposed of at the end of their useful lives, all have an impact on the environment. Pollutants released into the open environment at any of these steps can have bad effects on the environment, such as climate change, water and air pollution, acidification, and more. As the population grows, so does the number of people who buy fast-moving consumer goods (FMCG). This means that hard packaging is likely to grow quickly. Rigid plastic packaging has many benefits, including being strong against impact, stiff, and a shield. These factors have helped the rigid plastic packaging market grow over the past few years (Srivastava et al,.2022).

2.3 Biocomposite

Biocomposites are materials that are biocompatible and good for the environment. They are made up of many kinds of organic and artificial substances, like natural and man-made polymers, polysaccharides, proteins, sugars, ceramics, metals, and nanocarbon. Biocomposites are less expensive because of their biodegradability and other increased mechanical qualities, biodegradable polymer composites have significant potential in the industrial sector. Significant research is currently being conducted to improve the properties and production capacity of biodegradable polymers (Reshmy et al., 2021).

Biocomposites are less expensive and lighter than traditional composites, and they have the added virtue of being made from renewable and biodegradable materials, making them the environmentally better alternative (Afzal et al., 2021).

Biopolymer matrices reinforced with reinforcements derived from renewableresources are the subject of active investigation in the field of biocomposites. This is because the materials have promising mechanical qualities, can be recycled after use, and degrade naturally (Aliotta et al., 2019)

2.3.1 Biopolymer reinforce with natural fibers

Natural fiber composites, often called natural fiber-reinforced polymer composites, have seen an increase in value. Natural fibers (such as hemp, sisal, jute, kenaf, flax, and others) are utilized in polymer-based matrices as reinforcing material (fillers) (Kamarudin et al., 2022). Many natural fibers, as opposed to synthetic ones like carbon, glass, and aramid, were investigated for use as reinforcing in the production of polymer composites due to economic and environmental considerations (Protim et al., 2023).

The use of plant-based natural fiber as reinforcement of polymer-based materials can be targeted to minimize the usage of synthetic fibers and non-biodegradable polymers in composite structures. Excessive usage of petroleum-based plastics generates massive amounts of non-decomposable solid waste, severely depleting landfill capacity (Lau et al., 2017).

The use of natural fibers as reinforcement in the production of various composites has been the subject of much research on a global scale. In comparison to carbon or glass fibers, natural fibers offer several benefits, such as being more accessible, having a lower density, being more specific, resistant to corrosion, inexpensive, biodegradable, and causing less wear and tear on processing equipment (Kumar Yadav et al., 2023).

There are a few examples of biocomposites used in current packaging applications such as Lignin-poly (lactic acid) biocomposites. These biocomposites are utilized for food packaging applications and have favorable melt processing characteristics and flexibility (Esakkimuthu et al., 2022).

2.4 Processing Method

There are the most utilized methods for melt blending of polymer composite which are injection molding, twin-screw extruder.

2.4.1 Injection molding

Injection molding is a well-established method used to efficiently produce large quantities of plastic components. Injection molding is a manufacturing method that is widely used in a variety of industries, including consumer electronics, sports goods, cars, medical equipment, and optical lenses. This is because it is cost-effective, highly efficient, and can produce components that are both detailed and accurate (Chen et al., 2019).

This method involves shaping molten material to fit a mold's shape by pressing it into the mold with anything like a screwdriver or a ram while subjected to high pressure. The last step of production is to remove the finished product from the mold after it has solidified inside. Typically, an injection molding machine will have a clamping unit and a plastinating injecting unit. The material is fed into the heating barrel, and during the processes of heating,mixing, compression, and melting take place, through a hopper in the plastinizing inside unit.With the heating bands, it can regulate the temperature of various materials. Injection and shaping of the molten material take place in the cavity unit, which is typically composed of two halves that join to create a three-dimensional (3D) shape (Dezfooli et al., 2020).

There was a problem during using injection molding for the fabrication of biocomposite, when natural fiber composite materials were frequently associated with low physical resistance to moisture and temperature during production. Increased fiber loading cause agglomerations and voids in the molded composite. This is owing to the mixing yields of non-homogeneous feedstocks with defective fiber-matrix impregnation. Furthermore, biocomposite materials may fail to meet the flowability requirements of the plastic injection molding process. The injectability of biocomposite materials is strongly dependent on the early stages of feedstock preparation (Md Radzi et al., 2021).

Figure 2.1: Injection molding machine (Guanxin Plastic Machinery Co., 2020).

2.4.2 Twin screw extruder

Twin-screw extruders can handle solids and materials with a high viscosity, in addition to providing outstanding mixing efficiency, large energy inputs, and the capacity to manage these materials. Because of this, they are essential to a wide variety of businesses, including the chemical and pharmaceutical sectors, for blending, reacting, and compounding. The efficient functioning of these extruders necessitates the use of dynamic models that can accurately depict the processes of starting up, shutting down, and changing products. Moreover, these models can be utilized to optimize the configuration and operational parameters of screws (Cegla and Engell 2020).

The twin-screw extruder regulates the pressure and temperature within the barrel to precisely process the material. It can control the flow of heat in each barrel or zone to keep it at a certain temperature, and It can manage the pressure by configuring the design of the screws as well as the rotation speed of the screws. An intense mixing zone, a conveying zone,and a feeding zone are the three primary components that are found in the majority of twin-screw extruder structures. That the screws can be arranged in a variety of different configurations, a twin-screw extruder can be used for a variety of purposes (Dhaval et al., 2022).

The small diameter of the screw, the presence of grinding zones in the screw profile (which could result in fiber shortening), and the large ratio of screw length to diameter (which could cause thermal degradation of the biocomposite) could all contribute to poor mechanical properties of biocomposite produced by twin-screw extrusion. It should be noted, however, that biocomposite produced using a twin-screw extruder had lower porosity and water absorption, which could be attributed to the fact that the twin-screw extruder was equipped with a degassing module for the plasticized biocomposite during the extrusion process (Janowski et al., 2022).

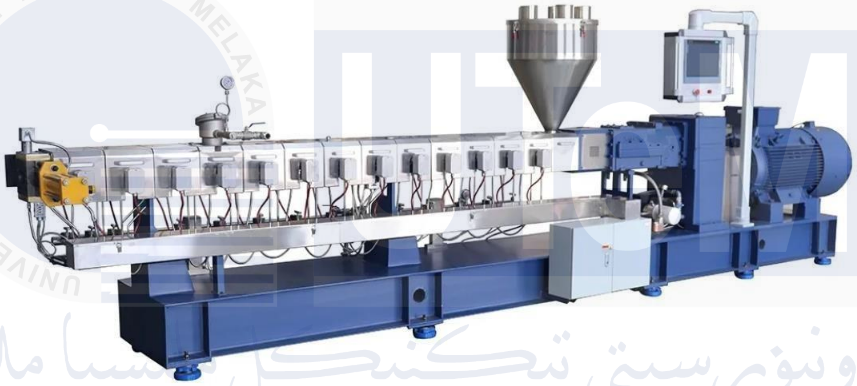


Figure 2.2: Twin screw extruder machine (Cowell (Nanjing) Extrusion Machinery Co., 2022).

2.5 Compatibility of Biocomposite at Fiber/Matrix Interface

One important attribute that impacts the mechanical properties of biocomposites is their compatibility with the fiber/matrix contact. The mechanical qualities, manufacturing advantages, and environmental benefits of natural fibers have sparked interest in their usage as reinforcement in polymer composites. Nevertheless, the hydrophilic properties of the fibers lower the degree to which they are compatible with the matrix. Because of this incompatibility, the composites have qualities that are not very good mechanically (Kabir et al., 2012).

The properties of bio-composites are greatly affected by the interaction between the fibers and the matrix, hence adding fibers to a matrix changes the matrix's properties significantly. To achieve the most desirable mechanical qualities, it is crucial to create strong contact connections. Strong adhesion between fibers and matrices significantly enhances several physical properties (Signori et al., 2012).

When there is insufficient contact between fibers and matrix, mechanical and physical characteristics are diminished. The hydrophilic nature causes the fibers to have a difficult time dispersion within matrices, which is one of the factors that contribute to weak interfaces. The hydrophobic matrix material is incompatible with hydrophilic fibers, and the composite's ability to transfer stress between the matrix and fiber is reduced. This is because the matrix material is hydrophobic. The microcracking that occurs because of changes in the dimensions of the fiber affects the adhesion that exists between the fiber and the matrix materials. To improve fiber/matrix adhesion, many methods have been used, such as surface treatments and fiber treatments. Surface treatments not only lessen moisture sensitivity but also enhance fiber/matrix adhesion (Zwawi 2021).

2.6 Surface Treatment of Natural Fiber

There are a few types of surface treatments for natural fiber which are acetylation, silane, and alkaline treatment.

2.6.1 Acetylation treatment

Acetylation is a chemical reaction that takes hydrogen atoms out of the hydrophilic hydroxyl branches of cellulose molecules. Acetic acid is usually used as a catalyst. One way this is done is by adding acetyl groups to the structure of the cellulose were hydrogen atoms used to be. Adding acetyl groups to the cellulose structure of natural fibers is called acetylation. This can be done with or without an acid agent. The sensitivity of natural fibres to the absorption of moisture is reduced through the process of acetylation. Therefore,enhancing the dimensional stability and durability of the fibres that are utilised for reinforcement, as well as the fiber-reinforced composite that is formed because of thisimprovement. Acetylation has been shown to produce fiber composites that exhibit superior performance across all fiber loadings, according to reports (Oladele et al., 2020). The hydroxyl group (-OH) in the natural fiber is changed to an acetyl group (CH3CO) by this process (Seisa et al., 2022). When acetylation is applied, the mechanical qualities get better. This is because lignin and extractible are removed, the cellulose content goes up a little, and asmall amount of hemicellulose turns into acetylated hemicellulose.

2.6.2 Silane treatment

The silane treatment acts as a coupling agent by filling the micropores that are present on the surface of natural fibres. This results in an increase in the adhesion that exists between the various materials that are present at the interface. Enhanced adhesion at the interface is achieved through the utilisation of a coupling agent, which is employed to build connections between the natural fibre reinforcement and the matrix (Seisa et al., 2022).

In the silane treatment process, the alkoxy group that can be broken down by water changes into an unstable silanol. The silanol subsequently undergoes condensation with adjacent silanol molecules, resulting in the formation of an oligomer. (OH) groups of silanol can form hydrogen bonds with cellulosic fiber, as described by equations 1 and 2. Additionally, the alkyl group of silanol facilitates chemical bonding with the polymer matrix.Consequently, the fiber treated with silane exhibits a high level of hydrophobicity because ofthe decrease in hydroxyl groups. This characteristic is beneficial for a hydrophobic polymer matrix due to its compatibility with the matrix. (Radoor et al. 2021).

$$CH_2CHSi(OC_2H_5) \rightarrow CH_2CHSi(OH)_3 + 3C_2H_5OH \qquad equation\ 1$$

$$CH_2CHSi(OH)_3 + fiber - OH \rightarrow CH_2CHSi(OH)_2O - fiber + H_2O \qquad equation\ 2$$

Silane formed a bridge at the contact, allowing for strong fibre-matrix interaction and thus good tensile characteristics. The stronger tensile properties of silane-treated composites were due to silane surface modification, which got rid of extra lignin, wax, and hemicellulose content from the natural fibre substrate. This made the bonding between the reinforcement and matrix phases of the composite better. Fiber-matrix interfacial bonding can also make polymer materials stronger against impacts (Md Nor et al., 2023).

2.6.3 Alkaline treatment

Alkaline treatment is a procedure in which natural fibre is submerged in a concentrated aqueous solution, such as NaOH, LiOH, or KOH, to induce significant expansion, leading to alterations in the intricate composition, size, shape, and physicalcharacteristics (Tan et al., 2017). This approach is commonly employed to alter the surface characteristics of natural fibers. This technique involves submerging the organic fiber in sodium hydroxide solution for a designated duration at a predetermined temperature. Alkali is added to fiber, which decreases the number of hydroxyl groups on its surface, breaks down non-cellulose components such as wax, oil, and lignin, and disturbs the hydrogen bonds inside the fiber. The result from the treatment makes the strength, and water-repellentproperties of fiber increase, leading to an enhanced the ability to be compatible with the hydrophobic polymer matrix (Radoor et al., 2021).

Alkaline treatment increased the surface roughness of the fiber and thus resulted in better fiber/matrix adhesion. The discontinuity particle fiber-reinforced epoxy weakened the material as the homogeneity of the material declined. In comparison to untreated fiber, alkaline treated fiber also resulted in better dispersion and stress transfer, thus improving the impact strength (Tan et al., 2017). The mechanical properties and load transfer were both improved after being treated with an alkaline solution, which strengthened the materials by strengthening the bonds between the fiber and matrix (Archana et al., 2022)

2.7 Properties of Biocomposite

The properties of biocomposites were mentioned about effect of fiber loading on the mechanical properties of PLA reinforced with Natural Fiber, effect of fiber treatment on the mechanical properties and effect of fiber size on the mechanical properties.

2.7.1 Effect of fiber loading on the mechanical properties of PLA reinforced with natural fiber

According to the experiment, the tensile strength of the materials did not improve at 10% kenaf loading, showing that there was poor dispersion and fiber distribution, which resulted in poor adhesion between the fiber and the matrix. This is because the polymer resin is incapable of transmitting and distributing the applied stress to the kenaf fibers, resulting in decreased strength. Fiber distribution and dispersion began to increase around 20% to 30% fiber loading, resulting in greater strength than 10% fiber loading. Similar results were obtained when 10-30% kenaf fiber was added to a PLA matrix. They reported that the tensile strength of the composite rose as the number of fibers in the composite increased. However, at 40% fiber loading, the materials' tensile strength declined due to insufficient matrixwetting of the fiber. Aside from that, because there are no polymer chains between the fiber layers, this could be generated by fiber-fiber interaction and fiber agglomerates in the matrix.The presence of fiber agglomerates would increase weak areas inside the composite, affecting its mechanical qualities significantly. The inclusion of kenaf increases the modulus, however, the addition of more fiber does not improve the modulus in composites (Ismail et al., 2018).

Tensile characteristics of PALF/PLA composites as a function of PALF content are shown in Figure 2.6. Figure 2.6 (a) shows that increasing the PALF loading content improved both the tensile strength and the tensile modulus of the composites. Figure 2.6(b) shows the elongation at the break of the biocomposite. The elongation at break increased when the PALF content was raised from 10% to 40% by weight. Composites exhibited a reduction in elongation at the break because of PALF aggregation when the fiber content wasraised to 50%by weight. (Kaewpirom et al., 2014).

The composite's Young's modulus increases as the fiber content increases. Young’s

Modulus was found to be 2490 MPa at 40% fiber weight. Tensile strength also increases with increasing fiber content, with 40% Wt. of fiber yielding a tensile strength of 62.1 MPa. The percentage of elongation increases with increasing fiber content in a specimen, starting low and progressively increasing to 4.5% at 40% Wt. of fiber.

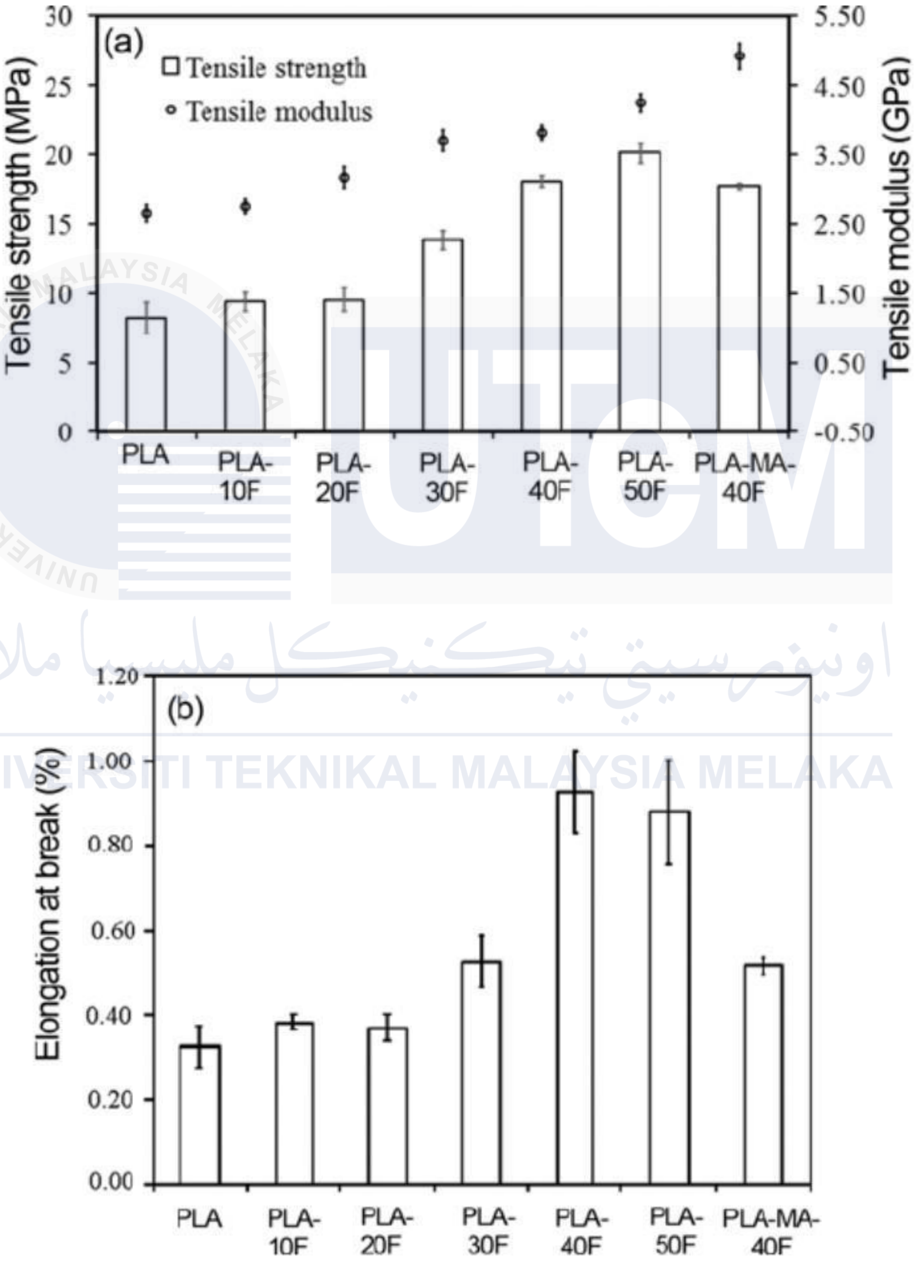


Figure 2.3: Tensile properties of PALF/PLA composites (a) Tensile strength and tensile modulus and (b) Elongation at break (Kaewpirom et al., 2014).

On the other hand, the flexural strength of the composites was improved by increasing the kenaf loading from 10% to 30% by weight. However, at 40% kenaf loading, flexural strength was reduced by approximately 16.32%. This suggests that 30 wt% is the optimum fiber loading and that increasing kenaf loading would result in lower flexural strength. The good interfacial adhesion between kenaf and polymer matrix is expected at 30%fiber loading due to chemical interactions such as hydrogen bonding. In the composite, hydrogen bonds exist between the hydroxyl groups in the kenaf fibers, the terminal hydroxyl groups, and the carbonyl groups of the PLA ester groups. With the existence of fiber bundlesand greater fiber-fiber interactions, insufficient wetting of the integrated fiber by the polymermatrix resulted in a 40% drop in the flexural strength of kenaf loading (Ismail & Ishak, 2018).

By increasing the weight percentage of composite fiber loading, the fibers can absorb both compression and shear, increasing flexural strength magnitude. On the other hand, increasing the fiber content beyond a certain point may reduce the likelihood of shearing action, which is the primary factor in determining the outer layer and its thickness.

Based on Figures 2.4 and 2.5 the young’s modulus and tensile strength of composites made of raw pineapple leaf fiber untreated PALF and Polylactic acid (PLA) and treated pineapple leaf fiber (TPALF) and PLA, with varying amounts of fiber. Figure 2.5 shows that the strength of the UPALF/PLA mixture goes up sharply by 40.78% when 10% of the fibers are added. This is compared to the strength of the virgin PLA. It found that the tensile strength of 15% and 20% fiber-loaded UPALF/PLA composites is 24.2% and 20.92% higher than that of pure PLA. For TPALF/PLA composites, adding 10% fiber increased the strengthby 44.33%. Adding 15% and 20% fiber increased the strength by 8.1% and 15.25%, respectively, compared to pure PLA. When adding 10%, 15%, or 20% fibers to UPALF/PLAcomposites, the tensile modulus goes up by 6.93%, 12%, and 20.8%, respectively, compared topure PLA. For TPALF/PLA composites, the strength went down by 4.27 percent when 10% fiber was added, and it went up by 0.27% and 11.47 percent when 15% and 20% fiber were added, compared to pure PLA. Greater agglomeration at higher loading fractions led to lowerstrength at higher loading fractions (Sheela et al., 2019).

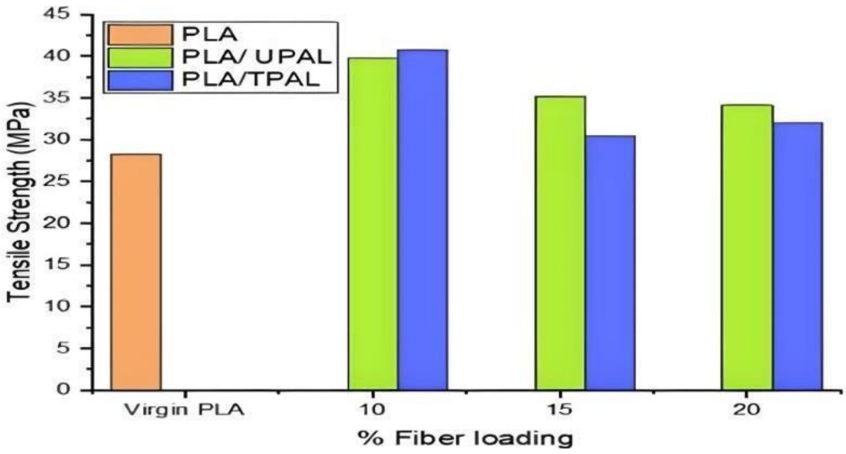


Figure 2.4: Tensile strength of UPALF/ PLA composites (Sheela et al., 2019).

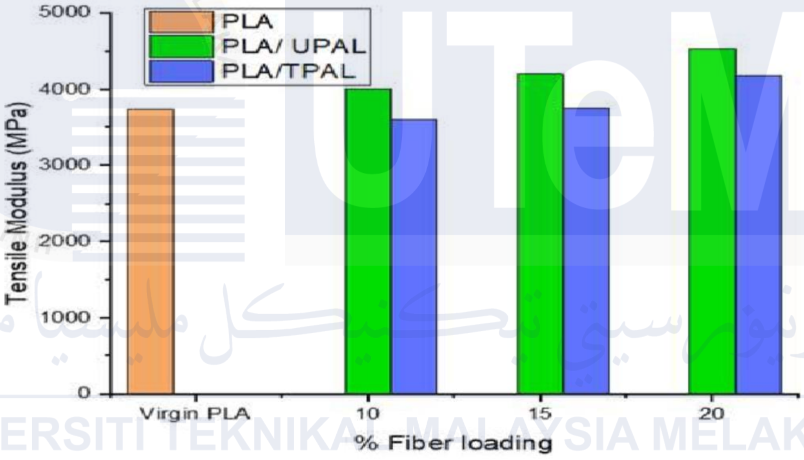


Figure 2.5: Tensile modulus of UPALF/ PLA composites (Sheela et al., 2019).

2.7.2 Effect of fiber treatment on the mechanical properties

Fiber treatment is critical in changing these qualities, ensuring improved compatibility between natural fibers and the matrix, and improving overall performance.

Natural fibers are typically surface treated to improve their mechanical and thermal properties before being used to make composite materials. Fiber surface modification can increase fiber-matrix interfacial bonding, roughness, and wettability while decreasing the fibers' moisture absorption. As a result, the mechanical and thermal properties of the composites produced can be improved. Physical and chemical treatments are the most common ways to alter the outer layer of natural fibers (Neto et al., 2021).

The measure of crystallinity, the average surface roughness, and the intensity of absorption bands can all be changed by treating the fiber surfaces. It is because of these changes that the panels made with this type of reinforcement absorb less water and grow less. This makes the composite material work better over its lifetime.

Changing the surface of the threads can make the material much stronger and stiffer, as shown by physical and mechanical tests on panels made with treated fibers. Surface treatments encourage the formation of interfibral gaps and the resin's anchoring mechanism to the fibers, which improves load transfer between the matrix and reinforcement (Sánchez et al., 2020).

The impact of alkali (NaOH) treatment on the mechanical characteristics of jute fiber-reinforced cement composite and the interaction between the fiber and matrix interface.Their results indicate that treating jute fibers with alkali enhances both tensile strength and % elongation, hence boosting the mechanical strength of cement composites. Moreover, the fibrillation of the fibers resulting from the alkali treatment enhances the surface area available for bonding at the interface between the fiber and the matrix (Jo et al., 2016).

The selection of treatment is important since certain treatments are more effective than others. Alkaline treatment is a frequently used chemical treatment process for natural fibers. According to the authors of the reference, the alkaline treatment has two effects on the surfaceof the fiber. Firstly, it makes the fiber surface rough, which can improve the adherence of the fiber to the matrix. Secondly, it removes hemicelluloses, waxes from fiber surfaces. As a result, the surface of the fibers becomes more chemically consistent, and the amount of cellulose exposed on the fiber surface increases, leading to better compatibility between the fiber and the matrix (Yimer & Gebre, 2023).

2.7.3 Effect of fiber size on the mechanical properties

Short fibers are good reinforcements for polymer materials. Adding even small quantities of fibers can significantly boost composite strength. However, high fibre dosage causes poor dispersion and interface adhesion, resulting in reduced stiffness and strength efficiency. Fiber length often improves mechanical qualities in low-content, short-fiber reinforced composite (Capela et al., 2017).

Tensile characteristics, also known as tensile strength, are higher for fibre lengths 20 mm, which indicates a significant connection between the matrix and the fibre. The fibre length of 25 mm results in a decrease in tensile strength. This is most likely due to the fact that it is difficult for the resin to permeate between the areas that correspond to the fibre and the resin. This leads to poor wetting characteristics, which in turn reduces the effectiveness of stress transmission at the matrix-resin interface. (Anand et al., 2022).

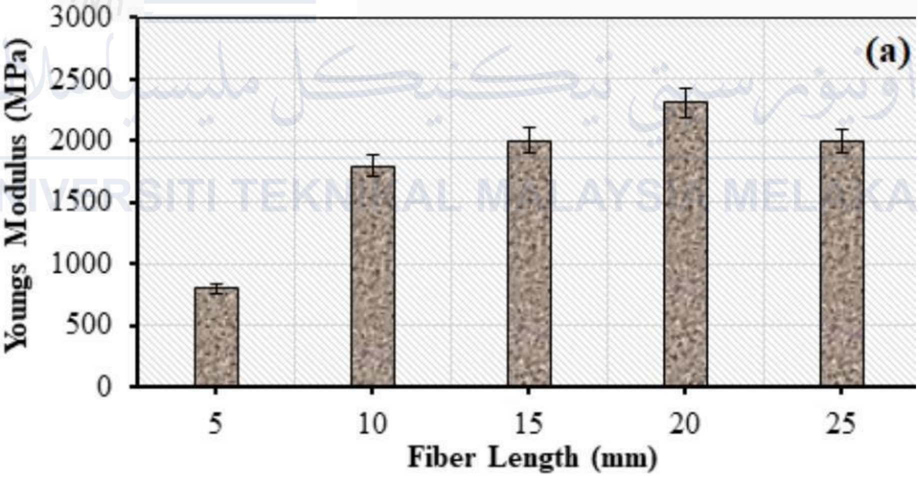


Figure 2.6: Tensile test result (a) Young’s modulus vs fiber length (Anand et al.,2022).

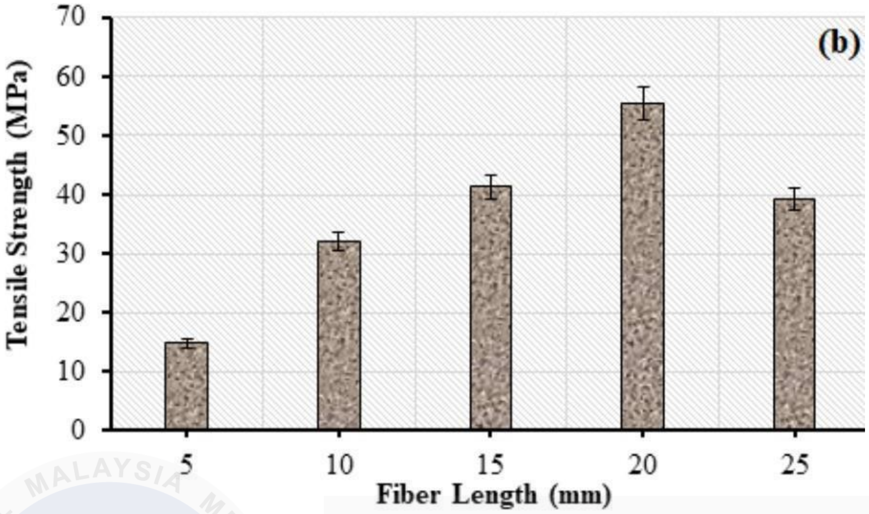


Figure 2.7: Tensile tests result for (b) Tensile strength vs. fiber length (Anand et al., 2022).

2.8 Thermal Degradation of Biocomposite

Thermal degradation of biocomposite refers to the destruction of these materials' molecular structures because of overheating, resulting in a loss of characteristics. When heat exposure causes a material, such as a polymer, to go through harmful chemical changes even in the absence of other substances like oxygen, the process is known as thermal degradation. The biocomposite will be subjected to a certain temperature and duration and after that, the effect of crystallinity and mechanical properties of the biocomposite will be tested to observe the performance of biocomposite.

The change in molecular chain organization, filler-matrix interfacial adhesion, and interaction throughout the organization resulted in changes in the overall thermal characteristics of the biocomposite.

2.8.2 Effect of thermal degradation on Crystallinity

When subjected to aging, the thermal behavior profiles of amorphous and crystalline structures in polymer composites differed. Depending on the aging intensity, the effect of aging on the crystallinity of the samples can be increased or diminished. The degree of crystallinity rises when a polymer experiences diverse thermal-oxidative aging processes, except during longer-duration aging studies in a higher temperature oven, which leads to the breakdown of the polymer chains. Two causes could cause the polymers' crystallinity to rise. The first possibility is that the samples were relieved of thermal stress (produced during the fabrication process), allowing the spherulites to become larger in size. The aging process in a high-temperature oven or chamber allows for the rearranging and reorganization of molecular chains. Another process that can occur is the re-alignment of broken chains (due tochain scissoring) into a more ordered structure in the semicrystalline portion's amorphous areas (as seen in Figure 2.11 below). The aging process proceeds more quickly in the non- spherulite zones due to the weaker bonds and cleaved-free regions located in the amorphous portion of the polymer chains. After rapid weathering, the degree of crystallinity of clean PLAincreased by up to 50% (Chang et al., 2020). The increase in crystallinity of PLA and its composites, when subjected to −20 °C to 65 °C at various times (Lila et al., 2019).

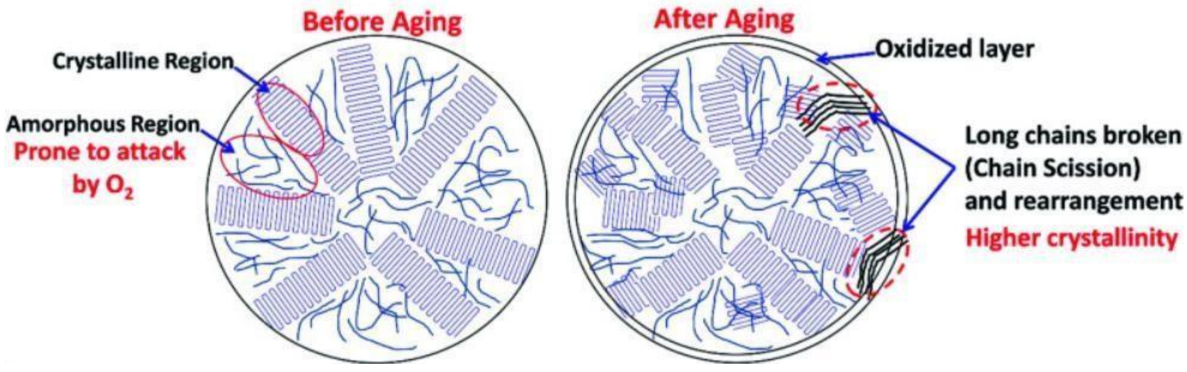


Figure 2.8: Structure changes of a semicrystalline polymer in high-temperature aging oven (Lila Etal., 2019).

2.8.3 Effect of thermal degradation on mechanical properties

The effect of temperature on biocomposites can have an impact on their performance and properties. Biocomposites are composed of fibers and biopolymers, such as cellulose, collagen, and polylactic acid (PLA). However, these materials tend to be unstable at temperatures higher than 200˚C and are not suitable for processing at elevated temperatures (Andrew & Dhakal, 2022).

Based on figure 2.12, the flexural strength of kenaf/PP composites peaks at 105 MPa at 30°C, but it significantly degrades to an average of 70 MPa as the temperature reaches 120°C. It was noticed that the flexural strength of pure PP decreased with rising temperature, which was the same trend as was seen (Radzuan et al., 2020).

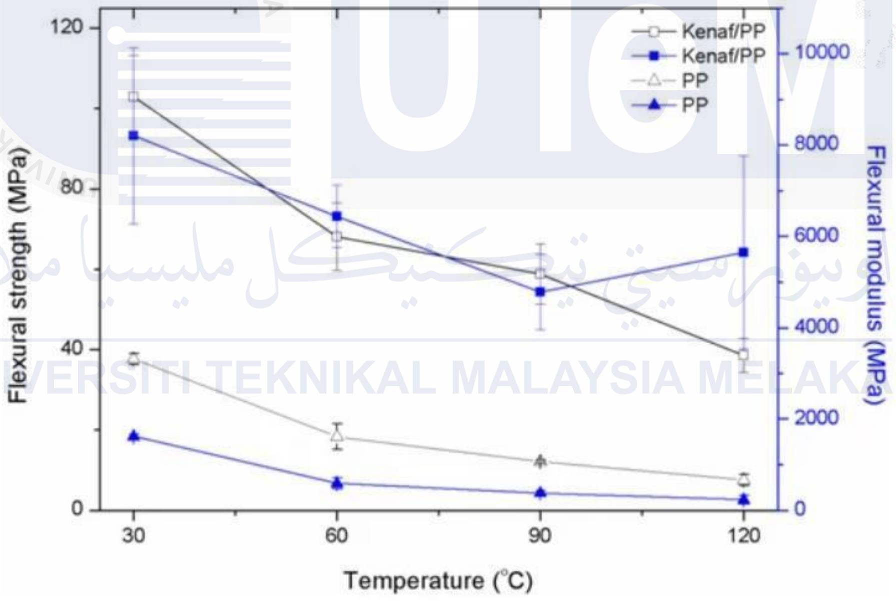
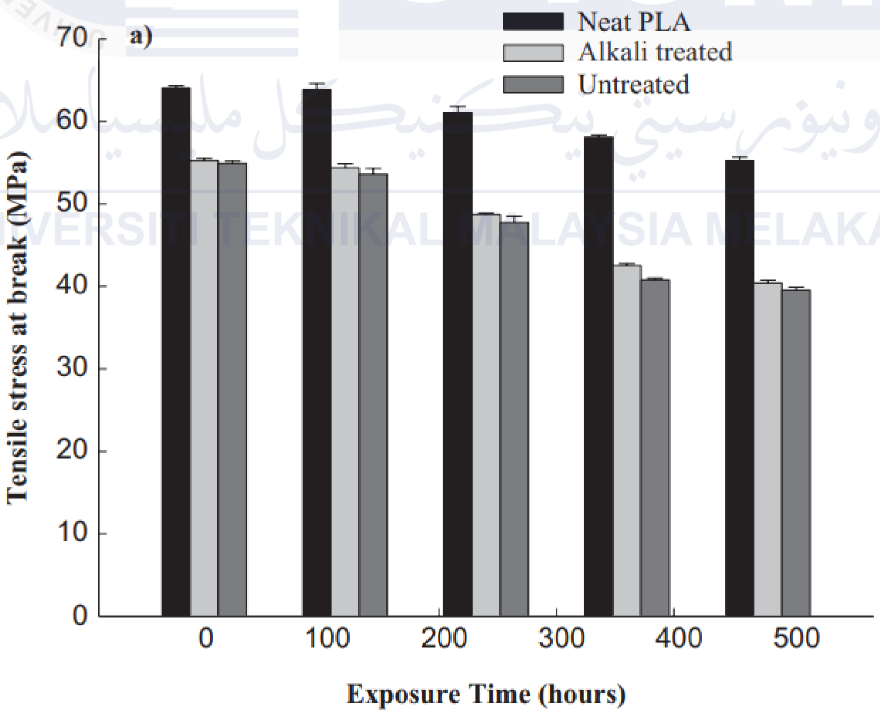


Figure 2.9: Flexural strength and modulus of kenaf/pp composites across temperatures (Radzuan et al., 2020).

Based on Figure 2.13, tensile strength (TS) declines with prolonged weathering. Untreated biocomposites show the most significant reduction in TS. Untreated biocomposites experienced a 28% loss in tensile strength from 54 to 39 MPa, whereas treated biocomposites saw a 22% decrease. Increased rapid aging time leads to a decrease in neat PLA due to plasticization, swelling, and photochemical degradation (Isadounene et al., 2018).

Figure 2.14 shows that untreated biocomposites had a 29% decrease in Young's modulus (YM) from 1593 MPa to 1134 MPa, whereas treated ones had just a 17% loss. Accelerated ageing led to decreased tensile strength and Young's modulus in both untreated and alkali-treated biocomposites. Unfortunately, untreated biocomposites showed the largest decrease in mechanical characteristics compared to alkali-treated biocomposites. After accelerated aging, the clean PLA turned yellow, whereas the biocomposites developed a darker brown color (loss of surface gloss) (Isadounene et al., 2018).



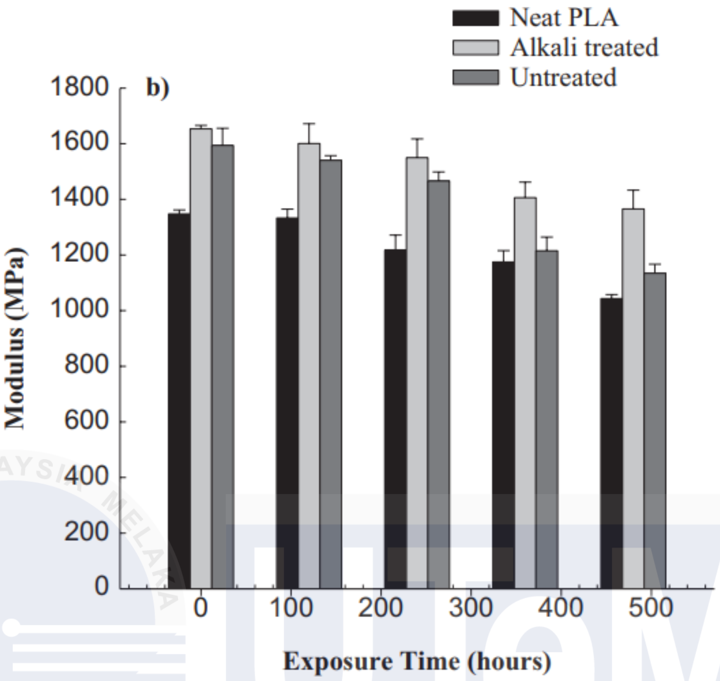


Figure 2.10: Effect of accelerated ageing on: (a) Tensile strength (b) Young's modulus of neat PLA, as well as the untreated and alkali treated biocomposites (Isadounene et al., 2018).

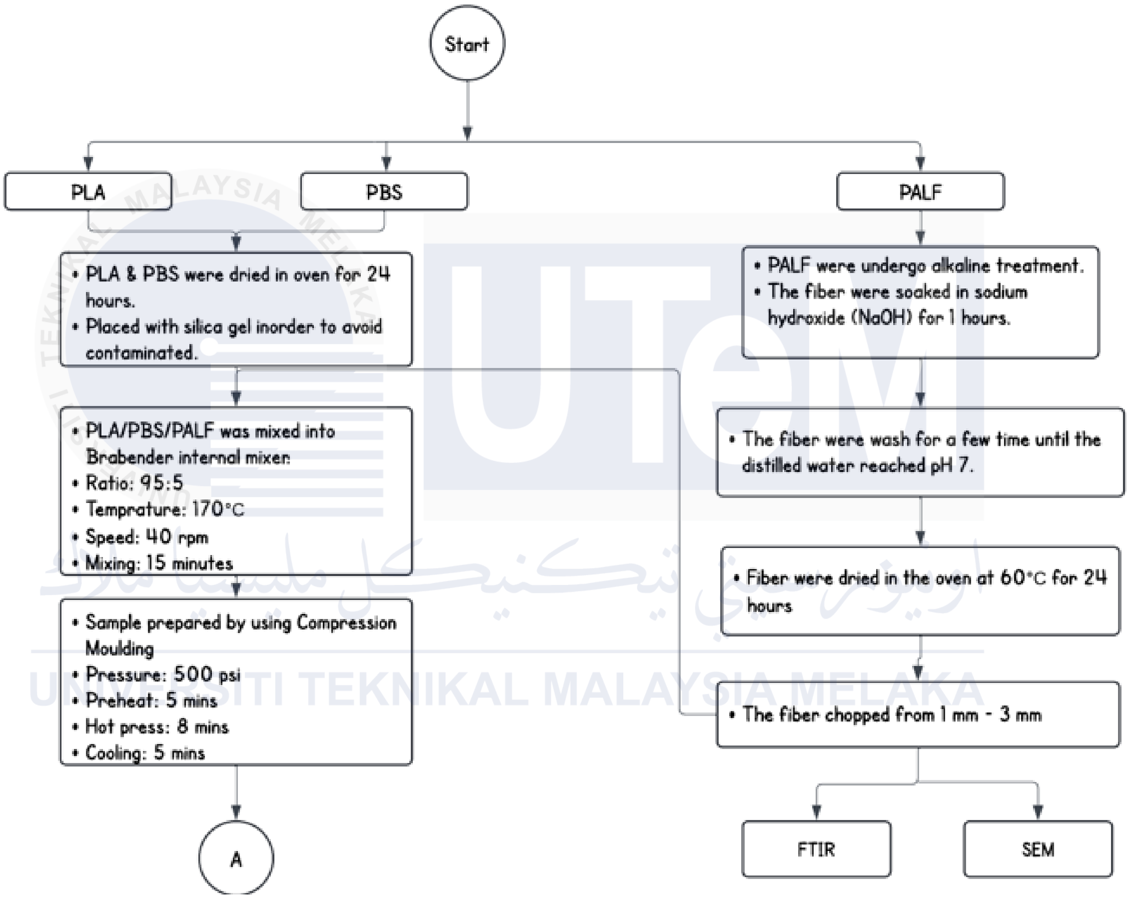
CHAPTER 3

METHODOLOGY

3.1 Overview

This chapter introduces the technique fabrication of biocomposite, the machine, and the apparatus used and the test that was used to study the effect of thermal degradation on the mechanical properties of PLA/PBS-reinforced pineapple leaf fiber. The research activities involved can be summarized into the following step.

1. Surface treatment of fiber
2. Blending of PLA/PBS
3. Fabrication of PLA/PBS reinforced with pineapple leaf fiber.
4. Thermal degradation test of biocomposite.
5. Analysis of crystallinity of biocomposite.
6. Mechanical test of the biocomposite.
7. Morphological analysis of the fracture surface.


Start
PLA
PBS
PALF
• PLA & PBS were dried in oven for 24 hours.
• Placed with silica gel inorder to avoid contaminated.
• PALF were undergo alkaline treatment.
• The fiber were soaked in sodium hydroxide (NaOH) for 1 hours.
• The fiber were wash for a few time until the distilled water reached pH 7.
• PLA/PBS/PALF was mixed into Brabender internal mixer.
• Ratio: 95:5
• Temprature: 170°C
• Speed: 40 rpm
• Mixing: 15 minutes
• Fiber were dried in the oven at 60°C for 24 hours
• Sample prepared by using Compression Moulding
• Pressure: 500 psi
• Preheat: 5 mins
• Hot press: 8 mins
• Cooling: 5 mins
• The fiber chopped from 1 mm - 3 mm
A
FTIR
SEM

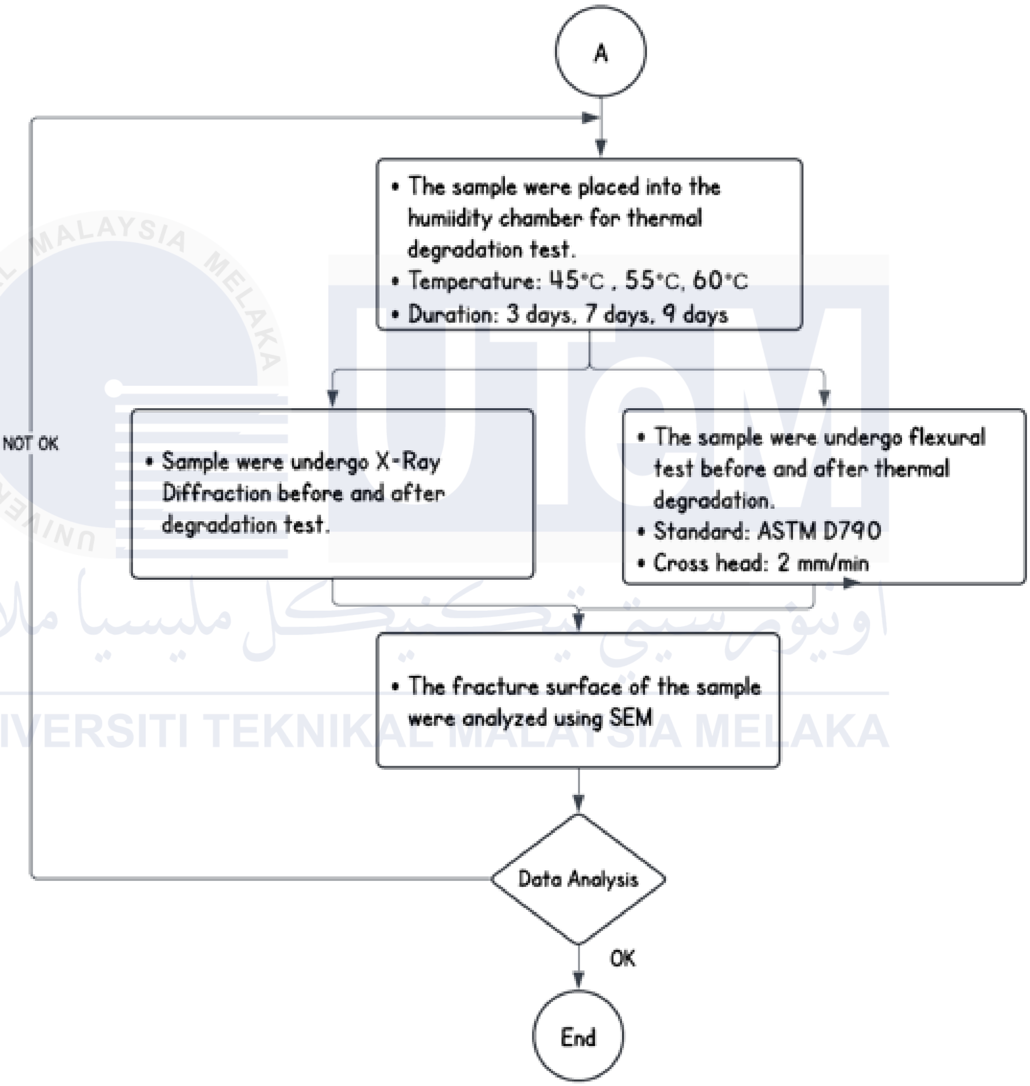


Figure 3.1: Flow chart containing a summary
the methodology of this study.

3.2 Materials

Materials used for this research are Polylactic acid (PLA) 6100 D, Polybutylene succinate (PBS), Sodium Hydroxide (NaOH), Dicumyl Peroxide, Maleic Anhydride, and Pineapple Leaf Fiber (PALF).

3.2.1 PLA 6100D

Polylactic acid was used as the matrix for the PLA used in this research was NatureWorks Ingeo™ Biopolymer6100D. The physical state of this PLA is pallets.

Table 3.1: Properties of PLA 6100D (NaturteWorks).

Physical Properties	Ingeo 6100D
Melt Density (g/cm^3 at 230°C)	1.08
Glass Transition Temperature (°C)	55-60
Crystalline Melt Temperature (°C)	165-180

3.2.2 PBS

Polybutylene succinate (PBS) has been available commercially since 1993 under the trade name of Bionolle™. PBS is known as the 1000 series.

Table 3.2: Properties of Polybutylene Succinate (PBS)
(Bionell Group,2013).

Physical Properties	Bionolle™ 1000 series
Density (g/cm^3)	1.26
Glass Transition Temperature (°C)	-32
Melting Point (°C)	114

3.2.3 Pineapple Leaf Fiber (PALF)

Pineapple Leaf Fiber is a natural fiber. PALF are in the form of two-ply yarn (640.000g/km) and is purchased from Mechasolve Sdn. Bhd, Malaysia.

3.2.4 Sodium Hydroxide (NaOH)

Sodium Hydroxide was used in the study is Sodium hydroxide pellets for analysis EMSURE®. The molecular weight is 40 g/mol. The physical state of this NaOH is solid. NaOH pellets come in white, and the melting point is 318 °C.

3.2.5 Dicumyl Peroxide

Dicumyl Peroxide used in this research was supplied by Sigma-Aldrich (M) Sdn Bhd. Dicumyl Peroxide has a molecular weight of 270.37 g/mol. This chemical is in the form of crystalline and in beige color. The melting point is between 39 °C – 41 °C.

3.2.6 Maleic Anhydride

Maleic Anhydride used in this research was supplied by Sigma-Aldrich (M) Sdn Bhd. Maleic Anhydride has a molecular weight of 98.06 g/mol. This chemical is white in color. The melting pointis between 53°C - 58°C.

3.3 Methodology

There are a few procedures to investigate the thermal degradation of biocomposite. The process starts with the surface treatment of pineapple leaf fiber followed by the fabrication process of biocomposite using and Brabender Internal Mixer and thermal degradation test of the sample. Before and after completing the test, the sample will undergo crystallinity and flexural tests.

3.3.1 Fiber Surface Treatment (Alkaline Treatment)

For one hour at room temperature, the PALF was immersed in a water bath solution containing 5% NaOH with weight. The 5g NaOH was diluted in 95 ml distilled water. The untreated pineapple PALF was immersed in NaOH solution for 1 hour at room temperature. The fibers were rinsed until the NaOH solution reached pH 7. The pH value of 7 indicated that the alkaline solution was fully removed. After being treated, the PALF were dried in a drying oven (Figure 3.2) overnight at 60°C for 24 hours to make sure they were fully dried. The treated PALF was placed in a sealed bag with silica gel to avoid further moisture absorption.

3.3.2 Length of fiber

PALF was chopped into small sizes before undergoing the mixing process with PLA/PBS. The size of the chopped fiber is 1 mm between 3 mm. The size of the fiber was proved using Scanning Electron Microscopy and the diameter was calculated using ImageJ software. The details of the measurement are in the appendix.

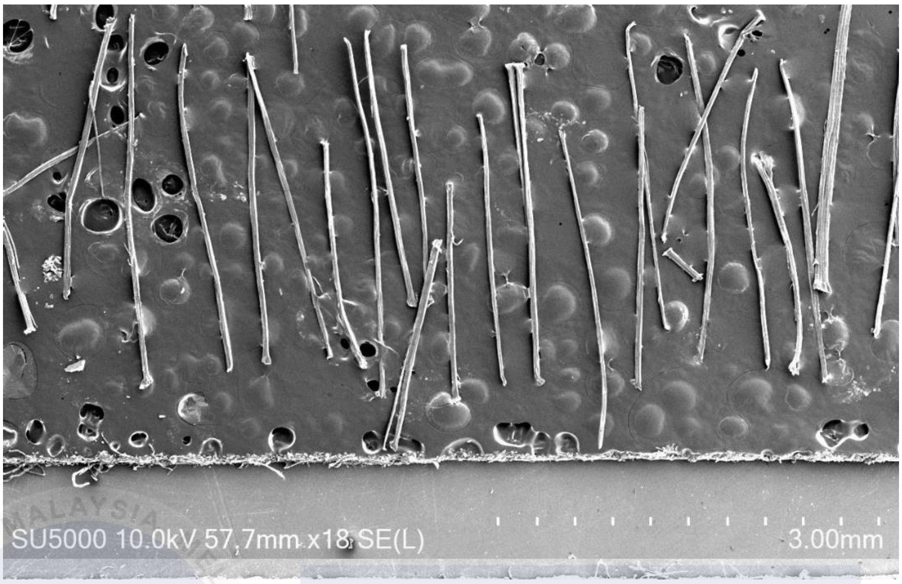


Figure 3.2: Length of chopped PALF.

3.3.3 Blending of PLA/PBS/PALF in Internal Mixture

PLA/PBS reinforced with PALF were produced by melt-mixing treated PALF and PLA/PBS matrix into Brabender Internal Mixture. Before the process, the PLA and PBS were dried for 12 hours at 60°C to minimize the moisture content. 0.1 phr Dicumyl Peroxide and 3 phr Maleic Anhydride were added into the mixer. The machine can only mix 46g per batch. The same steps were repeated to produce 270 g of biocomposite. The raw material was fed into the internal mixture for 15 minutes under a temperature of 170°C and a rotor speed of 40 rpm.

3.3.4 Fabrication of PLA/PBS/PALF in Hot Press Machine

The mixed raw material was crushed into small particles before undergoing compression molding using a stainless-steel mold platen with ASTM D790 standard size. Figures 3.3 and 3.4 show the size of each sample. The crushed material was preheated for 5 minutes at a temperature of 170°C and pressure of 200 psi hot pressed for 8 minutes and cooled down for 5 minutes.

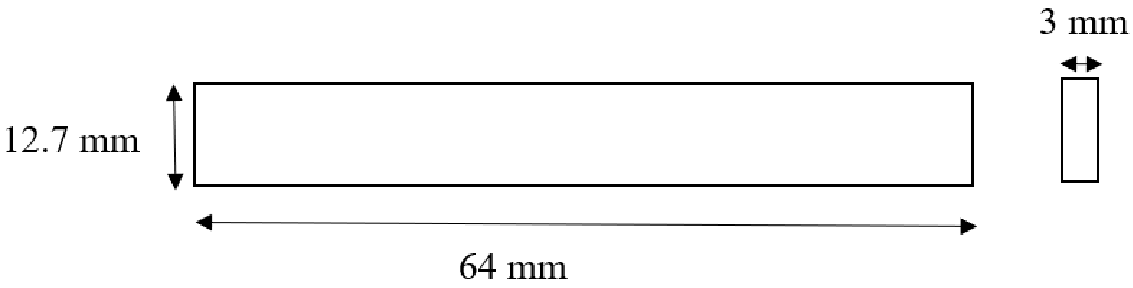


Figure 3.3: Schematic diagram of specimen for flexural test according to ASTM D790.

Figure 3.4: Sample after compression moulding.

3.4 Testing and Analysis of PLA/PBS/PALF Composite

The fiber was tested using FTIR. After that, the sample underwent X-ray diffraction to check the crystallinity of biocomposite before undergoing the degradation process. After that, the sample proceeded with a thermal degradation test. The mechanical test was conducted before the thermal degradation test and after the sample completed the degradation test. The mechanical test aims to determine the strength and stiffness of the sample before and after degradation. Lastly, the sample underwent SEM analysis to check the fracture surface.

3.4.1 FTIR Analysis after Alkaline Treatment

The alterations in functional groups of untreated and NaOH-treated fibers were examined using a Fourier-transform infrared spectroscopy machine (Model Jasco Ft/Ir-6100). The samples were examined using transmittance mode with a 2 cm^{-1} wavelength resolution, within the 500-4000 cm^{-1} range.

3.4.2 Thermal Degradation of Biocomposite

The experiment for the study of thermal degradation of PALF-reinforced PLA/PBS composites was conducted in the Mermert Humidity Chamber. All the samples were tested in the humidity chamber at temperatures of 45°C, 55°C, and 60°C for a duration of 3 days, 7 days, and 9 days. After the degradation test, the degraded samples were subjected to a flexural testing response to thermal degradation.

3.4.3 X-Ray Diffraction (XRD)

The crystallinity of the composites was analyzed before and after thermal degradation using Rigaku X-ray Diffractometer (XRD). The sample size is 2 x 2 cm. The sample was placed into sample XRD apparatus and exposed to X-rays. The diffraction angle was between 3°-90°

and with an increment of 0.01°. The percentage of crystallinity was calculated based on equation 3.

$$\% \, crystallinity = \frac{Area\ under\ all\ crystalline\ peaks}{Area\ under\ all\ crystalline\ and\ amorphous\ peaks} \times 100\% \quad \text{equation 3}$$

3.4.4 Flexural Test (ASTM D790)

A three-point bending test was performed on the specimen of the control sample basedon ASTM D790 after the thermal test by using the Shimadzu Universal Testing Machine. This testing was conducted to determine the flexural strength and Young's modulus of the specimens after the degradation period.

The sample was subjected to bending force acting perpendicular to the test piece. The specimen was simply supported in between the two supports and a 20 kN load was applied on the specimen until it started to deflect and rupture. The test was conducted with a crosshead speed of 2 mm/min. The specimens were placed on the two supported stages and load was applied with a support span of 48 mm. Shimadzu Universal Testing Machine (UTM) was used to conduct the flexural testing. The test was repeated with a minimum number of five times. The flexural was conducted in a standard laboratory atmosphere of 24°C ± 2°C.

3.4.1 Morphological Analysis

The effect of degradation on the mechanical properties of the PALF-reinforced PLA/PBS composite was studied by examining the fracture surfaces of the flexural-tested composite specimens by scanning electron microscope (Model Evo), Hitachi.

The specimen size and texture were not suitable for high-quality SEM imaging and therefore it was necessary to prepare them. The composite specimens were cut roughly to the appropriate size for SEM imaging. Double-sided carbon tape was used to attach them to the stubs of the scanning electron microscope (SEM). The samples were sputtered-coated with a layer of gold to improve surface conductivity and minimize charge effects during scanning electron microscopy imaging.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Characterization of PALF

Treated and untreated pineapple leaf fiber undergo FTIR analysis and Surface Morphology analysis.

4.1.1 Physical Properties of PALF

The surface of untreated and treated pineapple leaf fiber was analyzed using Scanning electron microscopy. Figure 4.1 (a)-(b) shows the surface morphology of PALF before and after alkaline treatment. Based on Figure 4.1 shows that the surface of treated PALF is roughercompared to untreated PALF. A previous study reported that the alkali-treated PALF has a higher degree of roughness due to the partial removal of hemicellulose and binder lignin (Mittal et al., 2018).

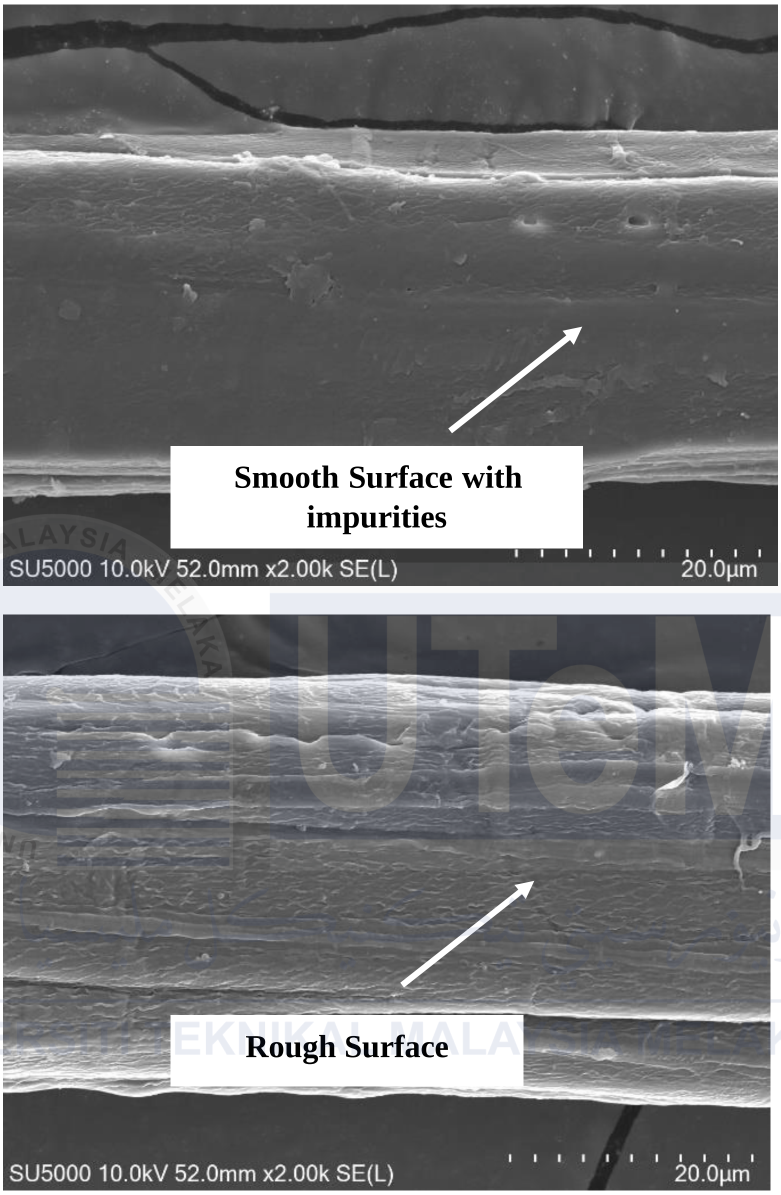


Figure 4.1: a) UPALF at magnification x2000

b) PALFNA at magnification x2000.

4.1.2 FTIR analysis

The FTIR spectra in Figure 4.2 shows reveals several changes that occurred in bothuntreated and treated PALF. The peak values for untreated pineapple leaf fiber are 3339 cm^{-1}, 2896 cm^{-1}, 1734 cm^{-1}, 1421 cm$^{-1,}$ and 1239 cm^{-1}. As observed the infrared absorption stretching between 3700 cm^{-1} – 3000 cm^{-1} corresponds to the hydroxyl group (-OH) of carboxylic acid and alcohol on lignin (S. Gnanasekaran et al., 2021). The peak observed between 2900 cm^{-1} and 2800 cm^{-1} is the result of the C-H stretch from the alkyl group and the peaks between 1734.81 cm^{-1} correspond to the C=O stretch. The peak was observed at 142 cm^{-1} and 1239 cm^{-1} result of C-O stretching was associated with the lignin and hemicellulose structure. There are obvious peaks observed at 1029 cm ^{1}. The peak corresponds to the C-O-C stretching.

The absence of individual peaks at 1734 cm^{-1} from FTIR spectra indicates that NaOH completes the removal of hemicellulose. The peak intensity corresponded to peaks around 1734 cm^{-1} corresponding to the non-cellulosic components, such as pectin, waxes, and lignin, which shows that alkaline treatment effectively removes unwanted content from the fiber surfaces.

The intensity of the OH peak at 3342.42 cm^{-1} was found to have slightly decreased compared to untreated pineapple leaf fiber, which is from 91.052 to 83.718, as shown in Figure 4.2 because of the interaction of alkali with PALF fibers which in turn leads to the modification of fiber composition. According to a previous study, alkaline treatment was highly effective in removing hemicellulose from the fiber (Senthilkumar et al., 2019).

Table 4.1: The wave numbers of peaks used for FTIR analysis and corresponding functional group and vibrational type.

Wavenumber	Functional Group	Vibrational Type
3400- 3200	O-H	O-H stretching
2900-2800	-CH_2-	C-H stretching
1740-1720	R-CHO	C=O stretching
1260-1180	C-O	C-O stretching
1029	C-O-C	C–O–C stretching

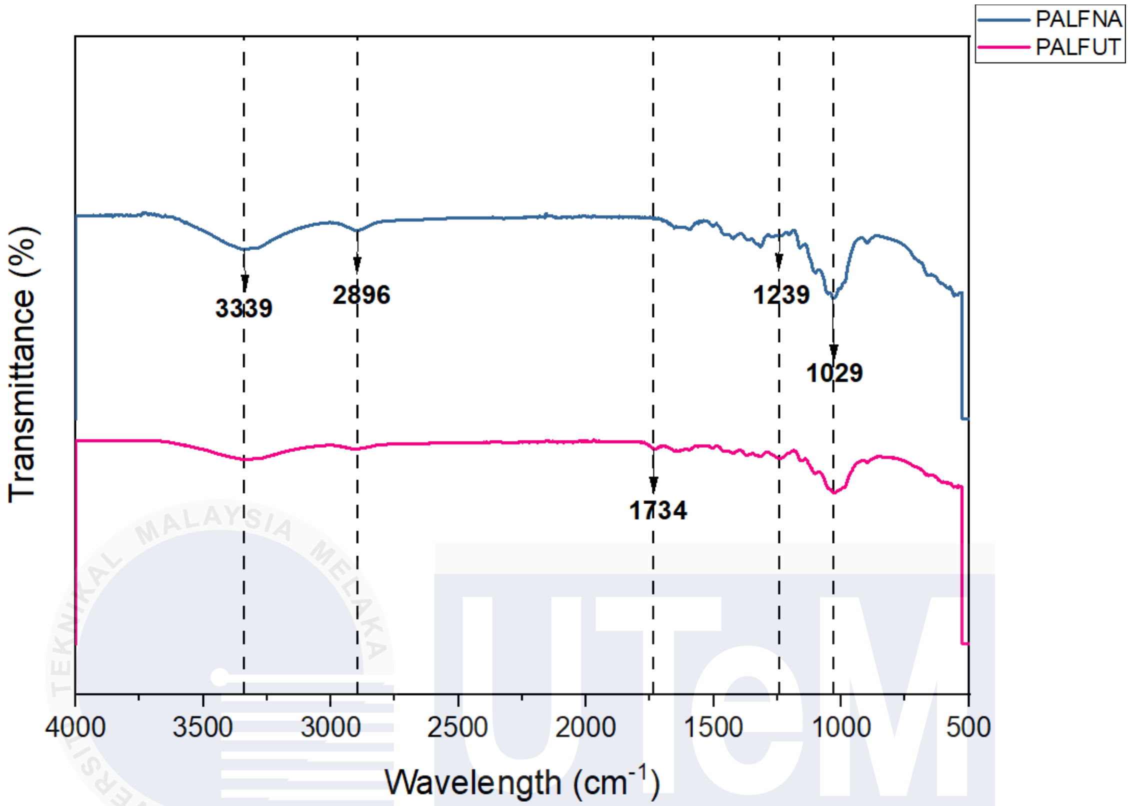


Figure 4.2: FTIR spectroscopy for PALFUT and PALFNA

4.1 Effect of Thermal Degradation on Biocomposite

The approach of this study is the effect of thermal degradation of PLA/PBS reinforced with PALF. The effect of thermal degradation was analyzed by looking at the physical properties, crystallinity, and mechanical properties of biocomposite.

4.1.2 Physical properties

Physical Properties of the biocomposite were analyzed by calculating the mass loss of the sample. The mass loss of sample PLA/PBS/PALF was determined by taking the average of three readings using a weighing scale. The sample PLA/PBS/PALF was weighed before and after thermal degradation. The weight loss of the sample increased in an increase in temperature and duration. The weight loss increased from 0.06% in 3 days, 0.31% in 7 days, and 0.36% in 9 days when exposed to a temperature of 45°C. When the sample has been exposed to a temperature of 55°C the weight loss also increased from 0.08% in 3 days, 0.19% in 7 days, and 0.74% in 9 days. This degradation in polymeric composites involves solid-gas reactions and is associated with chain division. It is also found that the weight loss and temperature difference have a linear relationship and the maximum weight loss occurs at the high temperature difference. Exposure to moisture in humid conditions reduces interfacial stress transmissibility, resulting in stiffness loss due to adhesion loss between fibers and the polymer (Haddar et al.,2020).

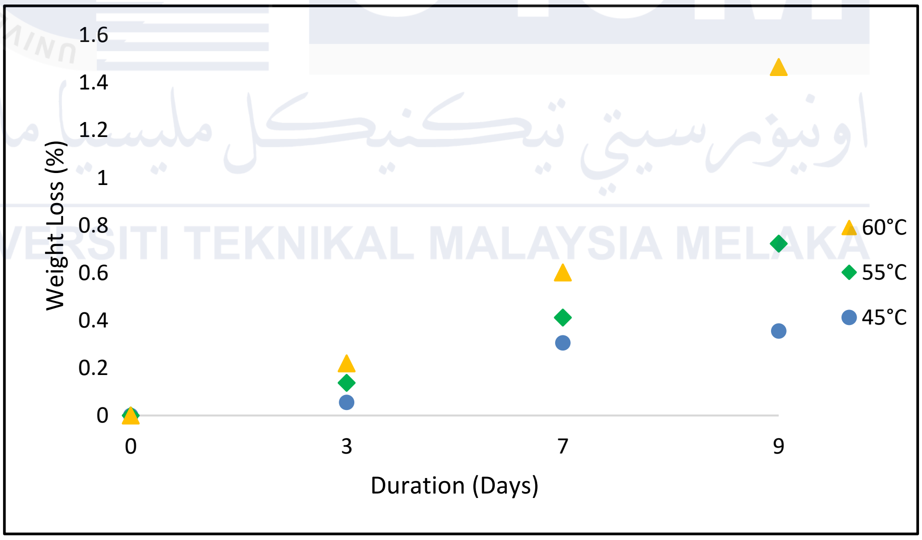


Figure 4.3: Weight loss of biocomposite sample during thermal test at 45°c and 60°c (3,7,9 days).

4.2.2 Crystallinity

Based on Figure 4.4, the intensity of the crystalline region increased with increasing time and temperature. This is because the increase has been attributed to a combination of the polymer's plasticization and the amorphous portions degrading more quickly than the crystalline sections. Increasing temperature leads to an increase in the crystallinity of biocomposite. Crystallinity can be related to the weight loss of the sample. Figure 4.3 shows the mass loss increases when increased in temperature and time. During thermal degradation, amorphous regions in polymer fibers tend to degrade more easily than crystalline regions, leading to a higher mass loss in the amorphous parts. This results in an increase in the relative crystallinity of the remaining material.

Figure 4.4 reveals the XRD patterns of PLA/PBS/PALF specimens before and after degradation at different temperatures (45°C and 60°C) and time (3,7,9 days). There are no sharp diffraction peaks detected in the PLA/PBS/PALF before degradation and during temperature 45°C at 3,7 and 9 days. During this time the sample was in amorphous condition. However, after a degradation at a temperature of 55°C and 60°C (3,7,9 days) one sharp peak was observed at $2\theta=$ 16.2° represents PLA. A sharp peak indicates the sample was crystalline while the narrow peak indicates the amorphous region. This showed that crystallinity increased when temperature and duration increased. The percentage of sharp crystalline peak increase was calculated in Table 4.2. Based on Table 4.2 crystallinity increases when the duration increases. The percentage of crystallinity of PLA increased by 7% from day 3 to day 9 for a temperature of 55°C and increased to 47.03% when the biocomposite was exposed to a temperature of 60°C. Based on the result the thermal degradation increased the crystallinity of PLA in biocomposite. The crystallinity is improved by longer times and higher temperatures, which enable more extensive chain alignment. This is because the polymer chains can move and align more efficiently due to the increased thermal energy, which raises the degree of crystallinity (Nagarajan et al.,2015). The percentage of sharp crystalline peak increase was calculated in Table 4.2. Based on the table crystallinity increases when the duration increases. An increase in crystallinity causes the mechanical properties of the material to decrease the effect of thermal degradation. The result can be proved based on Figure 4.5.

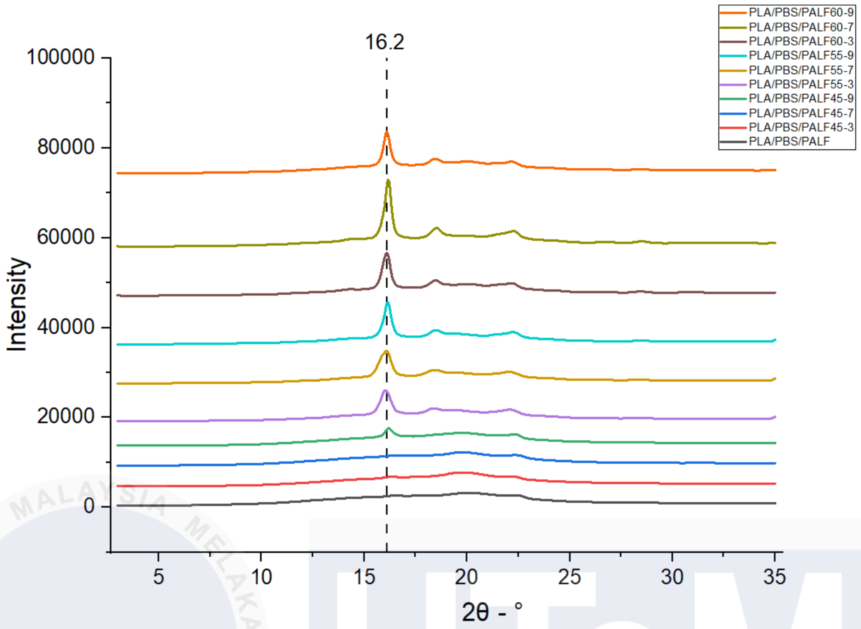


Figure 4.4: XRD spectrum for the control sample and after thermal degradation at temperatures 45°C,55°C, and 60°C for 3,7, and 9 days.

Table 4.2: Percentage crystallinity of PLA after thermal degradation.

Temperature (°C)	Duration (Days)	Crystallinity (%)
45	3	0
	7	0
	9	0
55	3	17.00
	7	17.92
	9	18.31
60	3	17.16
	7	19.25
	9	25.23

4.2.3 Mechanical Properties

The comparison of the effect of thermal degradation on the flexural properties of PLA/PBS reinforced with pineapple leaf fiber has been carried out using 45°C, 55°C, and 60° C for 3,7 and 9 days. The flexural strength and flexural modulus are shown in Figure 4.5. The initial flexural strength and modulus PLA/PBS/PALF is 20.08 MPa and 1.57 GPa. In Figure 4.5(a) and (b). This result was supported in Figure 4.6 (a) which shows less fiber pull-out and fiber void. After the thermal test at 45°C the flexural strength reduced from 39% in 3 days to 54 % in 9 days of exposure to that temperature from the control sample. However, the sample that tested for 7 days shows a decrease compared to days 9. Based on the previous study, the flexural strength should be decreased with the increased with time. This is probably due to a mistake during the preparation of the sample. The sample for 7 days contained a bubble that caused it easily to bend due to the bubble. Bubbles lower the material's overall strength because act as concentrators. Present void has the potential to cause early failure by causing cracks to form when under load. The result was supported in Figure 4.6 (c) where the fracture sample contained more void compared to sample Figure 4.6 (d) which undergoes a thermal test for 9 days. The measurement of void was calculated in the appendix. This slight decrease in flexural strength was due to the degradation rate of the matrix and fiber-matrix interface increase as the degradation increased (Felfel et al., 2015). For the flexural modulus shown in Figure 4.5 (b), the flexural modulus increased at a temperature of 45°C. The flexural modulus increased by 7.67% in 3 days to 24.11% in 9 days. The increased flexural modulus causes the material to be stiffer. This result can be shown in Figure 4.6 (b) - (d) the rough surface of the fracture surface and less void pull-out.

Figures 4.5 (a) and (b) showed that flexural strength and modulus decreased from low to high temperature and duration. Based on Figure 4.5 (a) the flexural strength has been reduced by 70% from its control sample when exposed to a temperature of 55°C in 3 days to 87% in 9 days while the flexural modulus increased in days 3 compared to the control sample but decrease when exposed to 7 and 9 days. This showed that the sample has high stiffness compared to the sample exposed in 7 days and 9 days. This result can be supported by Figure 4.6 (e)-(g) where less fiber was pulled out in 3-day exposed periods compared to days 7 and 9 days. After being exposed to 7 and 9 days both flexural properties decrease to 48.33%. This result showed that both flexural properties were decreased when exposed to temperature and duration. The reinforcing fibers can be pulled out of the matrix more easily because of the polymer matrix softening and losing strength as the temperature rises. This result has been proved by fracture surface Figure 4.6 (e) – (g) increase in fiber pull out and microcrack.

Based on Figure 4.5 (a) and (b) the sample was exposed to the 60°C temperature and showed a decrease in both flexural strength and modulus. The flexural strength reduced from 74.95 % in 3 days to 87.05% in 9 days test duration. The flexural modulus was also reduced by 10.19% in 3 days at 66.24% from the control sample. The decrease in flexural strength and modulus showed that the sample undergoing thermal degradation. Based on the previous study, it was discovered that the flexural strength and stiffness of the bagasse fiber reinforcement polymer bars reduced as temperature increased. This decline became more noticeable when the temperature approached the Tg of the bars. Tg of biocomposites is about 49.01°C. At this moment, the polymer transitions from a glassy to a rubbery material, losing its capacity to hold the fibers together and transfer stresses between them, resulting in a substantial drop in flexural strength and stiffness (Aween et., al 2023). This can be proved in Figure 4.6 (h)-(j) shows the fracture surface was smooth, with microcracks on the surface when the time increased. In addition, the flexural fracture surfaces showed fiber pull-out due to the fracture of PALF fiber during flexural loading.

The flexural strength of EPS polystyrene ranged between 0.0750-3.00 MPA and the flexural modulus was 0.00628-0.0342 GPa (MatWeb, 2024). The range of flexural strength based on the study was from 0.393 - 8.039 MPa and flexural modulus between 0.122 - 0.48 GPa. This showed that biocomposite has high flexural modulus and strength compared to EPS polystyrene material, making that sample suitable for packaging material in Abu Dhabi.

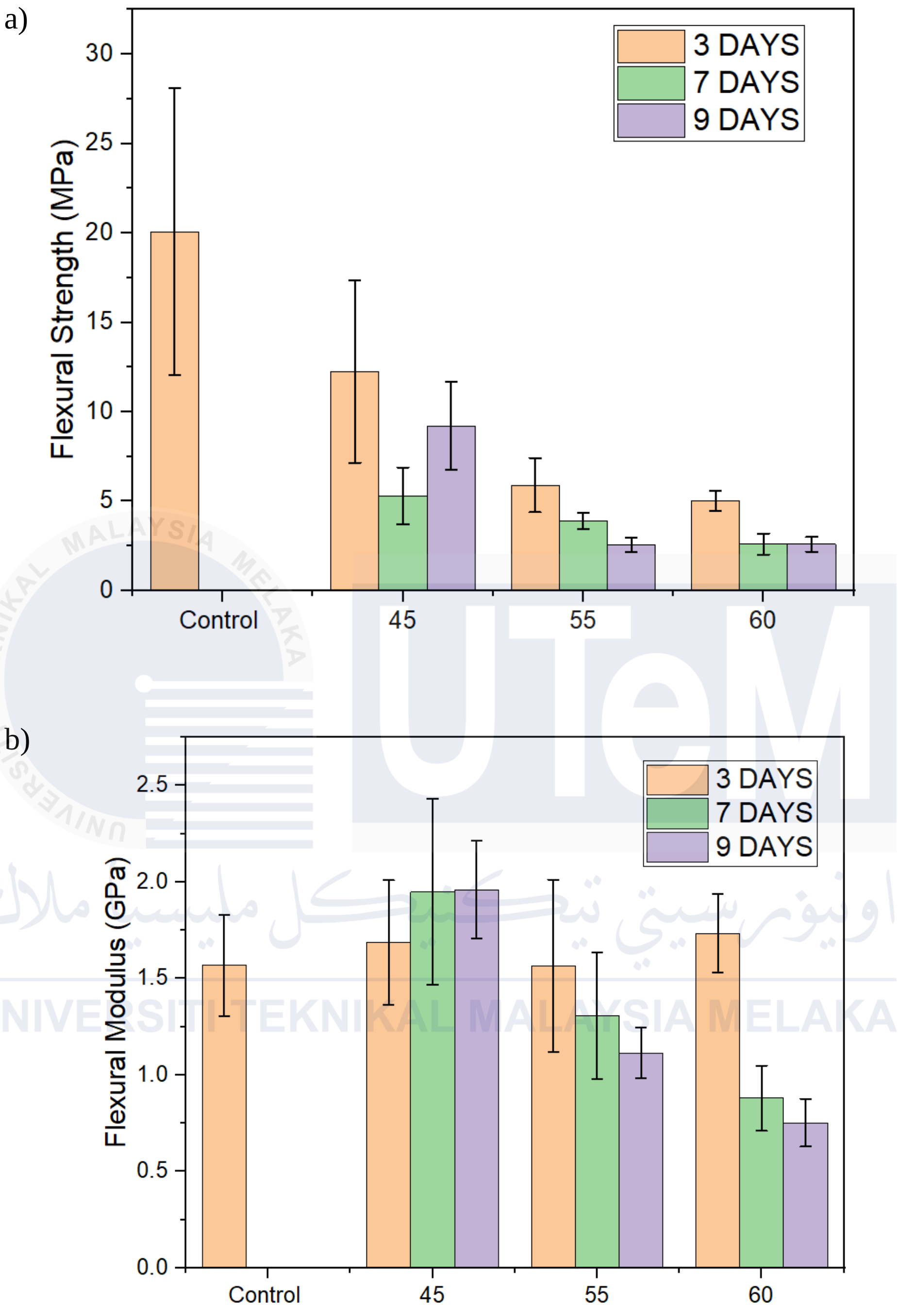
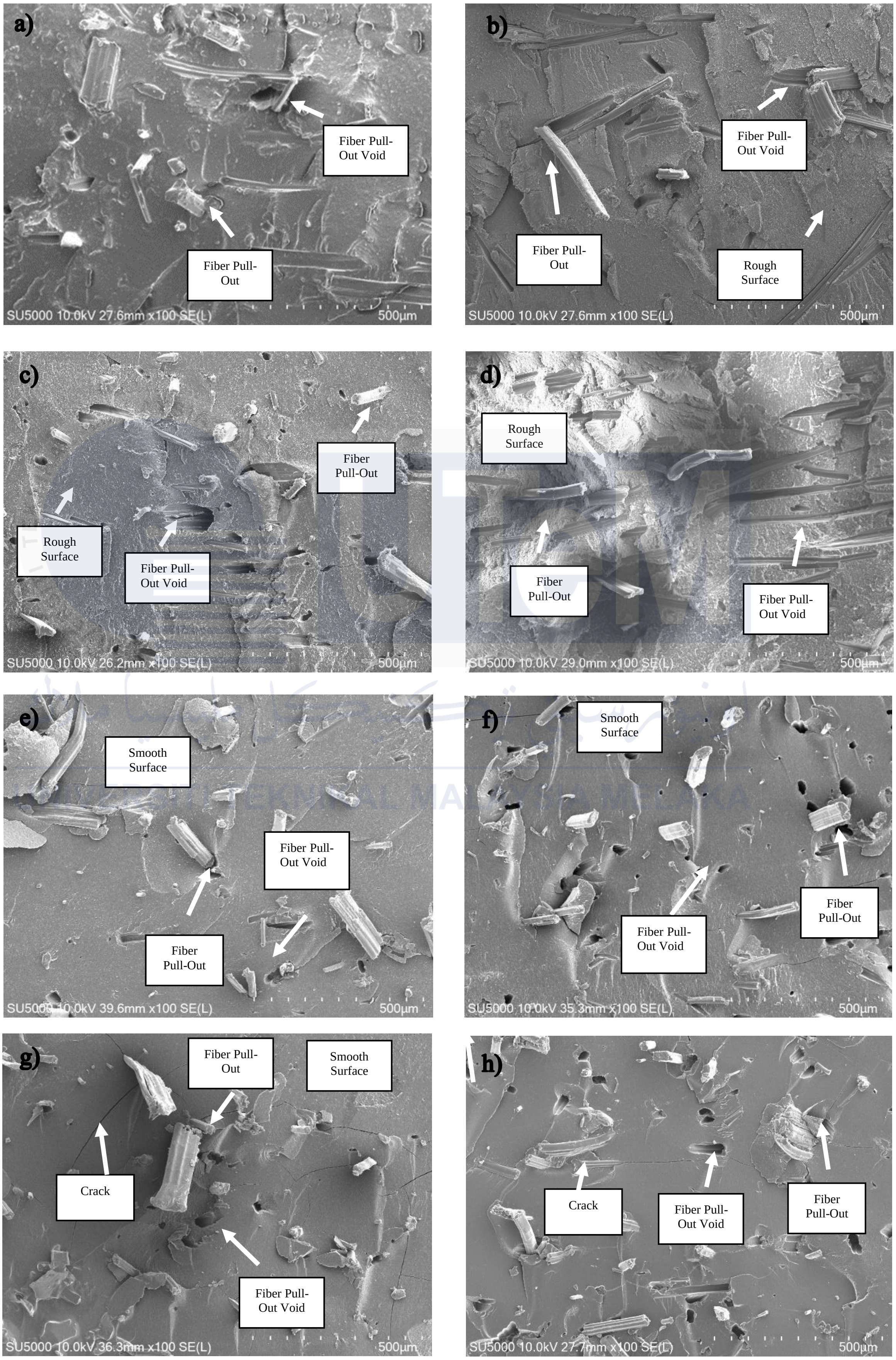


Figure 4.5: a) Flexural strength b) Flexural modulus for control sample at 45°C, 55°C, and 60°C for 3, 7, and 9 days.


a)
Fiber Pull-Out Void
Fiber Pull-Out
SU5000 10.0kV 27.6mm x100 SE(L)
500µm
b)
Fiber Pull-Out Void
Fiber Pull-Out
Rough Surface
SU5000 10.0kV 27.6mm x100 SE(L)
500µm
c)
Fiber Pull-Out
Rough Surface
Fiber Pull-Out Void
SU5000 10.0kV 26.2mm x100 SE(L)
500µm
d)
Rough Surface
Fiber Pull-Out
Fiber Pull-Out Void
SU5000 10.0kV 29.0mm x100 SE(L)
500µm
e)
Smooth Surface
Fiber Pull-Out Void
Fiber Pull-Out
SU5000 10.0kV 39.6mm x100 SE(L)
500µm
f)
Smooth Surface
Fiber Pull-Out
Fiber Pull-Out Void
SU5000 10.0kV 35.3mm x100 SE(L)
500µm
g)
Fiber Pull-Out
Smooth Surface
Crack
Fiber Pull-Out Void
SU5000 10.0kV 36.3mm x100 SE(L)
500µm
h)
Crack
Fiber Pull-Out Void
Fiber Pull-Out
SU5000 10.0kV 27.7mm x100 SE(L)
500µm

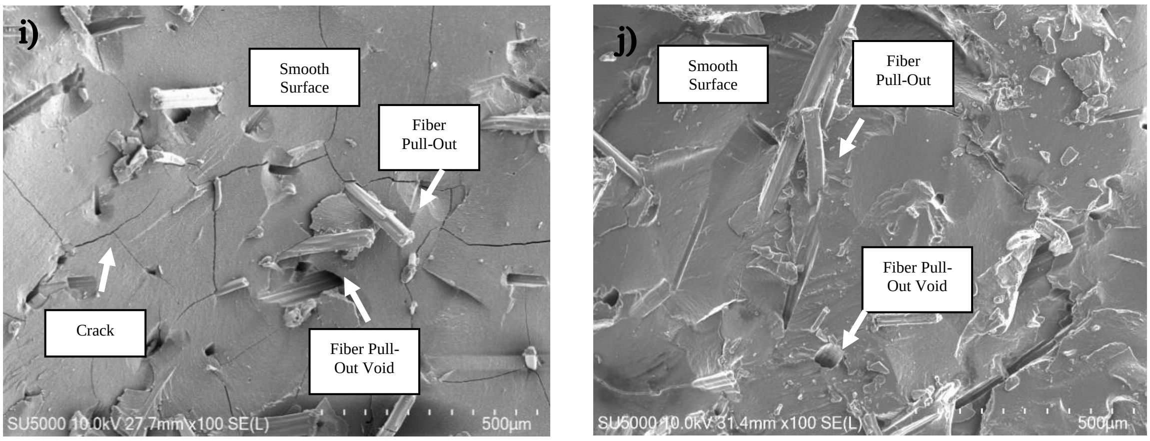


Figure 4.6: Fracture morphology of PLA/PBS/PALF composites as a result of flexural testing upon thermal degradation at a) Control Sample, (b) 45°C for 3 days, (c) 45°C for 7 days, (d) 45°C for 9 days, (e) 55°C for 3 days, (f) 55°C for 7 days, (g) 55°C for 9 days, (h) 60°C for 3 days, (i) 60°C for 7 days, (j) 60°C for 9 days.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusions

The first objective of this research is to study the effect of thermal degradation on the crystallinity of biocomposite. The data collected are analyzed and significant conclusions for this objective are as follows.

(a) The crystallinity of the biocomposite increased with the increase of temperature and duration.

(b) XRD spectrum showed the crystalline increased while the amorphous region decreased.

(c) The crystallinity increased due to degradation of the amorphous region of the biocomposites sample as temperature and duration increased. The removal of amorphous components allows better alignment of cellulose chains (Pickering et al., 2011).

The second objective of this research is to study the effect of thermal degradation on the mechanical properties of biocomposite. The significant conclusion for this objective is as follows.

(a) At a temperature of 45°C, the flexural strength was increased and decreased in flexural modulus.

(b) At a temperature of 55°C and 60°C, both flexural strength and modulus decrease due to the polymer transitions from a glassy to a rubbery material, losing its capacity to hold the fibers together and transfer stresses between them, resulting in a substantial drop in flexural strength and stiffness (Aween et al., 2023). Tg of biocomposite is 49.01°C.

(c) The fracture surface of the biocomposite sample after thermal degradation at a temperature of 45°C showed a smooth surface while at 55°C and 60°C showed a rough surface.

(d) The result of the flexural test showed that the biocomposite material that has been studied is not suitable for packaging material in Abu Dhabi. This is because flexural strength and modulus decrease when temperature and duration increase. Decrease in flexural properties making the material unsuitable for packaging material.

The flexural strength of EPS polystyrene ranged between 0.0750-3.00 MPA and the flexural modulus was 0.00628-0.0342 GPa (MatWeb, 2024). The range of flexural strength based on the study was from 0.393- 8.039 MPa and flexural modulus between 0.122- 0.48 GPa. This showed that biocomposite has high flexural modulus and strength compared to EPS polystyrene material, making that sample suitable for packaging material in Abu Dhabi.

5.2 Recommendations

After going through this research, there are few recommendations suggested so be able to call attention to the important aspects that needs to be focus on for coming research topic about gel coat in the future. The significant recommendation for this objective as follows.

(a) Exposing the biocomposite to a longer duration of the thermal degradation test such as 2 weeks or above. Packaging materials are frequently used to protect things for weeks, months, or even years. Longer testing times assist in forecasting how the material will perform throughout its whole intended duration.

(b) Analysis the sample using TGA and DSC. The thermal properties of the material can determine the glass transition temperature and melting point. To make sure that the packaging can function well.

5.3 Sustainability Element

This research was motivated by the innovation of green packaging using biocomposite material. Biocomposite material offers an eco-friendlier material compared to traditional petroleum-based composite. Biocomposite is a polymer reinforced with natural fiber. The petroleum-based packaging can affect the environment caused of chemical waste and synthetic material.

5.4 Life Long Learning Element

Based on this project, time management is very important to ensure the project can be completed within the time given. Planning is essential to project management for a reason, all of which helps a project be carried out and completed successfully. The hands-on experience and practical abilities in managing testing material and analyzing data are one of the outcomes of completing the project.

5.5 Complexity Element

Biocomposite samples were tested in the humidity chamber at different temperatures and duration which are 45°C, 55°C, and 60°C for 3,7, and 9 days. The most complex element in this study is to analyze the result and relate it with a suitable journal. The result from the flexural test was analyzed to check whether the data could be used or not. The purpose of this flexural test is to analyze the bending of the material as packaging material when subjected to temperature and duration

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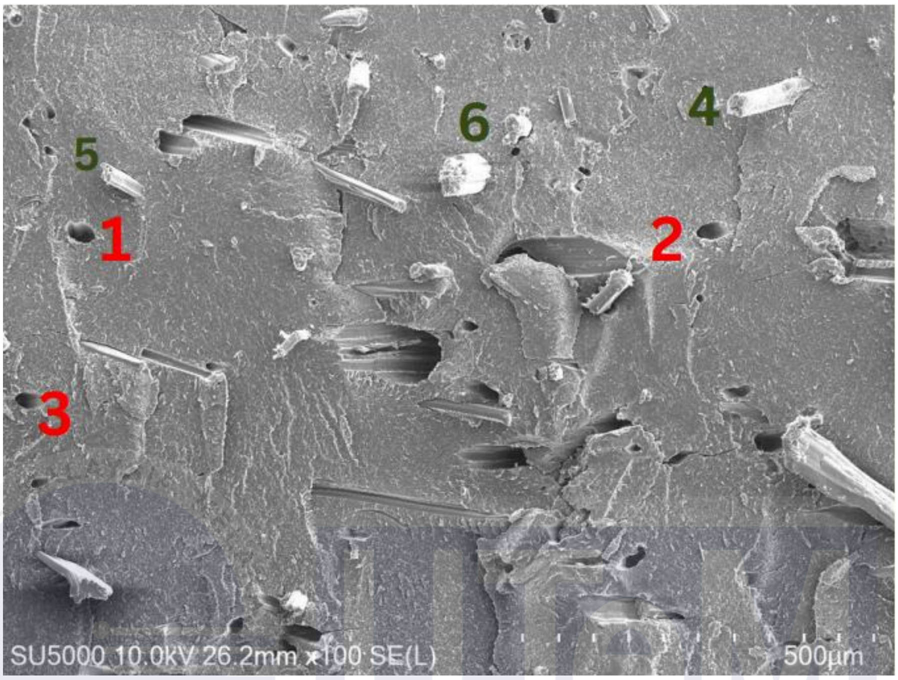
APPENDICES

Measurement of Fiber Length

PALFNA	Length
1	2.22
2	2.92
3	2.59
4	2.43
5	2.47
6	1.81
7	2.84
8	2.19
9	2.04
10	1.68
11	1.72
12	2.13
13	2.54
14	2.52
15	2.23
16	2.42
17	1.97
18	2.54
19	2.39
20	2.56
21	2.82
22	2.10
23	2.19
24	2.21
25	2.21
26	2.15
27	2.23
28	2.48
29	1.80
30	2.33
31	2.15
32	2.30
33	1.63

34	1.70
35	1.75
36	2.26
37	2.06
38	2.37
39	1.94
40	2.44
41	1.74
42	2.17
43	1.96
44	2.00
45	1.93
46	1.96
47	1.90
48	1.86
49	2.24
50	1.35
51	2.04
52	1.85
53	1.53
54	1.84
55	1.97
56	2.26
57	1.88
58	2.04
59	1.73
Average	2.13
Standard Deviation	0.39

Measurement of fiber void



Diameter of fiber pull-out void

	Area	Mean	StdDev	Min	Max	Angle	Length (μm)
1	24.094	100.703	51.452	43.458	209.833	87.955	27.577
2	27.125	96.91	42.643	71.438	236.862	118.443	26.865
3	24.219	77.039	24.688	46.901	146.25	105.945	23.885

Diameter of fiber

	Area	Mean	StdDev	Min	Max	Angle	Length (μm)
4	32.737	154.549	45.869	58.562	255	-54.039	31.378
5	21.825	190.337	42.047	100.452	250.111	-146.689	20.857
6	23.809	174.179	36.837	125.318	249.25	-135	22.537

Calculation of weight loss

Temperature 45°C

$$Weight\ loss\ for\ 3\ days = \frac{(2.410 - 2.409)}{2.409} \times 100\% = 0.06\%$$

$$Weight\ loss\ for\ 7\ days = \frac{(2.410 - 2.396)}{2.396} \times 100\% = 0.31\%$$

$$Weight\ loss\ for\ 9\ days = \frac{(2.447 - 2.438)}{2.409} \times 100\% = 0.36\%$$

Temperature 55°C

$$Weight\ loss\ for\ 3\ days = \frac{(2.428 - 2.430)}{2.430} \times 100\% = 0.08\%$$

$$Weight\ loss\ for\ 7\ days = \frac{(2.428 - 2.425)}{2.425} \times 100\% = 0.11\%$$

$$Weight\ loss\ for\ 9\ days = \frac{(2.390 - 2.381)}{2.381} \times 100\% = 0.37\%$$

Temperature 60°C

$$Weight\ loss\ for\ 3\ days = \frac{(2.445 - 2.447)}{2.447} \times 100\% = 0.08\%$$

$$Weight\ loss\ for\ 7\ days = \frac{(2.469 - 2.479)}{2.479} \times 100\% = 0.43\%$$

$$Weight\ loss\ for\ 9\ days = \frac{(2.529 - 2.510)}{2.510} \times 100\% = 0.74\%$$

Calculation of crystallinity

Temperature 55°C

$$Crystallinty\ index\ for\ 3\ days = \frac{5847.14}{34392.43} \times 100\% = 17.00\%$$

$$Crystallinty\ index\ for\ 7\ days = \frac{6328.75}{34550.61} \times 100\% = 18.31\%$$

$$Crystallinty\ index\ for\ 9\ days = \frac{6350.47}{35425.60} \times 100\% = 17.93\%$$

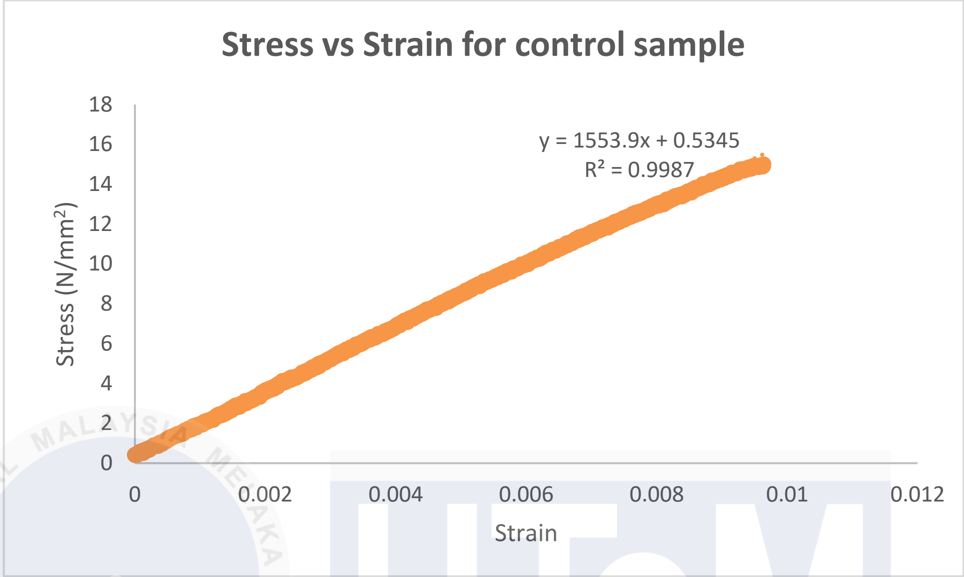
Temperature 60°C

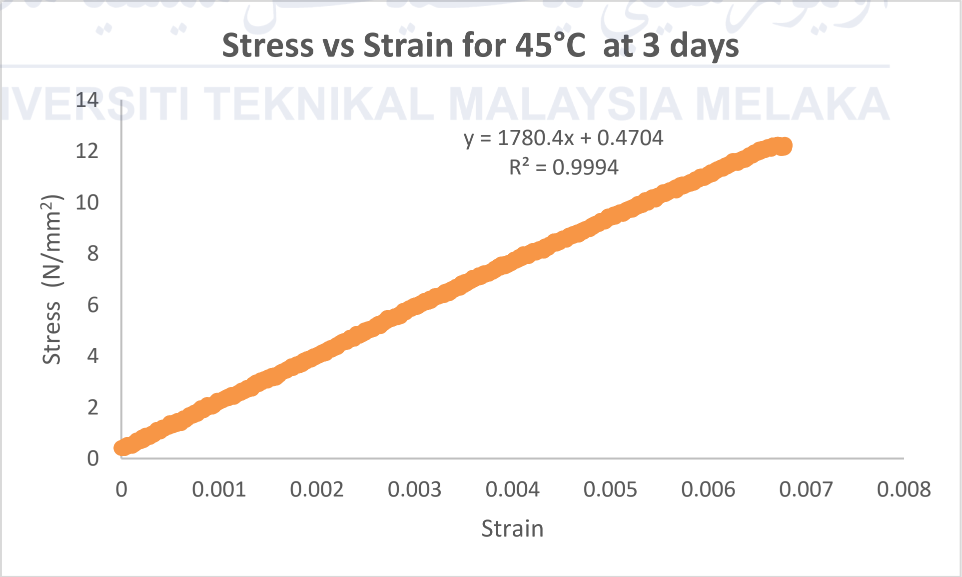
$$Crystallinty\ index\ for\ 3\ days = \frac{5960.08}{34723.32} \times 100\% = 17.16\%$$

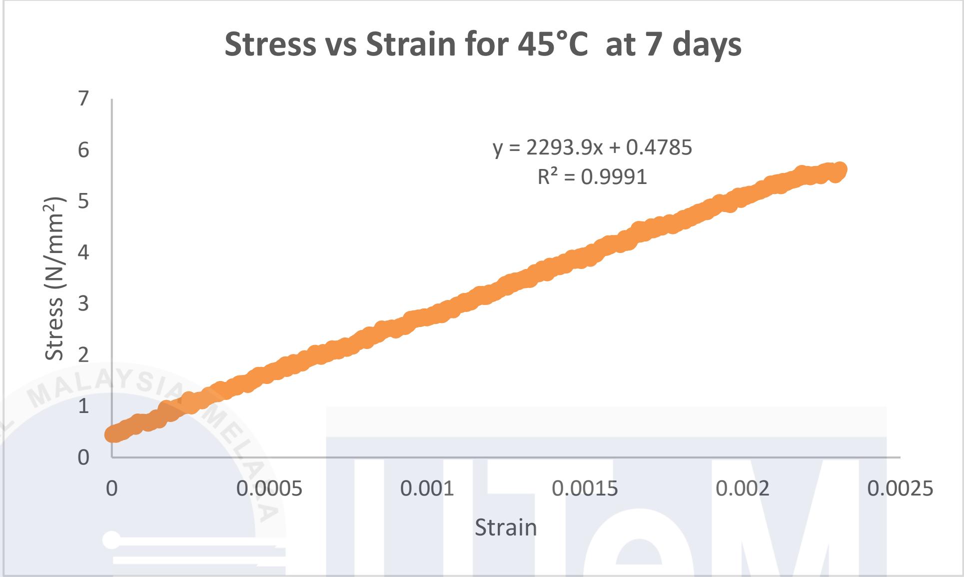
$$Crystallinty\ index\ for\ 7\ days = \frac{6974.30}{36228.19} \times 100\% = 19.25\%$$

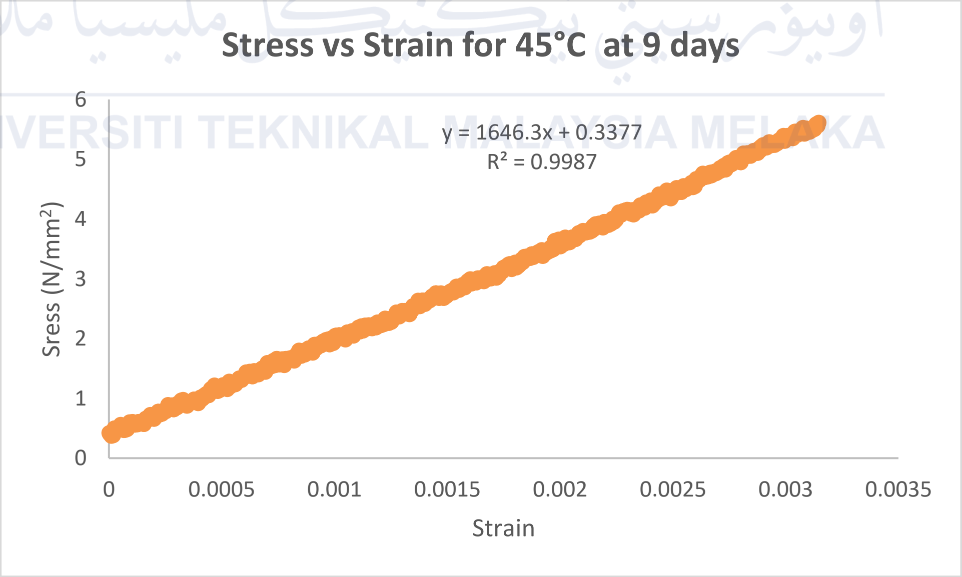
$$Crystallinty\ index\ for\ 9\ days = \frac{10542.47}{41791.24} \times 100\% = 25.23\%$$

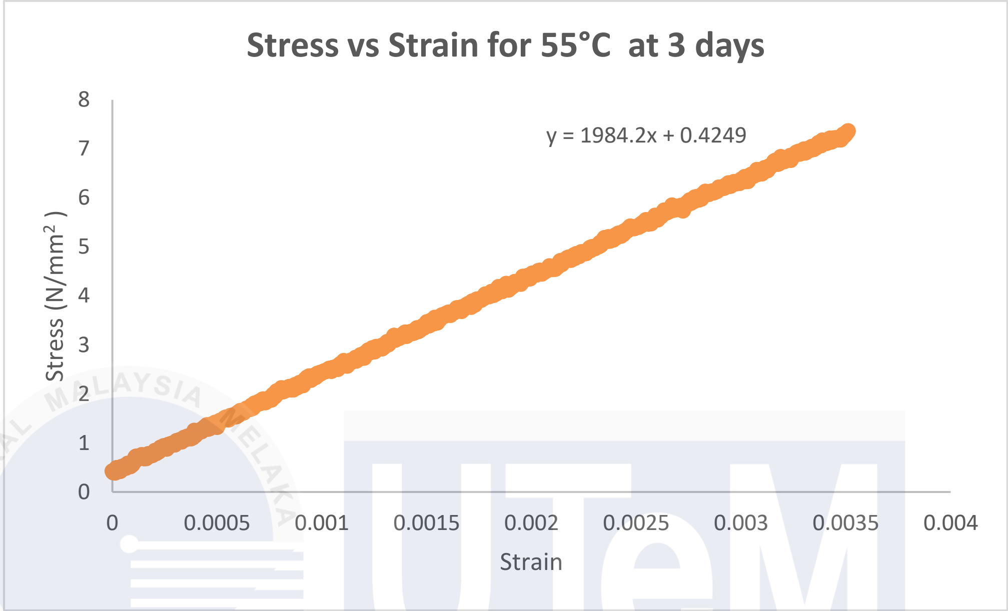
Stress vs Strain curve

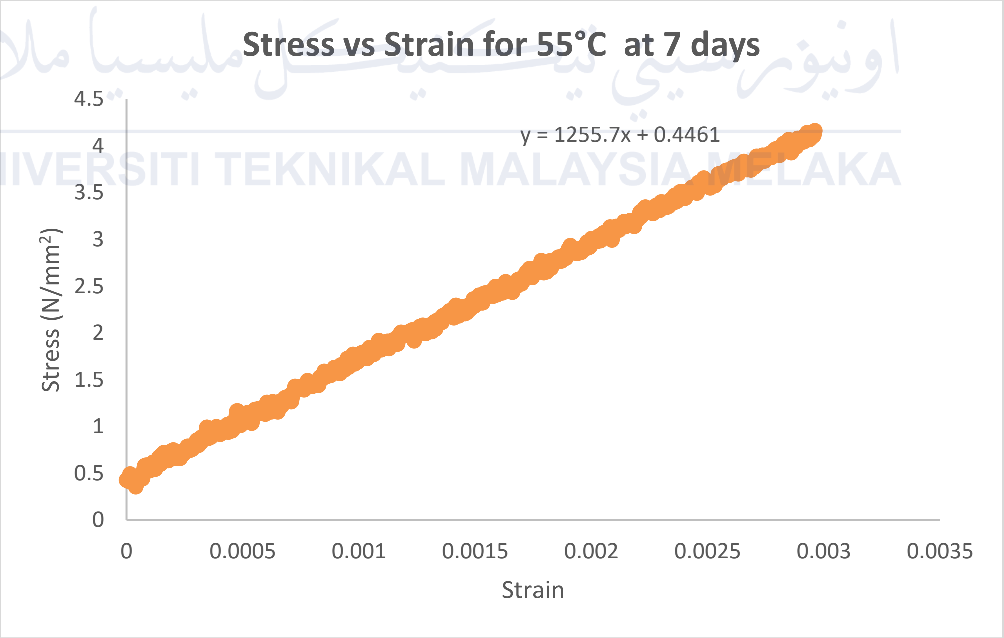


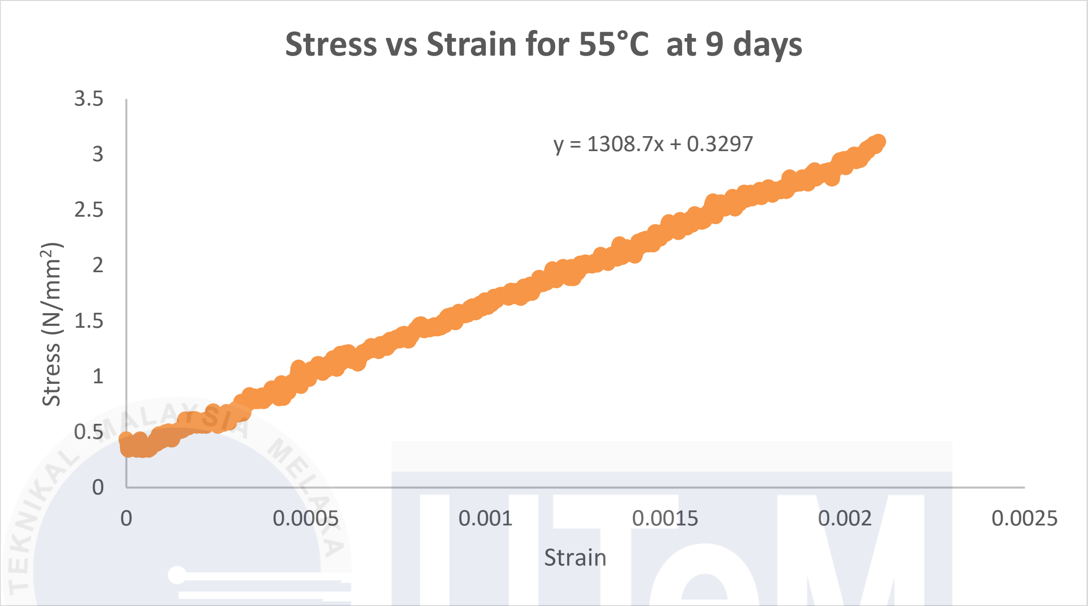


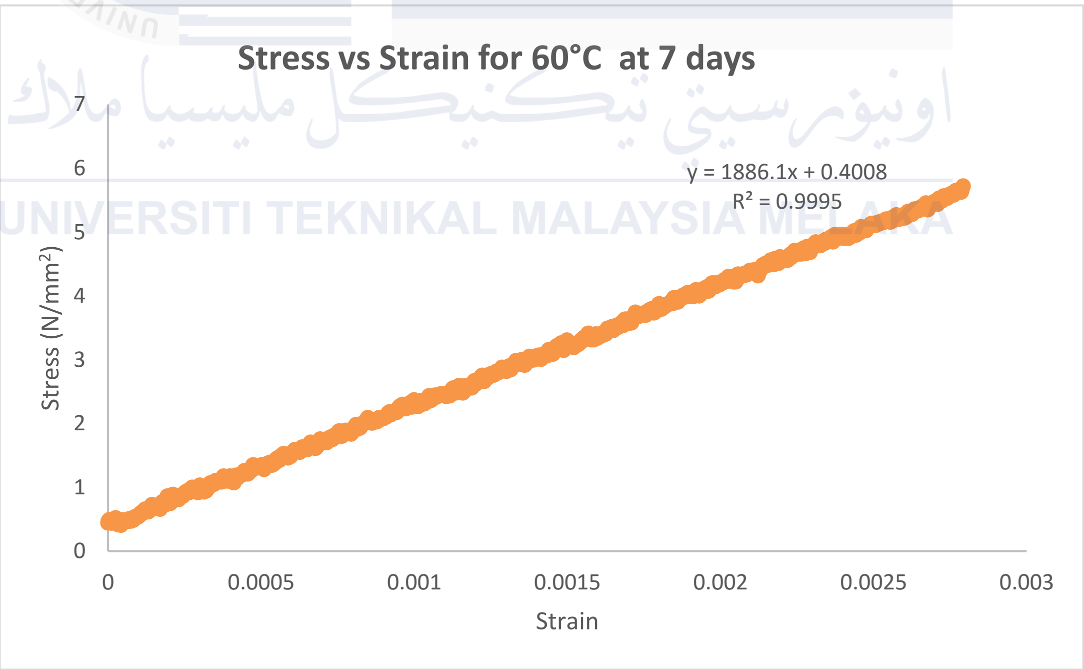

Stress vs Strain for 45°C at 7 days
y = 2293.9x + 0.4785
R² = 0.9991
Stress (N/mm²)
7
6
5
4
3
2
1
0
0
0.0005
0.001
0.0015
0.002
0.0025
Strain

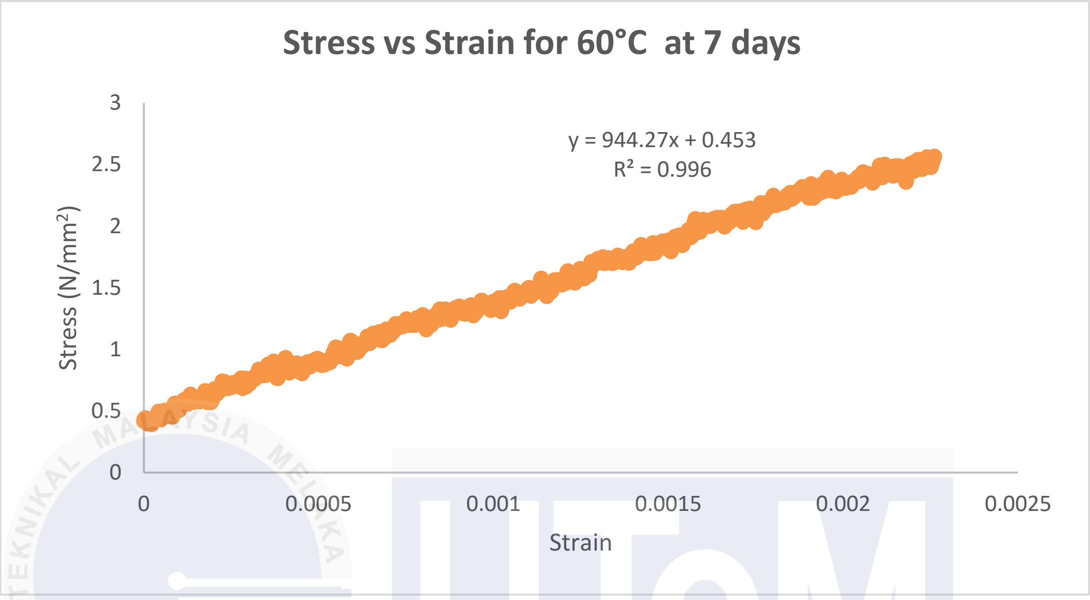

Stress vs Strain for 45°C at 9 days
y = 1646.3x + 0.3377
R² = 0.9987
Sress (N/mm²)
6
5
4
3
2
1
0
0
0.0005
0.001
0.0015
0.002
0.0025
0.003
0.0035
Strain

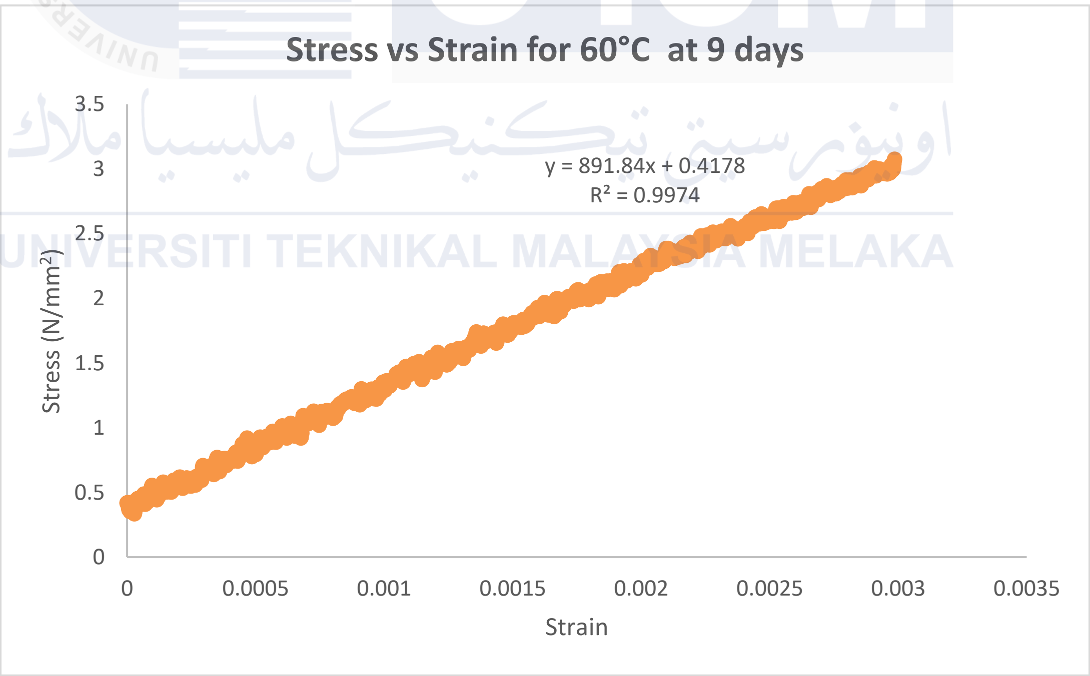

Stress vs Strain for 55°C at 3 days
y = 1984.2x + 0.4249
Stress (N/mm²)
0
1
2
3
4
5
6
7
8
Strain
0
0.0005
0.001
0.0015
0.002
0.0025
0.003
0.0035
0.004


Stress vs Strain for 55°C at 7 days
y = 1255.7x + 0.4461
Stress (N/mm²)
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
Strain
0
0.0005
0.001
0.0015
0.002
0.0025
0.003
0.0035


Stress vs Strain for 55°C at 9 days
y = 1308.7x + 0.3297
Stress (N/mm²)
3.5
3
2.5
2
1.5
1
0.5
0
0
0.0005
0.001
0.0015
0.002
0.0025
Strain


Stress vs Strain for 60°C at 7 days
y = 1886.1x + 0.4008
R² = 0.9995
Stress (N/mm²)
7
6
5
4
3
2
1
0
0
0.0005
0.001
0.0015
0.002
0.0025
0.003
Strain


Stress vs Strain for 60°C at 7 days
y = 944.27x + 0.453
R² = 0.996
Stress (N/mm²)
3
2.5
2
1.5
1
0.5
0
0
0.0005
0.001
0.0015
0.002
0.0025
Strain


Stress vs Strain for 60°C at 9 days
y = 891.84x + 0.4178
R² = 0.9974
Stress (N/mm²)
3.5
3
2.5
2
1.5
1
0.5
0
0
0.0005
0.001
0.0015
0.002
0.0025
0.003
0.0035
Strain