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Kenaf Fiber Mat/Polylactic Acid Biocomposites: Effect of Chemical Treatment on Mechanical and Physical Properties

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DECLARATION

I declare that this thesis entitled "Kenaf Fiber Mat/Polylactic Acid Biocomposites: Effect of Chemical Treatment on Mechanical and Physical Properties" is the results of my own research except as cited in the references.

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Tikar Gentian Kenaf Diperkuatkan Biokomposit Asid Polilaktik: Kesan Rawatan Kimia Terhadap Sifat Mekanikal dan Fizikal

ABSTRAK

Penyelidikan mengenai bahan berasaskan bio telah meningkat di seluruh dunia sejak ia menggabungkan bahan sumber tetulang mentah yang boleh diperbaharui dan mampan dan matriks biodegradasi untuk produk biokomposit generasi akan datang. Dalam penyelidikan ini, biokomposit asid polilaktik (PLA) diperkukuh tikar gentian kenaf (KFM) telah disediakan menggunakan teknik pengacuan mampatan. Untuk meningkatkan sifat mekanikal dan fizikal biokomposit, KFM telah dirawat secara kimia dengan natrium hidroksida (NaOH) selama 3 jam pada kepekatan 2%, 4%, 6%, dan 8%. Sifat tegangan dan penyerapan air bagi biokomposit KFM-PLA yang tidak dirawat dan alkali telah dibandingkan dan dikaji. Pencirian inframerah transformasi Fourier (FTIR) dan mikroskopi elektron pengimbasan (SEM) juga digunakan untuk menilai sifat kimia permukaan dan morfologi gentian akibat rawatan kimia. Kajian mendapati bahawa KFM dirawat alkali pada kepekatan 6% menghasilkan peningkatan ketara dalam sifat tegangan dan penyerapan air biokomposit PLA. Kajian morfologi oleh SEM membuktikan hubungan antara permukaan patah dan interaksi gentian-matriks yang baik dipercayai, dapat meningkatkan lekatan antara muka gentian-matriks selepas rawatan kimia. Secara keseluruhannya, boleh disimpulkan bahawa rawatan alkali pada KFM memainkan peranan penting dalam meningkatkan sifat mekanikal dan fizikal biokomposit KFM-PLA.

Kata kunci: Tikar Gentian Kenaf, Asid Polilaktik, Sifat Mekanikal, Sifat Fizik

Kenaf Fiber Mat/Polylactic Acid Biocomposites: Effect of Chemical Treatment on Mechanical and Physical Properties

ABSTRACT

Research on biobased materials has been increasing worldwide. It combines renewable and sustainable raw reinforcement resources materials and biodegradable matrices for the next generation of bio-composite products. In this research, kenaf fiber mat (KFM) reinforced polylactic acid (PLA) bio-composites have been prepared using the compression moulding technique. To enhance the mechanical and physical properties of the bio-composites, KFM was alkali treated with sodium hydroxide (NaOH). Treatment lasted for 3 hours at concentrations of 2%, 4%, 6%, and 8%. Tensile and water absorption properties of untreated and alkali-treated KFM-PLA bio-composites were compared and studied. FTIR spectroscopy and scanning electron microscopy (SEM) characterizations were also used to evaluate the surface chemistry and morphology of the fibers due to chemical treatment. The study observed that alkali-treated KFM at 6% led to a significant enhancement in tensile and water absorption properties of PLA bio-composites. Morphological studies by SEM proved the relationship between fracture surface and good fiber-matrix interaction, believed to enhance the fiber-matrix interfacial adhesion after chemical treatment. Overall, it can be concluded that the alkali treatment on KFM played an essential role in improving the mechanical and physical properties of KFM-PLA bio-composites.

Keywords: Kenaf Fiber Mat, Polylactic Acid, Mechanical Properties, Physical Properties

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LIST OF ABBREVIATIONS

A	Surface area
F	Maximum load force
FTIR	Fourier Transform Infrared Spectroscopy
H2O	Dihydrogen Monoxide
KFM	Kenaf Fiber Mat
KFM-PLA	Kenaf Fiber Mat Reinforced Polylactic Acid
L	Length
Na	Sodium
NaOH	Sodium Hydroxide
O	Oxygen
-OH	Hydroxyl Group
PA	Polyamide
PBS	Polybutylene Succinate
PET	Polyethylene Terephthalate
PHAs	Polyhydroxyalkanoates
PLA	Polylactic Acid
PP	Polypropylene
SEM	Scanning Electron Microscope
UTM	Universal Testing Machine
FTIR	Fourier Transform Infrared Spectroscopy
SEM	Scanning Electron Microscopy
MPa	Megapascal
GPa	Gigapascal

LIST OF SYMBOLS

%	Percentage
℃	Degree Celcius
wt%	Weight Percentage
w/t%	Weight Percentage / Volume
mm	Millimeter
kN	Kilonewton
min	Minute

CHAPTER 1

INTRODUCTION

1.1 Background of Study

A bio-composite refers to a composite material that is made by combining two or more different components, where at least one component is derived from renewable biological sources (Sengupta et al., 2017). It is a type of composite material that utilizes natural fibers or particles as reinforcement, often combined with a bio-based polymer matrix even though fossil-based polymers are also widely used for many applications. Bio-composites offer several advantages over traditional composites made from synthetic materials. They are renewable, biodegradable, and have a lower carbon footprint compared to petroleum-based composites. Furthermore, they possess good mechanical properties, including high strength and stiffness, and can be tailored for specific applications. Applications of bio-composites are diverse and include automotive parts, construction materials, furniture, packaging, consumer goods, and sporting equipment, among others (Lotfi et al., 2021).

In bio-composites, the reinforcement phase consists of natural fibers or particles such as bamboo, flax, hemp, jute, sisal, or wood. These fibers are chosen for their desirable mechanical properties, low weight, and eco-friendly nature (Yıldızhan et al., 2018). One type of natural fiber is kenaf fiber. The kenaf crop is primarily grown and found in Africa during the warm season, however it is primarily used in China and India as fabric for clothing. The advantages and objective of kenaf fiber to replacing glass and carbon fiber in considering environmental pollution and non-degradability issues are biodegradability, higher specific strength and stiffness, light weight, the production cost was low compared to other materials from natural fiber and kenaf is eco-friendly to the environment (N. Saba, 2015).

The matrix phase is typically composed of a bio-based polymer, which is derived from renewable sources like starch, cellulose, vegetable oils, or bio-based resins. The matrix holds the fibers together, providing structural integrity to the composite. Among the famous bio-based thermoplastic polymer used in the bio-based matrix is polylactic acid (PLA). Polylactic acid (PLA) is a famous bio-based thermoplastic used in bio-composites, extracted from corn and potato starch. It is non-toxic, recyclable, and biocompatible with mechanical properties similar to PET (polyethylene terephthalate) and PP (polypropylene) such as high stiffness and tensile strength (Bajpai et al., 2017). Thermoplastic polymer has the advantages that this plastic is able to be recycled to produce a new product and the resin of this thermoplastic is able to be revisable when melted and shaped without degrading by changing the mechanical properties. Conventional plastic is made from petroleum it will cause pollution to our environment. To replace conventional plastic materials with petroleum-based polylactic acid (PLA) polymers is because polylactic acid is derived from corn starch or potato starch that does not contain toxic to the environment.

However, everything in this world has its drawbacks to overcome these drawbacks from natural fibers and polylactic acid (PLA) are combined to become bio-composite so that the desired properties can be produced. Kenaf fiber mat reinforced with polylactic acid (PLA) is a bio-composite, but it has its disadvantages too. It is the problem with interfacial adhesion of kenaf fiber and polylactic acid (PLA) is not compatible together so it will occur weak performance and lack of adhesion during bleaching process. This is because kenaf fiber is hydrophilic while polymeric material like polylactic acid is hydrophobic. To enhance the interfacial bonding between kenaf fiber and the polylactic acid (PLA), it is treated with sodium hydroxide (NaOH) chemical treatment. This treatment may help between fiber and monomer to improve the thermal stability and interfacial strength. Other than that, kenaf fiber is hydrophilic it will absorb water on the surface from the environment, the water that absorbed will have changes in mechanical properties and interface of the composite. For example, the surface of the composite will crack and may cause a poor fiber matrix interface.

In this study, a kenaf fiber mat (KFM) reinforced with polylactic acid (PLA) was fabricated using the hot press compression moulding technique. The main objective is to investigate the effect of NaOH chemical treatment on the mechanical and physical properties of KFM-reinforced PLA bio-composites. The mechanical and physical properties will be characterised by tensile and water absorption testing, respectively.

1.2 Problem Statement

Environmental awareness and global warming issues have increased the growth and development of new bio-composites based research. This is due to the fact that various types of fossil-based plastic waste are non-biodegradable, contributing to climate change and risks of toxic release that can harm both human health and the environment (Sampson, 2021). The solution to solve this problem is to substitute non-degradable plastic to biodegradable plastic and synthetic fiber to natural fiber (Lotfi et al., 2021). Biodegradable plastics degrade under controlled conditions, including specific temperatures and humidity levels, or when exposed to microorganisms, oxygen, and light. Polylactic acid (PLA) is among the thermoplastics capable of controlled degradation over time, making it biodegradable, while polypropylene (PP) represents a non-degradable plastic. The properties of polylactic acid closely correlate those of polypropylene, making PLA a suitable alternative to PP in rigid products.

For many years, the usage of synthetic fiber in composite development has replaced the dependence on metal and ceramic materials. Synthetic fibers offer exceptional mechanical and thermal properties. However, they are hazardous, non-biodegradable, and relatively high in cost, both in terms of price and energy consumption during processing (Lotfi et al., 2021). On the other hand, natural fibers present themselves as environmentally friendly and cost-effective raw materials, offering acceptable mechanical and physical properties for specific applications that do not require heavy loading requirements. However, natural fibers such as kenaf, flax, hemp, and jute are primarily composed of cellulose. Due to their hygroscopic nature, these fibers have the ability to absorb moisture from their surroundings. Consequently, natural fibers exhibit high water absorbency, unlike synthetic fibers which typically demonstrate water resistance.

Due to the differences in phospholipid groups between natural fibers and biodegradable plastic (PLA), there is an inherent incompatibility between the hydrophilic natural fibers and the hydrophobic polylactic acid (PLA). This mismatch often leads to weak performance in interfacial adhesion when kenaf fiber mat is reinforced with polylactic acid (PLA). To improve the mechanical properties of the natural fibers, a chemical treatment involving sodium hydroxide (NaOH) is utilized. This treatment helps modify the kenaf fibers, making their functional groups easy to reinforcement with polylactic acid (PLA), thereby enhancing interfacial adhesion.

1.3 Objectives

1. To fabricate the KFM reinforced PLA using hot press moulding technique.
2. To evaluate the effect of NaOH chemical treatment on mechanical and water adsorption properties of KFM-PLA bio-composites.

1.4 Scope of Study

In order to characterise the mechanical (tensile test) and physical (water adsorption) properties of the kenaf fiber mat reinforced with PLA resin in this study with the objective of enhancing the strength and stiffness of the bio-composite materials, the kenaf fiber mat was selected to be immersed in NaOH chemical treatment. To evaluate the effect of NaOH concentration on the kenaf fiber mat, various concentrations were chosen, ranging from 0%, 2%, 4%, 6%, and 8%. These concentrations were selected to assess how altering the NaOH concentration influences the properties of the kenaf fiber mat. Subsequently, the kenaf fiber mat was prepared by sandwiching a layer of polylactic acid (PLA) on the upward and downward sides of the kenaf fiber mat. Moulding both of the materials with hot pressing technique at a temperature of around 180 °C. Once all the processing steps were completed, the mechanical and physical properties of the bio-composite materials were examined. Additionally, scanning electron microscopy (SEM) was utilized to characterize the surface fractures of the KFM-PLA bio-composites obtained from the tensile test.

1.5 Significances of Study

In the present study, polylactic acid (PLA) polymer is an environmentally friendly and non-toxic material that is suitable for use in food and medical applications. However, this research is to find out the other applications of PLA when reinforced with other materials such as kenaf fiber mat to become a new material that can be used in other industries. There has been remains limited research exploring its potential when PLA combined with kenaf fiber mat, particularly regarding concentration of chemical treatment, water adsorption, and tensile properties of the resulting KFM-PLA bio-composite. This study is to evaluate the effectiveness of sandwich-layer approach employing PLA plated with kenaf fiber mat. This research aims to provide valuable insights that could help improve industry practices and inspire innovation in creating sustainable, high-performance materials for various uses.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Bio-composites

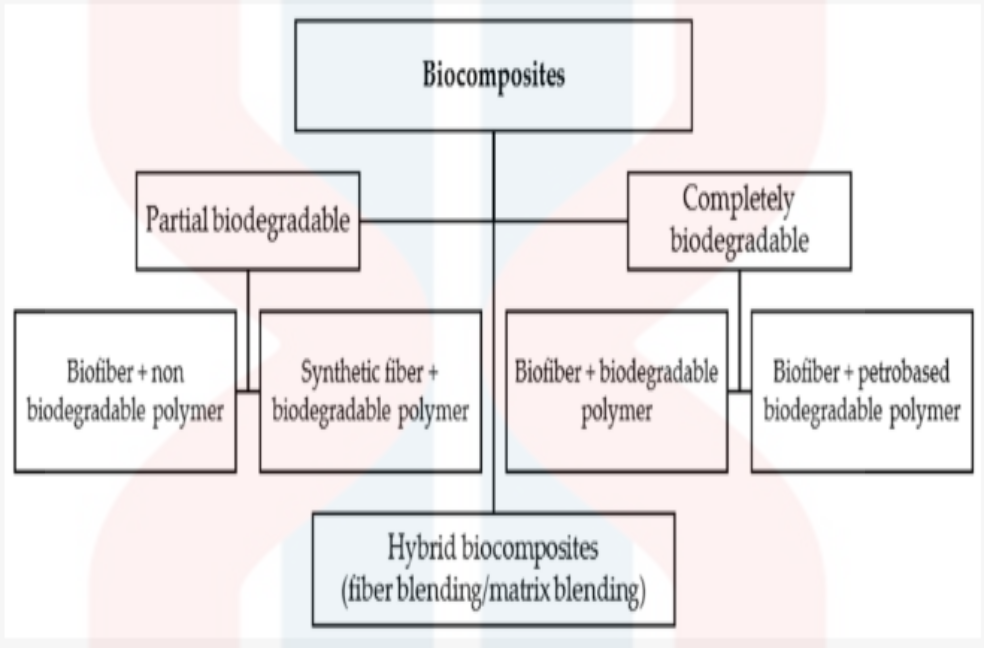
A bio-composite is a material composed of natural fibers as reinforcement and renewable polymer resin as the matrix. (Mondal et al., 2022). The reinforcement of bio-composites may be plant fiber or natural fibers, such as kenaf, sisal, jute, and cotton, while the matrix may be starch, vegetable oil, cellulose, or synthetic thermoplastic and thermoset materials such as polypropylene (PP), polylactic acid (PLA), polystyrene, and epoxy, which are typically produced from renewable resources. (Lotfi et al., 2021). The use of biopolymers in bio-composites addresses the disadvantages of pure biopolymers by improving mechanical properties and sustainability. This is because of the fact that natural fibers have a better strength-to-weight ratio, which improves the composite material's ability to withstand impacts and strength. In addition, the biodegradability of both composite materials is different, biopolymer does not break down easily, while bio-composite is able to control and degrade over time. Due to environmental concerns, trends and demand for eco-friendly and biodegradable products keep rising. At this time, bio-composite has become popular and recognised by the public.

2.1.1 Classification of Bio-composites

There are two classifications of bio-composites shown in Table 2.1 which are fully degradable and partial degradable that utilizes the basis of natural fibers or particles as reinforcement and bio-based polymer matrix (Mohanty et al., 2005). The bio-composite degradability or partial degradability is depending on the types of matrices reinforce with synthetic fibers or natural fibers. For fully biodegradable bio-composite is made from renewable polymer matrix reinforce with bio-fiber (Bahrami et al., 2020). The advantages of bio-based polymers in composites are that they can be converted into water and carbon

dioxide with environmentally friendly requirements. (Qin-hui et al., 2009). The partial biodegradable composite is made from bio-fiber reinforce with petroleum based traditional polymer as thermoplastic and thermoset.

Table 2.1: Classification of Bio-composites (Drzal, L. T., Mohanty, A. K., & Misra, 2001)



2.1.2 Thermoplastic based Bio-composites

Thermoplastic based bio-composites are the combination of natural fibers and polymer. The matrix of the composite composed of polymers that have the ability to reform and melt without degradation, it is derived from renewable sources like starch, cellulose, vegetable oils, or bio-based resins. The thermoplastic based polymer like polylactic acid (PLA), polyamide (PA) and polybutylene succinate (PBS) while the natural fibers that reinforced composite such as bamboo, flax, hemp, jute, sisal, or wood that provide high strength and stiffness, light weight and sustainability to the bio-composites. Thermoplastic based bio-composite normally found in packaging, biomedical, building and automotive industry (Pilla, 2011). This bio-composite materials application properties are unique such as toughness, high thermal and low electrical constant.

2.2 Polymer as Matrix for Bio-composites

The inherent strength of the polymer matrix is not greater than that of the fiber reinforced by the polymer matrix. Thus, combining fiber with reinforced polymer increases the strength and modulus of the composite materials. The polymer employed as a matrix for bio-composite is divided into two groups are thermoplastic and thermoset, which are made up of molecules with long chains and repeating chemical units. At the molecule level, thermoset and thermoplastic polymer differ; thermoset polymer involved chemical crosslinking of the molecules, whereas thermoplastic polymer does not have chemical crosslinking. (Mallick, 2017).

Thermoset polymer molecules are small in size and undergo chemical crosslinking. Transforming thermosets from a liquid to a solid state requires a curing process to take part in this state with the helps from heat or chemical agent to generate crosslinking reaction because thermoset is covalent bonded structure. Thermoset polymer has irreversible reaction which are it cannot be re-melt and reform back to the initial shape, but thermoset has high temperature stability, high strength and stiffness and durable properties. Thermoset is easily to shape from liquid transform to solid materials with going through chemical reaction to generate crosslinking (Meyer & Keurentjes, 2008).

2.2.1 Thermoplastic

Thermoplastics can be derived from both fossil fuels and renewable sources, such as starch, cellulose, vegetable oils, and bio-based resins. In thermoplastic crosslinking, all reactions are fully completed, meaning it does not undergo any chemical reactions like curing, and it exhibits greater toughness compared to thermoset polymers. Polymerization of thermoplastics results in long chains without branching and relatively weak intermolecular forces, such as van der Waals forces (Vaidya, 2015). Thermoplastic materials can easily be shaped and moulded into solid and liquid forms through heating. They are reversible and recyclable materials, meaning they can be re-melted, softened, and reshaped without degrading or changing the polymer's interfacial properties. Thermoplastics encompass two main types which are synthetic polymers and biopolymers. Synthetic polymers are derived from petroleum-based products and are not fully degradable, posing environmental hazards as they can persist for years without

degradation. In contrast, biopolymers are designed to be completely degradable in nature or with the help of living microorganisms. (Bajpai et al., 2014). The advantages of thermoplastics include good thermal resistance, ductility, impact resistance, and an infinite shelf life. (Vaidya, 2015). There are some examples of biopolymers and synthetic polymer polypropylene (PP), polystyrene, and polyester are examples of synthetic polymers, while polylactic acid (PLA) and polyhydroxyalkanoates (PHAs) are examples of biopolymers (Bajpai et al., 2014).

2.2.2 Polylactic Acid

Polylactic acid (PLA) represents a significant advancement in sustainable materials, being both recyclable and biodegradable. The properties of PLA closely match those of polypropylene (PP) and polyethylene terephthalate (PET). Polylactic acid is composed of less fossil fuel than synthetic polymer plastic that is manufactured from petroleum-based plastic (Henton et al., 2005). Over time, PLA degrades into water and carbon dioxide, minimizing environmental pollution. Consequently, PLA is poised to replace synthetic polymer plastics due to environmental concerns and the biodegradable nature of biopolymers. The application of PLA is mainly in food packaging and biomedical fields. The advantages of PLA are eco-friendly materials, biodegradability, biocompatibility, high tensile strength, and less energy used in production. The disadvantages of this thermoplastic polymer are low toughness, a slow degradation rate, low thermal resistance, and hydrophobicity (Farah et al., 2016).

2.3 Natural Fibers as Reinforcement Bio-composites

Natural fibers fall into three categories: animal fibers, mineral fibers, and plant fibers (Khalid, Al Rashid, et al., 2021), offer a promising alternative to synthetic fibers in reinforcing bio-composite materials due to their biodegradability and environmental sustainability. Extensive research by Amiandamhen et al. (2020); Khalid et al. (2021), natural fibers have numerous advantages over synthetic fibers, including high thermal resistance, low cost, fracture resistance, lightweight properties, and less energy consumption during manufacturing. Moreover, natural fibers exhibit impressive mechanical properties such as impact resistance, high stiffness, high strength, and high modulus. However, natural fibers have several limitations, including low fire resistance

in reinforced polymer composites and weak interfacial bonding with the polymer matrix due to rapid moisture absorption (Lotfi et al., 2021). To overcome this drawback, natural fibers must undergo chemical treatment to improve the interfacial bonding with the matrix phase. In summary, natural fibers when treated with chemical treatment, offer a sustainable and practical option for reinforcing bio-composite materials. This could lead to significant changes in how materials are made, towards a more environmentally friendly and resilient future.

2.3.1 Kenaf

Kenaf (Hibiscus cannabinus) is a crop originating from Africa. According to Webber & Bledsoe (1993), cellulose fiber of kenaf has been applied in the production of paper products and animal feeds. It is an annual crop that same with okra and cotton that plant in warm seasons (Webber & Bledsoe, 1993). The properties of kenaf have some similar with hemp, flax and jute fibers but the yield of kenaf present greater than hemp, flax and jute fibers (Hamidon et al., 2019). Kenaf has the properties of low structural integrity and strength compared to high performance and expensive synthetic fibers (Sharma et al., 2017).

2.3.2 Kenaf Fiber

Kenaf fiber has been classified into three it is cellulose, hemicellulose and lignin in chemical composition of fibers. The chemical composition of kenaf fibers is similar with wood fiber but there are different between bast and core kenaf fibers. The comparison stated in Table 2.2.

Table 2.2: Chemical composition of kenaf bast and core fibers (Tsoumis, 1991)

Chemical composition	Kenaf bast	Kenaf core	Softwood	Hardwood
Extractives (%)	5.5	4.7	0.2-8.5	0.1-7.7
Holocellulose (%)	86.8	87.2	60-80	71-89
α – Cellulose (%)	55.0	49.0	30-60	31-64
Lignin (%)	14.7	19.2	21-37	14-34
Ash (%)	5.4	1.9	< 1	< 1

The chemical composition of kenaf base and core fibers shows that extractive, holocellulose, alpha-cellulose and ash the percentages is higher than kenaf core but low percentages in lignin. According to Abdul Khalil et al. (2010) stated that if the alpha-cellulose is higher it will provide high strength and stiffness. Lignin plays an important role to support in plant which is it will hold the fibers act as binder in part of lignocellulosic plant.

According to the physical properties of kenaf core fiber, it possesses the greatest water adsorption properties. This is because when the percentage of lignin is higher, the rate of water absorption is lower, when the percentage of lignin is lower, the rate of water absorption is higher, which is related to the hydrophobic characteristic.

The mechanical properties of kenaf fibers has higher tensile strength compared with other bast fibers (Lotfi et al., 2021). It also depends on cellulose content, moisture content and fiber diameter on kenaf fibers. When cellulose content is higher in the kenaf fiber the mechanical properties is better, the fiber length and thickness will also provide high strength. For tensile strength it will increase depends on diameter of fibers, when the diameter of fibers increased the tensile strength of fiber will decrease while the diameter of fibers decreased the tensile strength will increase respectively.

2.4 Chemical Treatment on Natural Fiber Reinforced Polymeric Materials

2.4.1 Introduction

According to Aziz & Ansell (2004) and De Albuquerque et al. (2000), chemical treatment of natural fiber-reinforced polymeric materials is important because the interfacial between natural fiber and polymeric materials has poor compatibility and high-water adsorption, which will affect the mechanical properties and cause degradation. The surface of the composite will crack if it does not undergo any chemical treatment. This chemical treatment is intended to enhance the mechanical properties and water resistance of the natural fiber itself. Various chemical treatments include alkali, silane, acetylation, benzoylation, sodium hydroxide, acetic acid, isocyanates, maleate coupling agents, potassium permanganate, and peroxide (Ilyas et al., 2021). Alkali and silane are the most frequently used chemical treatment for natural fibers.

2.4.2 Alkali Treatment

Alkali treatment aimed at natural fibers, improve the interfacial adhesion between the matrix and reinforcement phase. Alkali treatment is also the cleaning process to clean the impurities as lignin, oils, wax on the fiber outer surface and to enhance the water absorption of the natural fiber with removing -OH group coating in the fiber matrix. The main function of alkali treatment is to destroy the hydrogen bonds that hold the fiber structure so that fiber able to reinforce with matrix. In alkali treatment NaOH is one of the chemicals that mainly used to improve the morphology of fiber surface. The fiber will be immersed in the NaOH chemical solution, the result performed will be depends on chemical concentration, treatment time for immersing the kenaf fiber and the temperature when immersing the kenaf fiber in NaOH solution (Hashim et al., 2017). Ibrahim et al. (2018) stated that NaOH treated natural fiber has improved the Young's modulus, tensile strength, fracture toughness, shear strength, flexural properties, the compatibility between fibers and matrix and reduce the water adsorption of the natural fibers.

The chemical reaction of NaOH polymerization with hydroxyl groups on natural fibers represent below:

$$\text{Fiber} - \text{OH} + \text{NaOH} \rightarrow \text{Fiber} - \text{O} - \text{Na} + \text{H2O} \qquad \text{Equation 2.4.2}$$

2.5 Effect of Chemical Treatment on Mechanical Properties of Natural Fiber

2.5.1 Introduction

Alkaline treatments are widely used chemical treatment method for natural fiber reinforced polymer composites. The application of alkaline treatment on natural fibers has benefited many researchers in enhancing the mechanical, thermal, and physical properties of such kinds of bio-composites. Brígida et al. (2010), Haque et al. (2012) and Zaman & Beg, 2014) has stated that coir fiber is one of the natural fibers that is widely used in fiber-matrix reinforcement composites. It has good mechanical and thermal properties and is low in cost. Due to some drawbacks of composite reinforcement between fiber and polymer matrix, which are poor interfacial compatibility, decreased mechanical properties of the composite, and increased water absorption of the composite, these will

restrict the continued use of fiber composites (Zhang et al., 2018). To overcome this drawback, conduct chemical treatment with alkaline treatment.

Alkaline treatment that will be applied is sodium hydroxide (NaOH) on natural fibers, it will help to remove impurities that contain on the fiber surface. Research done by Amiri et al. (2015) and Fiore et al. (2015) stated that the interfacial adhesion between the natural fiber and polymer matrix has been improved and the mechanical properties has been enhanced as high tensile, shear and flexural strength. The effect of alkaline treatment shows that elimination of non-cellulose components as lignin and hemicellulose it will also improve the interfacial adhesion and tensile strength of the composite.

In the observation of scanning electron microscope (SEM) shows that hydroxyl groups of coir fiber will be break by the 5 % NaOH solution immerse in room temperature for 24h and 48h will cause the crystalline cellulose increase it exposed on the surface and the interlocking of crystalline cellulose to improve interfacial adhesion of the natural fiber. The hydrophilic of natural fiber will be improve and better compatibility between fiber and matrix by NaOH.

2.5.2 Effect of Chemical Treatment on Mechanical Properties of Kenaf Fiber Mat Reinforced Polylactic Acid Bio-composites

Kenaf fiber mat (KFM) was chosen for reinforcing this composite. It is a desire material for reinforcement because it has superior toughness, low in cost and biodegradable. (Chan et al., 2013).

KFM immerse in different concentration of NaOH solution which is 3 %, 6 % and 9 % in room temperature for 3 hours and dried it for 48 hours. Concentration of 3 % NaOH solution shows that the impurities on the surface does not fully remove, concentration 6 % is the best concentration for NaOH chemical treatment and 9 % the impurities is fully removed from the surface, but the tensile strength was decrease and texture of the fiber surface was damage. The properties of the kenaf fiber with chemical treatment and without chemical treatment shows that the tensile strength is increase 13 % than without any chemical treatment kenaf fiber and when the concentration of NaOH is 6 % with the immerse duration for 24 hours, mechanical properties as tensile strength and flexural strength has been improved than the untreated kenaf fiber.

El-Shekeil et al. (2014) reported that when the 2 % concentration of NaOH added with 4 % of polymeric methylene diphe diisocyanate result that the mechanical properties as tensile strength, interfacial adhesion and wettability of the natural fiber increase. He also stated that different composition of kenaf fiber will result in different mechanical properties, interfacial bonding and water adsorption. The compositions of kenaf fiber are 20 %, 30 %, and 40 %, when the composition of the kenaf fiber increase the mechanical properties as tensile strength and strain will decrease. The result also shows that for 30 % composition of kenaf fiber content increase in mechanical properties among the others composition, the tensile strength and flexural strength has been increase but the impact strength and tensile strain is still low compared to 20 %. The 30 % of composition increase the hardness of kenaf fiber but kenaf fiber is not resistance to abrasion and thermal.

2.5.3 Effect of Chemical Treatment on Physical Properties of Kenaf Fiber Mat Reinforced Polylactic Acid Bio-composites

According Hashim et al. (2017), there is having changing on the physical properties of kenaf fiber when treated sodium hydroxide (NaOH). Kenaf fiber treated in different concentration of alkali treatment solution the result shows that the weight and density of kenaf fiber has been deducted. The alkali concentration has been classified into three concentration it is 2 %, 6 % and 10 %, for 2 % concentration of alkali treatment shows that the weight has decrease 13 %. The weight loss of the kenaf fiber is depends on the alkali concentration and immerse time, if the alkali concentration is high and the immerse time is longer the weight will be lost and the kenaf fiber increase respectively.

The water absorption of natural fiber when treated with sodium hydroxide (NaOH) shows that the percentage of water absorption is less than untreated natural fiber and the percentage of polymer's water absorption is also less. Polymer has hydrophobic natural properties so the water absorption is less while untreated natural fiber has hydrophilic natural properties so the water adsorption is high. Research by Yew et al. (2019), found that to reduce the hydrophilic natural properties, the coir fiber was treated with alkali treatment for 24 hours and 48 hours at room temperature compared with coir fiber without any treatment. The result shows that the coir fiber's hydrophilic natural properties have decreased, and the water absorption is lower than the untreated coir fiber. The compatibility of the natural fiber reinforced polymer has been improved as a result

of the removal of the layer of hemicellulose by alkaline solution and the alkaline solution penetration into the layer of lignocellulosic structure. The wettability of the natural fiber reinforce polymer also improved because the interlocking between the reinforcement and matrix has been enhanced.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

There are several materials will be used in this experiment:

1. Non-Woven Kenaf Fiber Mat (KFM)
2. Polylactic Acid (PLA)
3. Sodium Hydroxide (NaOH)

In this study, all of those materials were used in this experiment. Non-woven KFM were used as reinforcement materials while PLA resin were used as matrix materials for composite. To enhance the fiber-matrix interfacial adhesion the KFM was treated with chemical treatment. The chemical treatment used were NaOH for alkali treatment on kenaf fiber. All the materials were provided by University Malaysia Kelantan (UMK) under Faculty of Bioengineering and Technology. All measurements were taken some sample for conducting four characterizations to be carried out according with the specific size of KFM.

3.1.1 Equipments

The equipment used in this study for KFM-PLA bio-composite preparation is compression moulding. To determine the mechanical testing of bio-composite, the Testometric M500-50T Universal Testing Machine (UTM) was used to characterise the tensile properties of the composites. The physical properties characterization of bio-composite was done using an electronic weighing balance to measure the weight and the difference in water absorption. Lastly, scanning electron microscopy (SEM) was conducted on the tensile specimen's fractured surfaces with the purpose of examining the morphology of the fiber-matrix interfacial adhesion before and after chemical treatment with KFM.

3.2 Methods

3.2.1 Fiber Treatment

Kenaf fiber mat (KFM) was immersed in different concentrations of sodium hydroxide (NaOH) solution. There are two different groups of samples that were subjected to this chemical treatment as untreated and treated KFM. For treatment, KFM was treated at 2 wt%, 4 wt%, 6 wt% and 8 wt% for 3 hours at room temperature with different concentrations of sodium hydroxide (NaOH). After NaOH treatment, the KFM was cleaned with distilled water that contained 1% acetic acid to remove lignin and other impurities from the fiber cell wall. The sample and the details of its composition are listed in Table 3.1. The treated kenaf fiber was dried in an oven for 6 hours at 80℃.

Table 3.1: Treatment of KFM with different concentration of NaOH.

Sample	Concentration NaOH (w/v%)
0% of alkali-treated KFM	0
2% of alkali-treated KFM	2
4% of alkali treated KFM	4
6% of alkali treated KFM	6
8% of alkali treated KFM	8

Figure 3.1: KFM immerse in sodium hydroxide (NaOH).

Figure 3.2: KFM after dried in oven.

3.2.2 Sample Preparation

The KFM was prepared and cut with the desired dimensions of 150 mm long, 25 mm width, and 3 mm thickness. Kenaf fiber mat-reinforced polylactic acid (KFM-PLA) composite was prepared by utilising the hot press technique and mixing PLA resin with different chemical treatments of KFM. This sample is divided into two categories which is alkali treated and untreated KFM. For both categories of samples, the production step is exactly the same. Two layers of PLA were prepared using a 1mm stainless steel mould in compression moulding at 180 °C. The KFM-PLA bio-composites were prepared as a sandwiched structure in which KFM is the core and two layers of PLA are designed as skins using a 5 mm stainless steel mould. KFM was represented in one layer sandwiched between a thin PLA layer of 1 mm thickness. Followed by compression moulding, it would take 5 minutes to preheat in the machine, compress it for 8 minutes at 180 °C, and leave it cooling for 5 minutes using the compression moulding machine. Kenaf fiber mats reinforced with PLA bio-composites with various formulation designs were produced. The formulation of bio-composite is represented in Table 3.2.

Table 3.2: Formulations of composites

Samples	Composition
PLA	100% PLA
PLA/UKF	PLA + Unsaturated KF layer
PLA/2TKF	PLA + 2% NaOH treated KF layer
PLA/4TKF	PLA + 4% NaOH treated KF layer
PLA/6TKF	PLA + 6% NaOH treated KF layer
PLA/8TKF	PLA + 8% NaOH treated KF layer

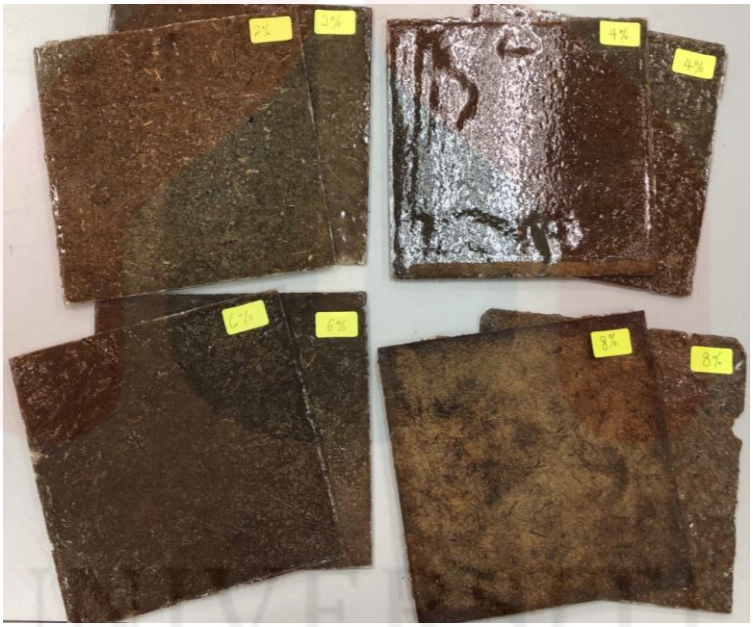


Figure 3.3: KFM-PLA bio-composites.

3.3 Characterizations

3.3.1 FTIR

FTIR is named as Fourier Transform Infrared spectroscopy. This testing is to identify and analyse the removal impurities on the fiber surface and functional groups that were conducted before and after chemical treatment when the light passed through the sample. FTIR is using visible light, which is infrared light with long wavelengths, to present information from the sample. As the sample absorbs light and causes it to vibrate, it will provide data about functional groups and chemical bonds. In order to determine an object's infrared spectrum, FTIR spectroscopy uses the Fourier transform method. The FTIR technique involves measuring the intensity of the transmitted or reflected light as a function of wavelength after the sample has been exposed to a wide variety of IR wavelengths, resulting in a spectrum. The spectrum serves as the molecular fingerprint of a substance. It makes it possible to detect new compounds, figure out how molecules combine together, examine functional groups, and maintain track of chemical processes. This non-destructive method works with solids, liquids, and gases (PhD, 2022).

FTIR applied to kenaf fiber mat composite was able to check the composition of the chemical and the structure properties, such as interfacial bonding, the compatibility between the fiber and matrix, and the fiber properties. For interfacial bonding, FTIR is able to analyse the bonding between kenaf fiber and polymer. It was present when applying chemical treatment and occurring chemical interaction between fiber and polymer, the improvement of the mechanical properties of the composite interfacial bonding. FTIR can also determine the compatibility of kenaf fiber reinforced with a polymer matrix. The mechanical properties of the composite will be affected by chemical interactions and changes at the fiber-matrix interface, which can be identified by spectral features. The properties of fiber can be used to study the chemical composite and structure using FTIR spectroscopy. The result will show whether the fibers include cellulose, lignin, hemicellulose, or chemical changes on the surface of the fiber.

3.3.2 Mechanical Properties

Tensile testing is a mechanical testing technique to test the material's stretching strength until fracture and how long it is able to resist stretching when applied force. This test was performed using a Universal Testing Machine (UTM) according to ASTM D638 with a cross-head speed of 5 mm/min and a load capacity of 50 kN at room temperature. Test specimens were prepared in dumbbell shape with dimensions of width and length (25mm x 150mm) and 3mm of thickness. Tensile strength, elastic modulus, and elongation break were analysed from the stress-strain curve graph in this tensile test. The tensile strength test outcome was in KFM-PLA-treated chemical treatment and untreated chemical treatment bio-composite. Tensile strength, elongation break, and elastic modulus were measured with the following equations 3.1:

Tensile strength = (maximum load force (F))/(Surface area (A))

Elastic modulus = stress / strain Equation 3.1

Elongation break = $\frac{l-l_0}{l_0}$ x 100%

l = Original length of test piece

l_0= Length of test piece at break

3.3.3 Physical Properties

To determine the physical properties of KFM-PLA bio-composite conduct a water absorption test and measure the thickness of the sample. The dimensions of the dumbbell-shaped sample are (150mm x 25mm x 3mm) Dry all samples in an oven at 110°C for 24 hours, and make sure all of the moisture in the sample is fully released from the composite sample. Weight the sample using an electronic weight balance to get the initial weight and a vernier capillary to get the thickness of the sample. The water absorption test and thickness swelling test were carried out by immersing KFM-PLA bio-composite in water at room temperature. The result of the water absorption was measured 24 hours after the experiment started to get the results of water uptake and thickness. To measure the weight and thickness of the sample, make sure the water on the surface is dried with a dry cloth. The measurement of the water uptake and thickness swelling was calculated and measured using equations 3.2 and 3.3:

Water uptake = $\frac{Wf-Wi}{Wi}\ x\ 100\%$ Equation 3.2

where

Wi = Initial Weight

Wf = Final Weight

Thickness swelling = $\frac{Tf-Ti}{Ti}\ x\ 100\%$ Equation 3.3

where

Ti = Initial Thickness

Tf = Final Thickness

Figure 3.4: Water absorption for 24 hours.

3.3.4 Scanning Electron Microscope

The sample's fracture morphology was determined using scanning electron microscopy (SEM). To examine the bio-composite's fracture surface, a suitable tensile test sample was collected and observed under the SEM. SEM is an advanced technology used to investigate the surface topography and morphology of materials in detail. The data that was provided from SEM was at the micro- to nanoscale level with details about the size, shape, and composition of the materials.

By using SEM, kenaf fiber-reinforced polylactic acid is able to investigate the interface of the fiber matrix, matrix properties, fiber dispersion, and fracture analysis. The fiber-reinforced matrix interface can be investigated using SEM, and the bonding and adhesion between the fibers and matrix can be examined by the SEM image. When the interfacial interactions performed well, the mechanical properties of the composite increased, as indicated by the well-bonded interface. Next, the measurement of fiber dispersion in the polymer matrix is evaluated by SEM. It is able to check the orientation of the fibers as well as how dispersed or clustered the kenaf fibers are. The alignment and uniform distribution of fibers improved the mechanical properties of the composite when reinforced with a polymer matrix. After that, the shape and structure of the polymer matrix can also be studied by SEM. It can be used to identify matrix porosity, voids, and phase separation on the surface. Last, in kenaf fiber-reinforced polymer composites, the fracture surface can be analysed using SEM. It can be discovered more about the failure mechanisms, such as interfacial debonding and matrix cracking, according to the composite fracture surfaces.

3.3.5 Research Flow Chart

The research flow chart was divided into three stages. Stage 1 is material preparation and Stage 3 is for analysis, evaluation and comparison of the obtained experimental data as shown in Figure 3.5.

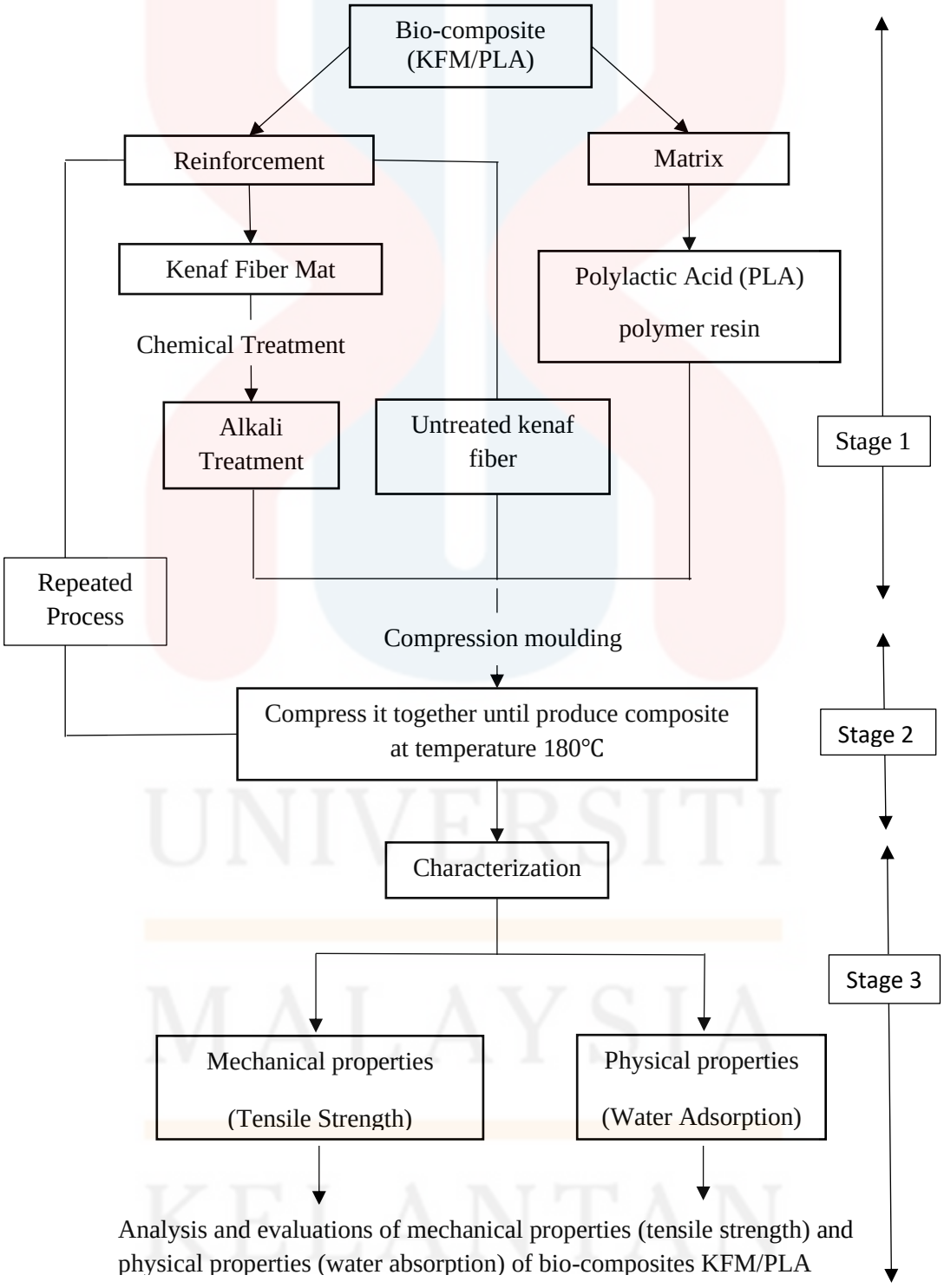


Figure 3.5: Research flow chart for KFM reinforced PLA bio-composite.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Mechanical Properties Of KFM-PLA Bio-composite

4.1.1 Tensile Strength

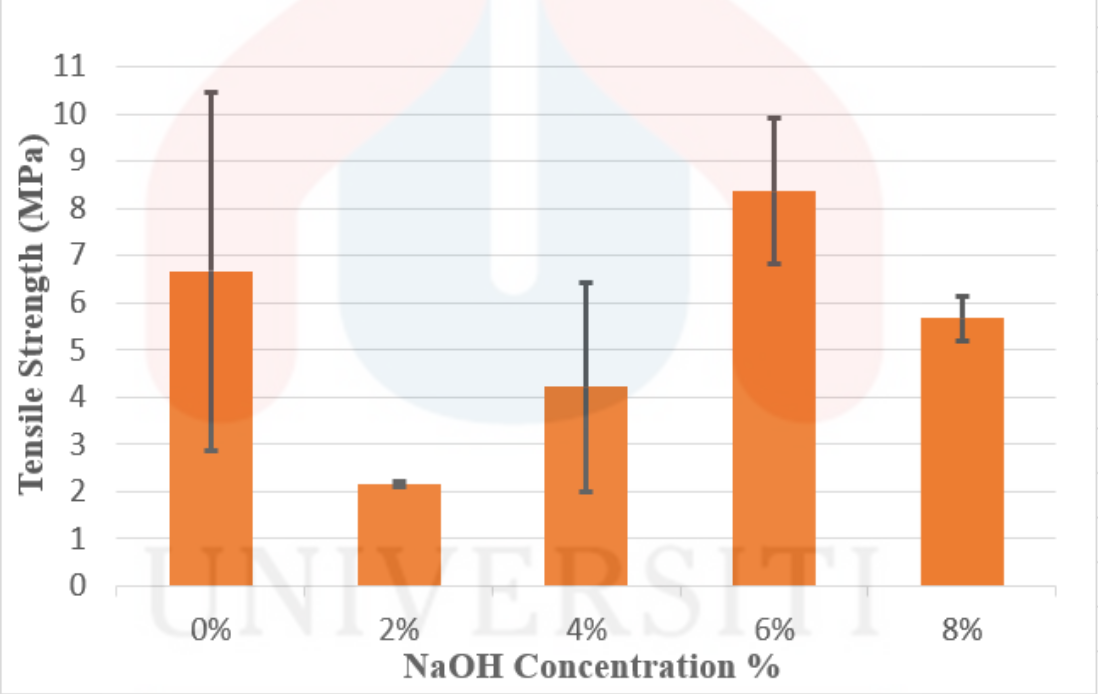


Figure 4.1: Tensile strength of KFM-PLA bio-composites.

Figure 4.1 presents the average tensile strength measurements for untreated and alkali-treated KFM-PLA bio-composites at four different percentages. Tensile strength for 2% alkali treated composite shows the least tensile strength (2.146 MPa), 4% alkali treated composite (8.359 MPa), untreated composite (4.080 MPa), 8% alkali treated composite (5.662 MPa) and 6% alkali treated composite shows the highest tensile strength (8.360 MPa) compare to untreated composite. But the decrease for 8% alkali

treated composite is still higher compared to the untreated and 2% alkali treated composite that exhibit lower tensile strength.

Figure 4.1 illustrates that tensile strength increases with NaOH concentration from 2% to 6%, then decreases at 8%. It was explained that the improvement in strength resulted in impurities being removed and an increase in homogeneity. Many researchers have addressed that alkali treated natural fiber-reinforced polymer composite demonstrate enhancement in the mechanical and physical properties resulting from fiber-matrix interfacial bonding mechanisms viz chemical adhesion and mechanical interlocking (Vigneshwaran et al., 2020). The surface of the fiber will produce better adhesion to matrix and reinforcement when it is clean, the surface roughness will be increase so the mechanical properties will be enhanced (Aida et al., 2020). Higher tensile strength indicted by the removal of voids or impurities in natural fibers. NaOH chemical treatment improved the reinforcement between fiber and matrix due to hemicellulose and cellulose has been removed from the fiber it provided better adhesion to matrix and fiber. The degree of polymerization, lignin extraction, hemicellulose substance and breakdown of hydrogen bonds within the network structure will be affected by alkali processing which is chemical treatment with sodium hydroxide (Alnaid et al., 2018). When the concentration is too high as 8%, it will result in interlaminar bonding damage to the fibers and the tensile strength will be reduced (Ikhwan Ibrahim et al., 2017). For the untreated kenaf fiber has poor interfacial bonding which created voids between the kenaf fibers and the polymer matrix (Razali et al., 2018).

In the other research according to Feng et al., (2020) conducting alkali and silane treatment on mechanical properties of kenaf and pineapple leaf fiber reinforced composite showing that when chemical treated kenaf bio-composite with concentration of 5% (20.21%) tensile strength is better than 7% of chemical treated kenaf bio-composite (17.57%). Due to the chemical treatment will has negative impact on mechanical strength of cellulose fiber based composite materials so when the concentration of NaOH above the level of 5% as 7% of treated bio-composite, degradation of fiber will occur, the load-carrying capacity of fiber was decrease and the mechanical strength of the bio-composite was decrease.

4.1.2 Tensile Modulus

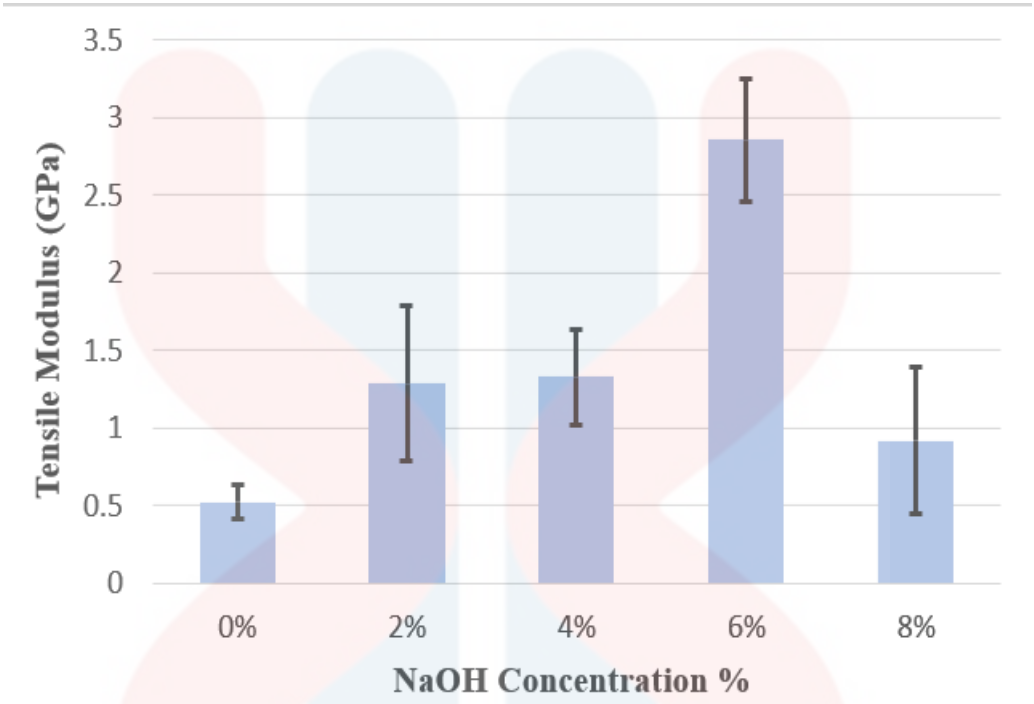


Figure 4.2: Tensile modulus of KFM-PLA bio-composites.

The graph bar represented in Figure 4.2 shows that the tensile modulus for untreated sample and four different treated composition which is 2, 4, 6 and 8%. The tensile modulus keeps increasing from untreated composite with the lowest data at 0.522 GPa, followed by 2% alkali-treated composite at1.29 GPa, 4% at 1.33 GPa, reaching the highest at 6% with 2.86 GPa, and decreasing at 8% to 0.917 GPa tensile modulus.

Tensile modulus is one of the analyses that describes how stiff or rigid the bio-composite is under stress. Based on the result of the Figure 4.2, it shows that the NaOH treated KFM-PLA bio-composites has better tensile modulus than the untreated fiber. This is because hemicellulose is the most reactive component in the structure of fiber. When the concentration of chemical treatment on kenaf fiber is continuously rising, the tensile modulus will consistently increase as can be observed in the graph from 0% to 6% but decrease until 8%. The reason is that the interaction between the fiber and matrix and the lack of a polymer chain between the fiber layers may also be the source of the tensile modulus decrease (Rahman et al., 2018). When kenaf is reinforced with PLA, the strength of the short fiber will decrease due to the short fiber's inability to support the stress that transfers from PLA (Kamarudin et al., 2019). The weak structure of the bio-composite

was influenced by the fiber's uneven forms, which made it impossible for the fiber to sustain stress transfer from the polymer matrix. In the different perspective, the surface will be rougher when the hydrophobic properties from the fiber has been removed which will increase the ability to transfer the stress in fiber reinforced with polymer bio-composite (M. Mohammed et al., 2022).

4.1.3 Elongation at Break

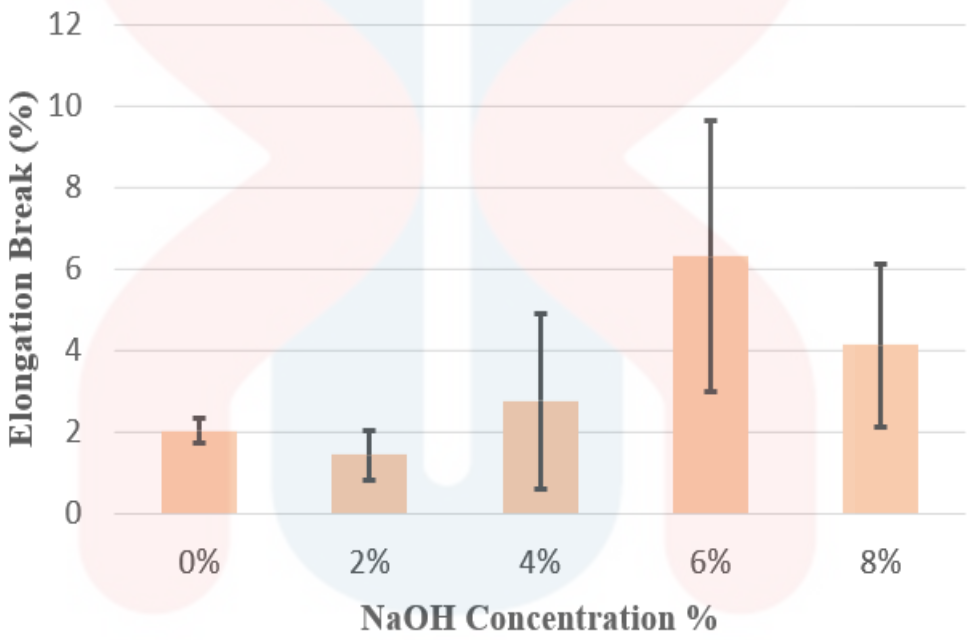


Figure 4.3: Elongation at break of KFM-PLA bio-composites.

Based on Figure 4.3, the average measurement of elongation at break for 5 different specimens that are untreated and 4 different percentages of alkali-treated KFM-PLA bio-composite are 2, 4, 6, and 8%. The tensile elongation at break decrease from untreated composite with the 2.04%, decreased to 2% concentration with the lowest data at 1.48%, increasing at 4% with 2.78%, reaching the highest at 6% with 6.323%, and decreasing at 8% to 4.14%.

Elongation at break describes how long it can stretch before breaking. The higher elongation at break for the 6% alkali-treated composite proved that if the lignin and pectin were removed, it would increase the kenaf fiber's elasticity. The elongation of kenaf fiber increases with increasing percentages of the alkali treatment. Based on the result, the elongation at break for the untreated kenaf fiber was 2.044% and improved around

2.479% to reach 6%. The alkali-treated composite had the highest elongation at break of 6.323%. It shows that intermolecular or hydrogen bonds breakdown after releasing too much strength. As the concentration increases, the percentage of the elongation break will also increase (Islam et al., 2022). In this study NaOH is used to improve the interfacial adhesion between kenaf fiber reinforce PLA with removing the lignin and wax on the surface of kenaf fiber to produce the surface rougher. The standard deviations for untreated fiber and alkali-treated with 2%, 4% and 8% is lowest compared with the value for 6% alkali-treated composite.

4.2 Physical Properties For KFM-PLA Bio-composite

4.2.1 Water Absorption

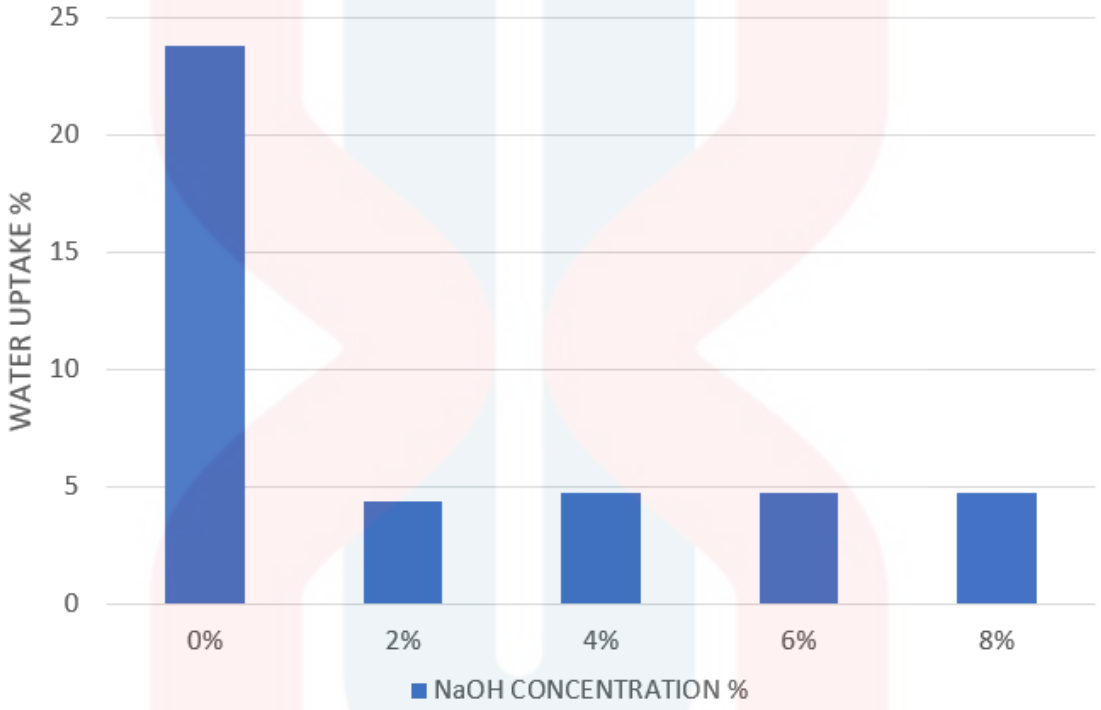


Figure 4.4: Water uptake of KFM-PLA bio-composites.

Water uptake for KFM-PLA bio-composite was determined over 24 hours at room temperature involve 5 different specimens that are untreated and 4 different compositions of alkali treatment for KFM-PLA bio-composite 2%, 4%, 6%, and 8%. Figure 4.4 reveals a slight decline in water uptake from untreated kenaf fibre reinforced PLA (PLA/UKF) 23.81% to alkali-treated kenaf fibre reinforced PLA (PLA/TKF) 4.76%. The untreated bio-composite has the highest percentage of water uptake compared to four different compositions of alkaline-treated bio-composite. This can be attributed to the poor wettability and interfacial adhesion of untreated kenaf fibre when reinforced with polymer matrix. Untreated fibers retain lignin and hemicellulose on their surface, creating voids and resulting in a hydrophilic nature that increases water uptake. It has hydrophilic nature of cellulose fiber that will increase the proportion of water uptake. Natural fibers like untreated kenaf fiber contain pores and hydroxyl groups, further influencing water absorption through porosity and voids in the fiber reinforced matrix. Hydroxyl groups

interact with water molecules via hydrogen bonds, increasing water uptake, especially in regions of poor interfacial bonding. Interfacial adhesion, which will speed up and attract the moisture that absorbed by the fiber. Interfacial bonding is one of the factors that results in poor bonding in voids and small gaps when reinforced with matrix. (Ghori et al., 2023). Mohammed et al., (2020) mentioned that higher water absorption in untreated fiber reinforced polymers to their hydrophilic properties and porous structure. Conversely, treated bio-composites demonstrate superior water resistance due to alkali treatment, which reduces hydroxyl groups in the fiber structure, decreasing water absorption and enhancing interfacial bonding with the matrix.

According to Azammi et al. (2020), emphasize that alkali-treated fibers exhibit reduced hydrophilicity, leading to strengthened interfacial bonding with matrix. Alkali treatment promotes strong intermolecular bonding between the fiber and matrix, thereby reducing void volume and limiting water absorption on the surface (Ismail & Ishak, 2018).

Table 4.2: Maximum water uptake of KFM-PLA bio-composite

Composite	Maximum Water Uptake %
PLA/UKF	23.81
PLA/2TKF	4.35
PLA/4TKF	4.76
PLA/6TKF	4.76
PLA/8TKF	4.76

4.2.2 Thickness Swelling

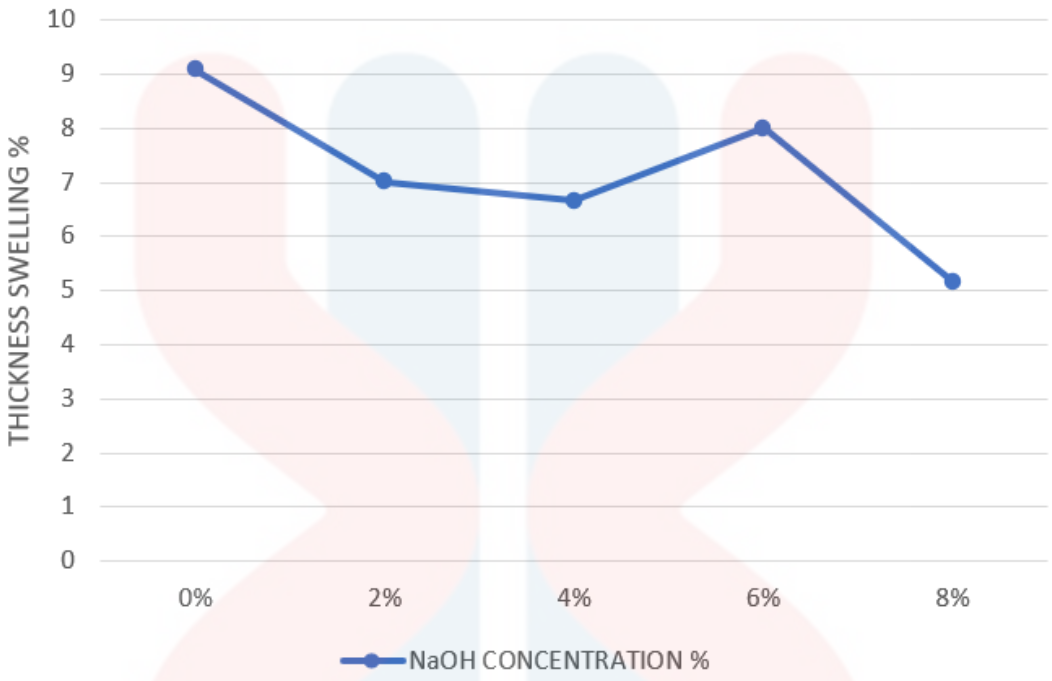


Figure 4.5: Thickness swelling of KFM-PLA bio-composites.

Similar to water uptake, untreated fiber based composites possess polar groups that attract water molecules through hydrogen bonding. Consequently, moisture accumulates in cell walls, leading to fiber swelling at the fiber-reinforced matrix interface and resulting in lower water resistance. (A. R. Mohammed et al., 2020). The results for five different untreated specimens and four different concentrations (2%, 4%, 6%, and 8%) of alkali-treated KFM-PLA bio-composites on thickness swelling tests, after being immersed in water for 24 hours, are shown in Figure 4.5. The maximum thickness swelling for untreated bio-composite (PLA/UKF) with the highest thickness swelling at 9.09%, followed by 2% alkali treated bio-composite (PLA/2TKF) at 7.02%, 4% at 6.67% decrease significantly further increase at 6% with 8.00% while reaching the lowest value at 8% with 5.17%. Usually, it results from water absorb into the interface though the micro cracks that are triggered by fiber swelling. This is because kenaf fiber is hydrophilic and polar, it has high hydroxyl group while polymer matrix is hydrophobic. Hydrophilic for kenaf fiber will absorb more water and fiber swelling compared to hydrophobic for polymer matrix.

Three different concentration of alkali-treated bio-composite as 2%, 4% and 8% show slightly decreasing result of thickness swelling are 7.02%, 6.67% and 5.17%. The decreasing result is because of the good interfacial adhesion and fewer voids appear which means it exist hydrogen bonding in treated kenaf fiber and polymer matrix. It shows that fewer microcrack in interface of treated fiber reinforced matrix. The bonding between the fiber and matrix enhanced which helps to prevent water to absorb by the composite so the thickness swelling will be reduced (Mohd Hafidz et al., 2021). For 6% of alkali-treated bio-composite demonstrated that poor interfacial adhesion due to the amount of water that diffuse into the composite was large compared to the concentration which mention before this (Ramlee et al., 2019). The most stable thickness swelling was 8% alkali-treated bio-composite due to it result in lowest percentages of swelling after one day of immersion.

Table 4.2: Maximum Thickness Swelling of KFM-PLA

Composite	Maximum Water Uptake %
PLA/UKF	9.09
PLA/2TKF	7.02
PLA/4TKF	6.67
PLA/6TKF	8.00
PLA/8TKF	5.17

4.3 Fourier Transform Infrared Spectroscopy Analysis

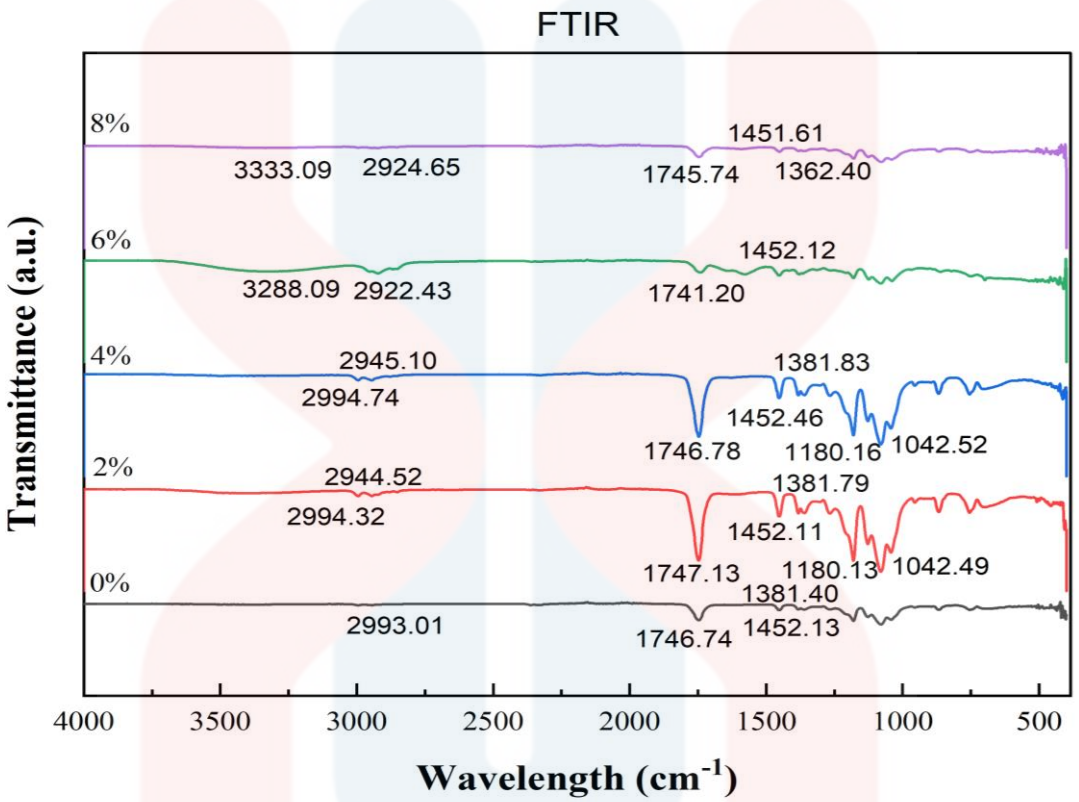


Figure 4.6: FTIR Spectra.

Figure 4.6 shows the Fourier Transform Infrared Spectroscopy (FTIR) spectra for untreated kenaf fibre and for kenaf fiber treated with four different concentrations of alkali (2%, 4%, 6%, and 8%) in the fingerprint region between 4000 and 400 cm1. The broad absorption band at 3333.09 cm1, as shown in Figure 4.6 for the 8% treatment, indicates O-H stretching vibrations of hemicellulose and lignin. Peaks around 2800 – 2900 cm1 for both untreated and alkali-treated kenaf fiber (2%, 4%, 6%) are associated with the C-H group stretching vibration in cellulose and hemicellulose. This bond typically represents a carboxylic acid group. The treated kenaf fiber exhibits higher absorbance values in this range, indicating weakened cellulose and hemicellulose compared to untreated fiber. This weakening results from the partial removal of hemicellulose, where the glucose chain is broken down by the hydrogen bond during the chemical treatment process (Ahsan et al., 2019).

The peak around 1740 cm^{-1} corresponds to C=O stretching in hemicellulose of acetyl group and cyclopentanone occur vibration. The intensity of these peaks decreased after chemical treated, demonstrating that most of the lignin has been eliminated. A stretching peak was detected around 1300 – 1400 cm^{-1} for treated and untreated bio-composite exhibited the bending vibration of C-H groups of alkane from methylene group that present in cellulose, lignin and hemicellulose. Every concentration of chemical treatment for bio-composite and untreated bio-composite has showed peak in this region as untreated (1381.40-1452.13), 2% treated bio-composite (1381.79-1452.11), 4% treated bio-composite (1381.83-1452.46) and 8% treated bio-composite (1362.40-1451.61). 8% treated bio-composite is weaker compared to untreated bio-composite, 2% treated bio-composite and 4% treated bio-composite which refer to decrease in hemicellulose. Transmittance at 1180 cm1 results from asymmetrical C-O-C stretching in cellulose and hemicellulose. The strong peaks at 2% (1042.49 cm1) and 4% (1042.59 cm1) are attributed to CO-O-CO stretching characteristic of anhydride groups present in cellulose acetate.

4.4 Scanning Electron Microscopy

4.4.1 Morphologies of Tensile Fracture Surface of KFM-PLA Bio-composite

A Scanning Electron Microscope (SEM) scans surfaces with a focused electron beam to observe samples at the microscale. The SEM image of tensile fracture surface of KFM-PLA for untreated kenaf fiber and alkali-treated kenaf fiber with different concentrations is shows in Figure 4.7. From Figure 4.7 (a) it appears that untreated kenaf fiber surface is smooth and consists of some impurities as hemicellulose, lignin and wax that cover the surface of the fiber. The presence of wax and hydroxyl groups on the fiber surface causes it to be hydrophilic, reducing the interfacial adhesion strength with the matrix. (Aravindh et al., 2022).

The void section that is represented in Figure 4.7 (a) result from an incompatible interaction between kenaf fiber and matrix. Insufficient bonding between the matrix and reinforcements leads to poor interfacial results, while kenaf fiber aggregation further weakens the mechanical properties of the composite. Moreover, these voids negatively impact the load transfer within the bio-composite.

Figure 4.7 (b-f) reveals the morphological changes and concentrations of alkali-treated kenaf fiber which are 2%, 4%, 6% and 8%. Based on Figure 4.7 (b-c) showed a partial removal of impurities from the surface with increasing alkali treatment concentration. However, as the concentration continues to rise, the surface structure suggests inadequate enhancement of fiber adhesion and compatibility with the matrix. While the tensile strength increases from 2% to 6% alkali treatment, it decreases at 8% concentration due to the fiber structure damage (Liu et al., 2019).

In comparison to lower concentrations, Figure 4.7 (e) indicates that an 8% alkali treatment results in fiber structure damage, adversely affecting mechanical properties. Higher concentrations lead to rougher surface roughness (Salih et al., 2020). Figure 4.7 (f) shows partial bonding between fibers and the matrix, with tensile strength decreasing after the peak at 6% concentration. According to Zhang et al. (2018), reported that the highest concentration NaOH treatment in the research which is 10% result that the bamboo fiber's surface has been damaged and caused critical fiber fibrillation, damaging the integrity of the fiber bundles. When the alkali concentrations much higher, the

strength of the sample were decrease and there will remove partial of cellulose. Higher alkali treatment will cause the mechanical properties damaged. The researcher also stated that 6% of alkali treatment is the optimum concentration were better suited for the production of bamboo fiber reinforced epoxy composites.

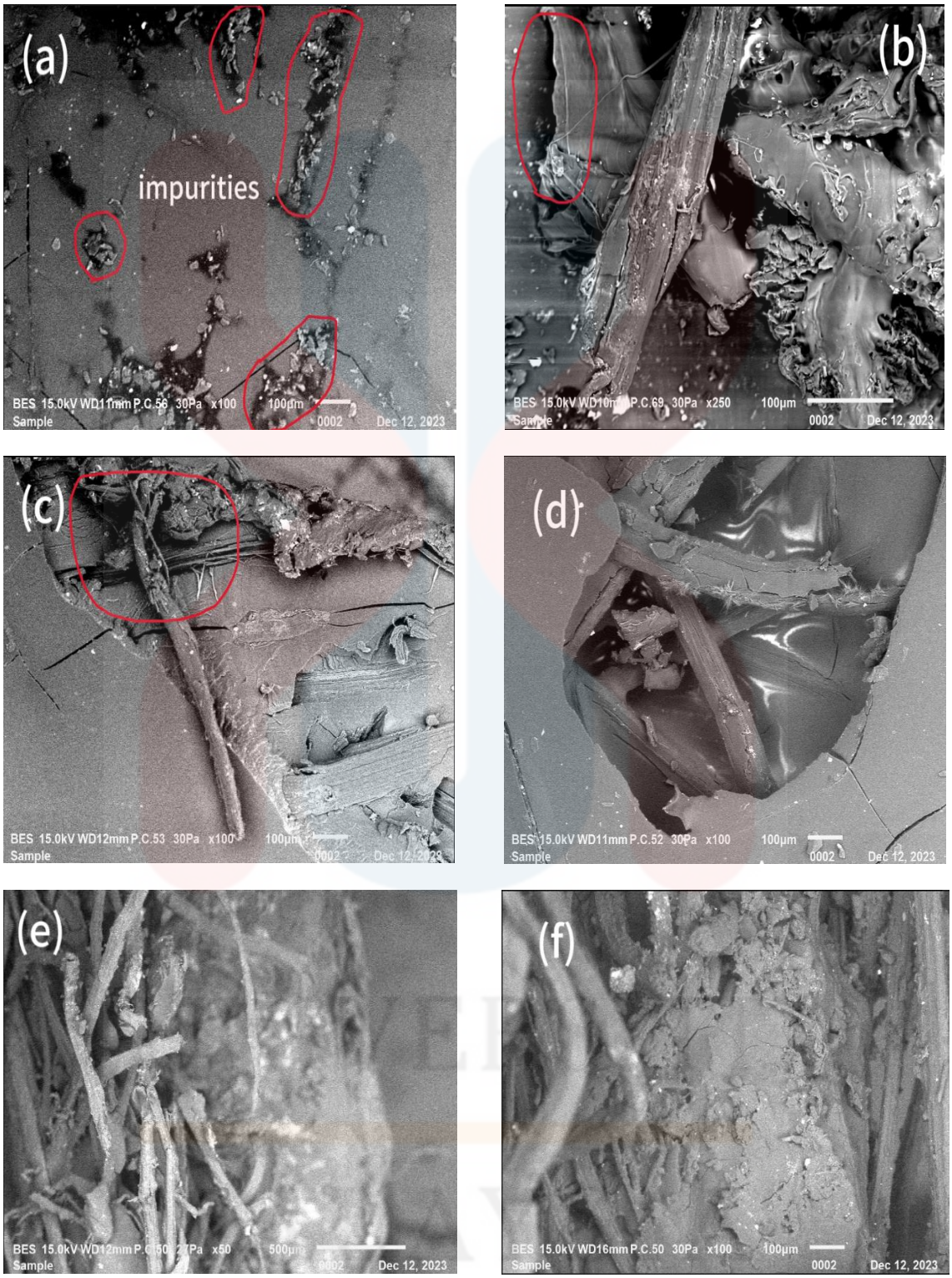


Figure 4.7: SEM microstructure of surface fracture after tensile testing (a) untreated kenaf fiber, (b) 2% concentration of NaOH treated kenaf fiber (c) 4% concentration of NaOH treated kenaf fiber (d) 6% concentration of NaOH treated kenaf fiber (e-f) 8% concentration of NaOH treated kenaf fiber.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

4.5 Conclusion

In conclusion, kenaf fiber mat reinforced polylactic acid (KFM-PLA) bio-composite was manufactured using a compressing moulding machine to compress the sample of this alkali-treated kenaf fiber bio-composite. Overall, NaOH alkali-treatment on kenaf fiber was shown to be the most effective method for enhancing interfacial adhesion and interlocking with polylactic acid by tensile strength and water absorption tests. This treatment removed impurities from the fiber surface and increased surface roughness. The mechanical testing, physical testing, and SEM results discovered that kenaf fiber treated with NaOH alkali treatment considerably enhanced its tensile properties and improved its hydrophilic structure since the kenaf fiber is easy to absorb water, and the image of the SEM showed the impurities of the fiber were fully removed from the surface. A 6% NaOH concentration represents the optimum concentration for enhancing the mechanical properties of the bio-composite compared to untreated kenaf fiber. Higher concentration of alkali treatment will cause the fiber to be damaged, as can be observed in the tensile strength result, which showed that 8% was the highest concentration. The data trend shows that 8% alkali treatment is lower than untreated bio-composite. As a consequence, alkali treatment on kenaf fiber has enhance the mechanical and physical properties of KFM-PLA bio-composite.

4.6 Recommendations

For future research, it is recommended to explore other mechanical testing methods, such as flexural strength testing, to evaluate the bio-composite's resistance to deformation and bending strength after alkali treatment. Additionally, investigating alternative treatments to NaOH, including silane, peroxide, sodium chloride, and maleated coupling agents, could provide insights into further enhancing the bio-composite's properties. Testing these various treatments may reveal more about the potential for improving the durability and performance of kenaf fiber reinforced composites.

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