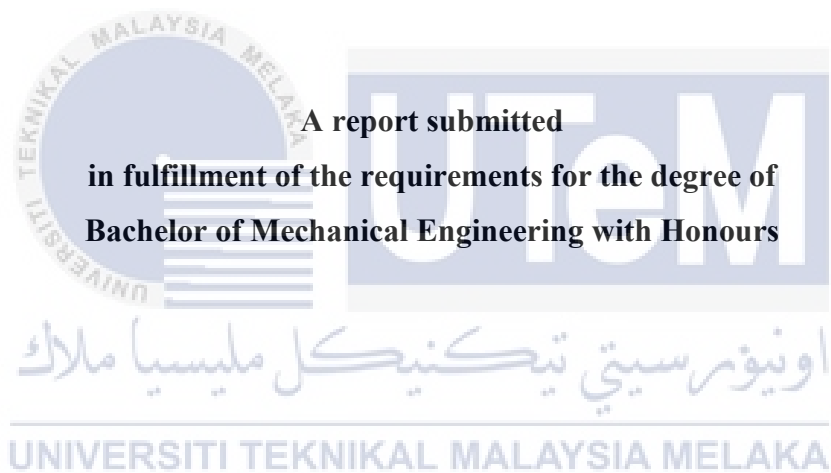


**AN INVESTIGATION ON PHYSICAL, MECHANICAL AND MORPHOLOGICAL
BEHAVIOUR OF CORN STARCH REINFORCED WITH ALKALINE TREATED
PINEAPPLE LEAF FIBRE**

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2018

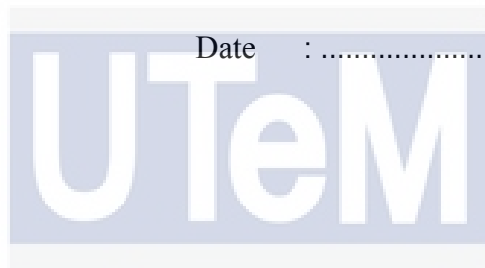
DECLARATION

I declare that this project entitled “Investigation on Physical, Mechanical and Morphological of Corn Starch Reinforced Alkaline Treated Pineapple Leaf Fibre” is the result of my own work except as cited in the references.

Signature :

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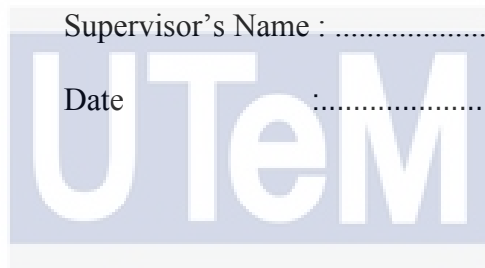
APPROVAL

I hereby declare that I have read this project report and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering with Honours.

Signature :

Supervisor's Name :

Date :



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DEDICATION

To my beloved mother and father



ABSTRACT

Recently, natural fibre reinforce composite (NFPC) is knowingly as attentive source and have potential replacement over common synthetic fibres in several applications that commonly related to automotive and construction industry. The purpose of this study is to investigate the physical, mechanical and morphological behaviour of natural composite of corn starch reinforced alkaline treated pineapple leaf fibre. Composites can be defined as material with multifunctional system that have certain characteristic which cannot be found in other discrete material. Previously, pineapple plant is not fully utilised and has become as the agriculture waste until the researchers found that pineapple leaf can be turned into fibre that is beneficial to certain industries and applications. Due to rapid technology era and high knowledge people nowadays, pineapple leaf fibre has been used widely for composite production because the fibre own good mechanical properties and shows superior properties based on the high cellulose content and low microfibrillar angle (Kengkhetkit and Amornsakchai, 2012). Several steps have been done to evaluate the physical, mechanical and morphological behaviour of natural composite between pineapple leaf fibre and corn starch. Initially, pineapple leaf fibre and corn starch was prepared according to the certain fibre loading. Several tests were conducted on the samples to determine the physical properties such as moisture content, water absorption and density. Regarding to the results obtained, all of these physical properties is increase linearly with the increasing fibre loading wt%. Besides, mechanical properties such as tensile, flexural strength and hardness also determined by mechanical tests. The results show that tensile properties is optimum at 60 wt% while for the flexural strength is maximum at 40 wt% fibre loading. Based on this study, the physical and mechanical properties are depending on the fibre loading wt%. The characterization of the fracture tensile also figured out by conducting Scanning Electron Method (SEM). Work frame also provided as for better improvements in the future.

ABSTRAK

Sejak kebelakangan ini, komposit serat semulajadi diketahui umum sebagai sumber yang mendapat perhatian dan berpontensi menggantikan serat sintetik dalam beberapa aplikasi yang berkait rapat seperti dalam industri automotif dan pembinaan. Tujuan kajian ini adalah untuk mengetahui tentang sifat fizikal, mekanikal dan morfologi yang wujud pada komposit serat semulajadi yang melibatkan tepung jagung dan serat daun nenas yang telah dirawat dengan alkali. Komposit boleh didefinisikan sebagai bahan yang mempunyai pelbagai kegunaan dan sifat yang tiada pada material lain. Sebelum ini, daun nenas tidak digunakan dan menyebabkan ia menjadi bahan buangan dalam sector agrikultur sehingga pengkaji menyedari bahawa daun nenas boleh diekstrak menjadi serat yang bermanfaat untuk beberapa industri dan aplikasi. Dalam era pembangunan yang pesat dengan teknologi dan manusia yang berpengetahuan tinggi, serat daun nenas telah digunakan secara meluas untuk pembuatan komposit kerana serat daun nenas mempunyai sifat mekanikal yang bagus dari segi kandungan selulosa dan sudut mikrofibrila yang rendah (Kengkhetkit and Amornsakchai, 2012). Beberapa langkah telah dijalankan untuk mengkaji sifat fizikal, mekanikal dan morfologi yang ada dalam komposit semulajadi yang melibatkan tepung jagung dan serat daun nenas. Langkah awal telah dijalankan dengan menyediakan tepung jagung dan serat daun nenas kepada beberapa komposisi serat dari segi peratusan berat. Beberapa ujian juga telah dijalankan untuk mengetahui sifat fizikal yang terdiri daripada kandungan kelembapan, serapan air dan ketumpatan. Berdasarkan keputusan yang diperolehi, kesemua keputusan fizikal ini menunjukkan kenaikan yang sekata mengikut peratusan berat serat yang makin bertambah. Selain itu, sifat mekanikal seperti kekuatan tegangan, kekuatan lenturan dan kekerasan juga dikaji melalui ujian mekanikal. Keputusan menunjukkan kekuatan tegangan adalah optima pada 60 peratus berat serat, manakala kekuatan lenturan memberi nilai maksima pada 40 peratus berat serat. Berdasarkan keputusan yang diperolehi, sifat fizikal dan mekanikal komposit semulajadi ini adalah bergantung kepada peratusan berat serat daun nenas. Ujian morfologi juga dijalankan untuk mengetahui pencirian komposit pada permukaan yang terjejas setelah menjalani ujian tegangan. Rangka kerja untuk menaik taraf kajian pada masa akan datang juga telah disediakan.

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LIST OF ABBREVIATIONS

PALF	Pineapple Leaf Fibre
ASTM	American Society for Testing and Material
SEM	Scanning Electron Microscopy
NFPC	Natural Fibre Reinforced Polymer Composite
CMC	Ceramic Matrix Composite
PMC	Polymer Matrix Composite
MMC	Metal Matrix Composite
PP	Polypropylene
PE	Polyethylene
PS	Polystyrene
PVC	Polyvinyl chloride
TPS	Thermoplastic Starch
UTS	Ultimate Tensile Strength

LIST OF SYMBOLS

m	- Mass
ρ	- Density
V	- Volume
A	- Area

r - Radius

F - Force

$\text{wt}\%$ - Weight percentages

σ - Stress

NaOH - Sodium Hydroxide

OH^- - Hydroxide

H_2O - Water

Na^+ - Sodium

CO_2 - Carbon Dioxide

l - Length

w - Width

t - Thickness

CHAPTER 1

INTRODUCTION

1.0 Background of Study

Natural fibres have been used since the dawn of civilization. For thousand years, natural fibres were used by the mankind as the essential things to insulate the bodies against warm and cold weather, protect the food from vermin and even decorate the environment. In addition, clothing, housing, and even baskets that used to store and carry food back then were made of natural fibre. The earliest evidence of human using fibres found that date back to 36000 BP when the discovery of wools and dyed flex in a prehistoric cave in the Republic of Georgia. Until the last hundred years or so, the only fibres used by mankind were natural fibre and the first man-made fibre that known as synthetic fibre was discovered in 1865.

Recently, natural fibre composites and synthetic fibre composites were commonly used in worldwide as they are useful for the high-tech applications such as composites part for automobiles, material industries, food product industries and etc. Although there are good development in using the synthetic fibre composites based product nowadays, there are lot of crucial environmental issues that can affect to our health, leads to global warming and disposal of plastic products in our world. One of the common issues is protecting the environment from using non disposable material. Many researchers have considered to spread the goodness of using natural fibre composites rather than synthetic fibre composites in industrial and our daily activities. Composites with natural fibres have advantages such as lower density, lower processing cost, good thermal insulation and reduce skin irritation compared to glass fibre composites. Nowadays, there are a lot of bio-composite products

that have been commercialized from plant based fibres. Some examples of composites based on natural fibres include kenaf, jute, sugar palm fibres and banana pseudo stem.

The pineapple leaf fibre is one of the natural fibre which has great potential to be used in bio-composite engineering. Pineapple plant can be found abundantly in Malaysia even though the top pineapples producing countries are Costa Rica, Brazil, Philippines and Thailand. Pineapple plants have contributed development in socioeconomic in Malaysia as our country has the industries that produced canned pineapple, pineapple extracted juice and etc. Pineapple fruit has high demanding recently, but there is still lack of use of the plant because pineapple leaf normally become agriculture waste. Many researchers try to make value added for pineapple leaf and transform them from “trash” to “cash”. The fibre is extracted from pineapple leaf using extract machine in order to soften the fibre for easy handling. Pineapple leaf fibre have its own advantages such as it having highest cellulosic content nearly 80% and Young Modulus shows highest tensile strength when compared to other natural fibres (Mohammed *et al.*, 2015)

This study discusses the physical, mechanical and morphological behaviour between alkaline treated pineapple leaf fibre and corn starch. Besides that, the effect of fibre loading wt% of pineapple leaf will be discussed thoroughly. The effect of alkaline treatment and the specific length of pineapple leaf fibre is also taken into account

1.1 Problem Statement

Currently, studies on new combining composite materials focus on enhancing their mechanical and physical properties while reducing the production cost. Many researchers shown their effort in studies and producing the composite materials that are eco-friendly and sustainable to industries worldwide.

Commonly, petroleum and natural gas are raw materials for plastic manufacturing that contribute in many industries such as packaging, building and construction, aerospace and automotive. They are assumed to depletion within next 40-60 years that make the mankind think and give a lot of attention in renewable energy and sustainable industry. Natural composite abundant materials such as cellulose fibre have widely utilized as natural fibre reinforcement in a composite to partially replace the petroleum-based plastic material. As for the natural fibre composite that obtained from plant only require short time of growth before it is ready to harvest. The benefits are not only increases productivity of the product, but natural fibre also reduced cost, improves mechanical properties and composite weight reduction.

The natural fibre composite can be extracted from plant such as kenaf, jute and pineapple. This study will be focused on pineapple leaf fibre as it has good potential to replace current reinforced materials such as glass and carbon fibre that can be easily get. Pineapple is one of the popular fruit crop grown in Malaysia and the planting of pineapple is presently confined to the peat soil area commonly located in the state of Johor and in other states such as Kedah, Perak, Negeri Sembilan, Kelantan, Terengganu and Sarawak. The pineapple industry in Malaysia is the oldest agriculture based export oriented industry even though it is relatively small compared to palm oil and rubber. Nowadays, pineapple industry plays important role in the country's socioeconomic development because of the huge production every year. However, tons of pineapple leaf have become agricultural waste after harvesting that attracted the researcher to make use of it for value added in certain industries. Previous study proves that pineapple leaf fibres that have undergo extraction process called mechanical milling and the fibre knowingly consist of cellulose about 70-80 wt% with higher specific modulus and strength (Panyasart *et al.*, 2014). Hence, the pineapple leaf fibre

seems to be suitable for making them as the products but there are still some improvement need to be done to enhance their properties.

These days, we can see many products that have been commercialized are made up from natural fibre composites. However, most of the product that made up from natural fiber composites that also known as biodegradable and good for environment are actually not fully natural. For instance, plastic bags, food packaging and automobile parts is made up from natural fibre composites but still using synthetic as the matrix. In this study, natural matrix that is corn starch will be used to blend with the pineapple fibre leaf. The advantages of using corn starch as the matrix are low cost and availability from agricultural crop. Starch based matrix, however are brittle and hydrophilic, therefore the processing and application using it is limited. In order to overcome these drawback, glycerol that is colourless, odourless and viscous will be added as the non-toxic plasticizer into the corn starch. These plasticizers function is to decrease the absorption of water in starch, which prevents the material from becoming brittle.

1.2 Research Objectives

The objectives of this study are stated as below:

1. To investigate the effect of fibre loading of the alkaline treated PALF reinforce corn starch composite to the physical and mechanical properties.
2. To evaluate the effect of fibre loading of the alkaline treated PALF reinforce corn starch to the morphological properties.

1.3 Scope of Study

The aim of this research is to accomplish a greater understanding about the pineapple leaf fibre's properties in terms of its mechanical behaviour, physical and morphological

properties. The pineapple leaf fibre was treated using alkaline treatment in order to evaluate the effect of treatment to their properties. Finally, the fibre was lay up with natural matrix which is corn starch to form natural composites samples. The methodology in this research is experimental investigation. The research was classified into three different phases.

The first phase of the research is to evaluate the characterization of pineapple leaf fibre after undergo alkaline treatment. The pineapple leaf was collected randomly from Johor. The pineapple leaf fibre was extracted from the pineapple leaf using mechanical milling method. The extracted pineapple leaf fibre then treated with alkaline treatment and used as the research samples. This study does not cover the details on the pineapple plant and the biological effect of the plant in term of location and species. This study focuses on the characterization of physical (density, water absorption and moisture content), mechanical (tensile strength and flexural strength properties) and morphological behaviour. The results were then compared according to the different loading of pineapple leaf fibre.

The second phase of this research is to determine the mechanical behaviour of treated pineapple leaf fibre according to its specific length (1cm). After undergo alkaline treatment, the pineapple leaf fibre is scratched to soften and as to ensure them will be mixed perfectly with the matrix/binder in the next stage. The pineapple leaf fibre then cut into short length (1 cm).

The treated short pineapple leaf fibre then lay up together with the natural matrix/binder which is corn starch. Due to the brittleness of corn starch as the matrix, corn starch was mixed with the non-toxic plasticizer that is glycerol. In this stage, several sample with the different ratio between pineapple leaf fibre and corn starch have been made. The aspect of ratio between pineapple leaf fibre and corn starch are the crucial part in this research as to determine their mechanical, physical and morphological properties. Tensile and flexural test were conducted to investigate the mechanical behaviour of pineapple leaf

fibre. A Scanning Electron Microscopy (SEM) analysis was conducted to evaluate the morphological properties. The results of this natural composite samples were compared according to the pineapple leaf fibre loading as to obtain the optimum result in mechanical properties.



1.4 Gantt Chart For PSM I & PSM II

Figure 1.1 and Figure 1.2 show the Gantt chart which included the activities performed for PSM I and PSM II.

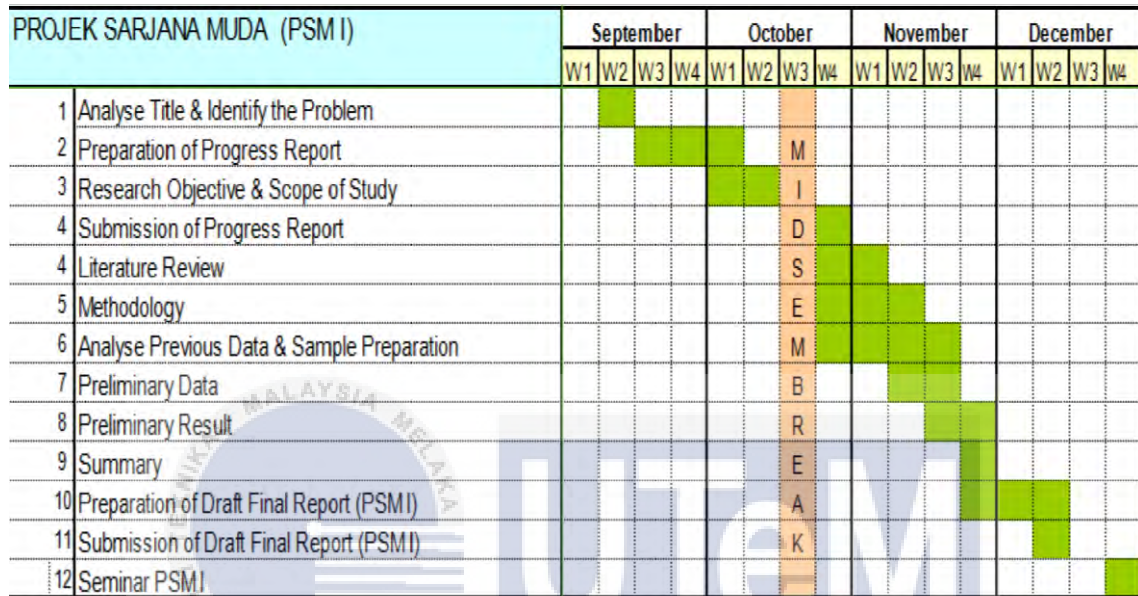


Figure 1.1: Gantt Chart for PSM I

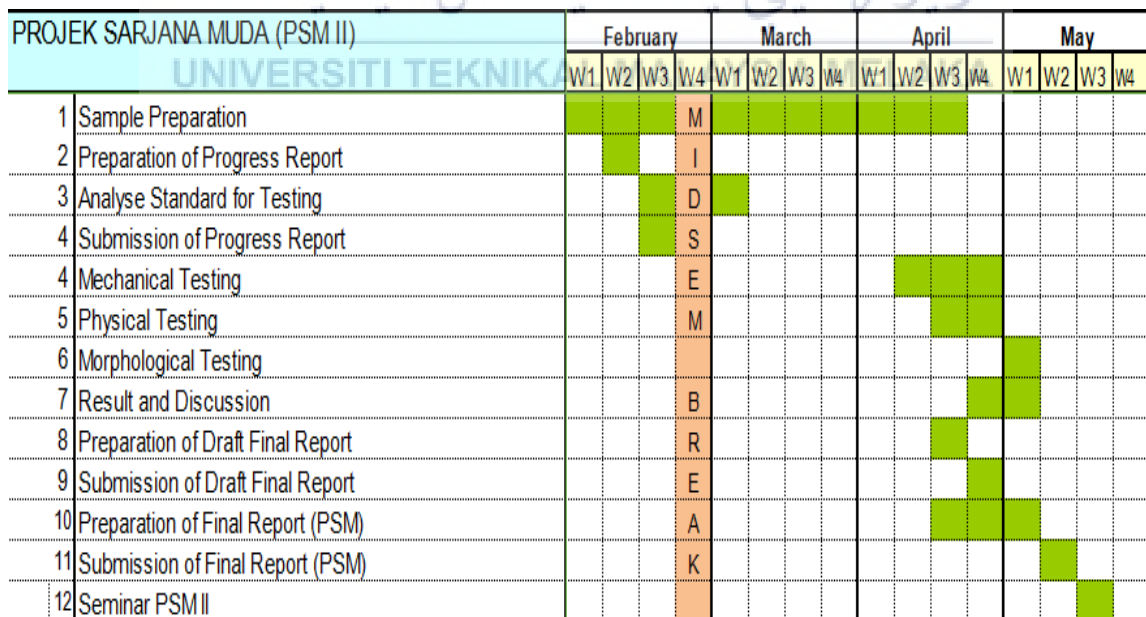


Figure 1.2: Gantt Chart for PSM II

CHAPTER 2

LITERATURE REVIEW

2.0 Composite

As thousands years ago, the function of the composite material have been noticed by the mankind. For instance, ancient brick making that combined straw and brick for building construction was the earliest man-made composite material dating back 6000 years ago. The combined material that used for building construction back then enhanced the structure of the building such as sturdier and long lasting.

Composites can be defined as material with multifunctional system that have certain characteristic which cannot be found in other discrete material. Besides that, the combination of certain material in order to perform composites are knowingly have their own unique properties. Based on the previous study, composite materials have better heat resistance compared to the other material with single component (Milanese, Cioffi and Voorwald, 2011). Composites also can be defined as individual material that consist of one or more discontinuous phases firmly fixed in continuous phase. Constituent materials is the term related to the composites and classified into two main categories; continuous phase and discontinuous phase. Continuous phase refer to the matrix that also known as binder, while reinforcement agent or filler is in term of discontinuous phase. Plasticizer is an additive material that plays an important role in order to enhance certain properties in the composites (Padmaraj *et al.*, 2013).

Recently, the mankind show their awareness for the environmental issues such as global warming, pollutions and tremendous usage of earth resources which are not good for

our lives. Many researchers are attracted to investigate and find the ways in order to cover these drawbacks. Generally, green composite is one of the major findings that is acceptable by the worldwide as it can be biodegradable, renewable resources and environmental friendly. Green composites is a biopolymer that combined and improved with natural fibre which means better for environment compared to the glass and plastic reinforced composites. Nowadays, a lot of researches focus on replacing the glass and plastic reinforced have been performed to find the material with good mechanical properties yet not harmful to environment. In the previous study, it shows that green composites such as natural fibre composites are highly recommended because of the effective cost materials, easy to get and good mechanical properties which suitable for parts of building and construction such as partition board, wall, floor and etc.(Munirah, Abdul Razak and Hassan, 2007). Composites cannot be made from component with varying linear expansion characteristics. The composites must be attached as the interface area between the matrix materials and the reinforcement. Figure 2.1 shows the classification of composites material that base on matrix and reinforcement.

Matrix material	Reinforcement Material
• Metal matrix Composite (MMC) • Polymer Matrix Composite (PMC) • Ceramic Matrix Composite (CMC)	• Structural composite (sandwich and laminate composites) • Fibre reinforcement • Particulate reinforcement

Figure 2.1: Classification of Composites Material based on Matrix and Reinforcement.

In addition, each side of the interfaces and the jointed component must consist of two interfaces while interphase occurs. Laminate is one of the example of composites material that can provides specific function by having different layers of materials with certain behaviour.

2.1 Natural Fibre

The level of awareness about environmental issues and community had risen from day to day according to the continuous production of things and products that have been used in daily life. To ensure the sustainability of the petroleum consumption, some acts and regulations have been introduce to the community as to achieve and maintain the green environmental. In Malaysia, Environmental Quality Act 1974 which is administrated by Division of Law is one of the valid act that control the pollution of the environment. Natural fibres have become as a good source for material production in common industries as it provides good properties and environmental friendly compared to the synthetic fibre. The detail of natural fibres are classified as shown in Figure 2.2:



Figure 2.2: Classification of Natural Fibres

Natural fibres are obtained from plants and animals which at their end of lifecycle are hundred percent biodegradable. As the biodegradable source, natural fibres are considered as good for environment and not harmful to human's health. To maintain the sustainability of natural resource, renewable resource such as natural fibres play an important role towards the green environment. The plants that produce cellulose fibres can be categorized into seed fibres, bast fibres, leaf fibres, core fibres, grass and reed fibres and all other kind of wood and roots (Mohammed *et al.*, 2015). Natural fibre reinforced polymer composite (NFPC) is known as an attractive source in several applications that commonly related to automotive and construction industry.

Nowadays, natural fibres have become as one of the potential replacement over common synthetic fibres such as aramid and glass that have been used in several applications previously. Natural fibres own the physical properties such as low density, less expensive, easy availability from recycle sources, which is obviously unlike synthetic fibre. Some of the examples of natural fibres; coir, hemp, flax, jute, kenaf, oil palm, pineapple leaf, and sisal that have been used widely in common applications are as shown in Figure 2.3 (a),(b),(c),(d),(e),(f),(g) and (h).



Figure 2.3: (a), (b), (c), (d), (e), (f), (g) and (h): Types of Fibres

Natural fibres also tends to show good mechanical properties and thermal properties. For instances, natural fibres have good tensile modulus and flexural modulus in term of mechanical properties, and can be easily recycle without pose any serious health hazard as it has good thermal properties. Each natural fibres have their own surface morphologies, mechanical and physical properties as shown in Table 2.1 (Pai and Jagtap, 2015). Natural fibres consist of (cellulose, hemicellulose, lignin, pectin, and waxy substances) that allow moisture absorption from surroundings that can lead to weak adhesion between fibre and polymer. The cellulose fibre produce an amorphous matrix of the hemicellulose and lignin which gained from microstructure of the natural fibres. There are several factors that can affected the properties of natural fibres such as orientation of fibres, strength of fibre, length of fibre, interfacial adhesion property of fibre, chemical treatment and etc.

Table 2.1: Mechanical and Physical Properties of Selected Natural and Synthetic Fibres
(Pai and Jagtap, 2015)

Fibre	Density (g/cc)	Moisture Content (%)	Tensile Strength (MPa)	Young Modulus (GPa)	Elongation (%)
Flax	1.5	10.0	500-1500	27.6	2.7-3.2
Hemp	1.47	10.8	690	70	2-4
Kenaf	1.45	-	930	53	1.6
Jute	1.3	12.6	393-773	26.5	1.5-1.8
Coir	1.2	8.0	593	4-6	30
Cotton	1.5-1.6	-	400	5.512.6	7-8
Bamboo	0.8	-	391-1000	48-89	-
Banana	1.35	-	529-579	8-20	1-3.5
Pineapple	1.52-1.56	11.8	413-1627	34.5-82.5	1.6
Henequeen	1.49	-	430-580	10.1-16.3	3-5
Sisal	1.5	11.0	510-635	9-22	2-2.5
Bagasse	0.55-1.25	-	20-290	2.7-17.0	0.9
S-glass	2.5	-	4570	86	2.8
E-glass	2.5	-	2000-3500	70	0.5
Aramid	1.4	-	3000-3150	63-67	3.3-3.7
Carbon	1.4	-	4000	230-240	1.4-1.8

2.1.1 Pineapple Leaf Fibre PALF

Ananas Comosus is a scientific species referred to pineapple plant. Pineapple plant is one of the most popular tropical fruit that is short lived perennial plant with height of 0.75 m – 1.25 m. Pineapple plant has short and thick stem, shallow root and waxy, succulent, sword-like leaves that can reach 50 cm -180 cm with the thick of leaf up to 0.27 cm. The leaves can be seen in variable colour, such as from uniform green to variously stripped with

red, yellow, ivory near the margin. The factor that affected the growth of the pineapple plant is the sunlight distribution and spacing. The strength of the fibre will increase as the sunlight distribution is increase. Strong refined fibre which is look alike silk is resulting from the high distribution of sunlight. Next, high demand of pineapple fruit from the worldwide lead to the huge plantation and rapid exportation of this fruit. Previously, pineapple plant is not fully utilised and has become as the agriculture waste until the researchers found that pineapple leaf can be turned into fibre that is beneficial to certain industries and applications.

This study focuses on pineapple leaf fibre functions and pineapple leaf is one of the high quality natural fibre that is useful in several applications such as plastic reinforcement, automotive and thermal and sound insulation. Nowadays, pineapple fibre leaf has become attractive in the eyes of researchers because of the potential of their characteristics and properties which is better compare to the synthetic fibre. In addition, pineapple leaf fibre consists of multicellular, lignocellulose material, and the fibre can be seen as ribbon-like structure that bind together with lignin, pentosan-like material under optical microscope (Munirah, Abdul Razak and Hassan, 2007). Pineapple leaf fibre has been used widely because the fibre own good mechanical properties and shows superior properties based on the high cellulose content and low microfibrillar angle (Kengkhetkit and Amornsakchai, 2012). The specific modulus and strength of pineapple leaf fibre when divided by density are obtained to be close or even higher than glass fibre. Hence, the implementation of the pineapple leaf fibre can replace the other synthetic fibre and give significant changes towards the effort of reducing oil-derived product which is quite expensive.

2.1.2 Extraction Method of PALF

Extraction method is defined as action of separate something that require effort or force. This term is use to extract pineapple leaf which known as one of plant source that contribute huge amount in agriculture waste. As the pineapple leaf has the potential uses in certain industries and application according to the properties of its fibre; extraction method is the first step to obtain this useful fibre. Extraction method is important as to separate the gummy substance from the pineapple leaf fibre and eliminate the water that contain in the leaf until the thread like fibre produced. Time is one of the parameter that must be consider during fibre extraction because chemical in plant leaves degraded after stalk has been remove. The fibre will be harder to extract as the lengthy of time taken for the pineapple leaf to undergo the extraction method. This is because the gummy resin or sap substance will increase the adhesion between pulp and leads to difficulty during fibre extraction. Extraction method of pineapple leaf can be done in two different ways which are known as manual process and mechanical process. Manual method consist of immersion process which is done by soaking the pineapple leaf into water for a certain period. Pineapple leaf contains of microorganism which produced the gummy substance inside the leaf and that need to be remove. The function of the gummy substance is to make fibre decompose and fibre inside the leaf can be separate for each other easily. Manual method is conduct by extracting the pineapple leaf on long bench by using scrapping tools that namely by 'ketam'. The procedure of manual extracting is shows in Figure 2.4 (Adam and Yusof, 2015).

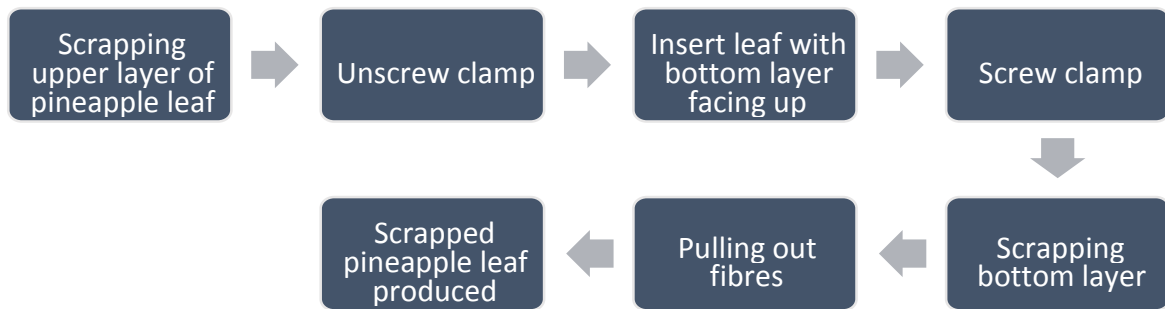


Figure 2.4: Manual Extracting Method (Adam and Yusof, 2015)

Next, mechanical method can be conducted using the Pineapple Leaf Fibre Machine to separate the fibre from the leaf. Pineapple Leaf Fibre Machine consists of two blades to remove waxy layer on pineapple leaf fibre instead of forcing by crushing process that usually done by the other machine such as decorticator or crusher. Figure 2.5 shows the Pineapple Leaf Fibre Machine and Figure 2.6 shows the mechanism of Pineapple Leaf Machine. Damage of fibre can be avoid by using the Pineapple Leaf Fibre Machine as this machine is not crushing the leaf (Adam, Yusof and Yahya, 2016). Mechanical method of extracting pineapple leaf fibre shows in Figure 2.7.



Figure 2.5: Pineapple Leaf Fibre Machine

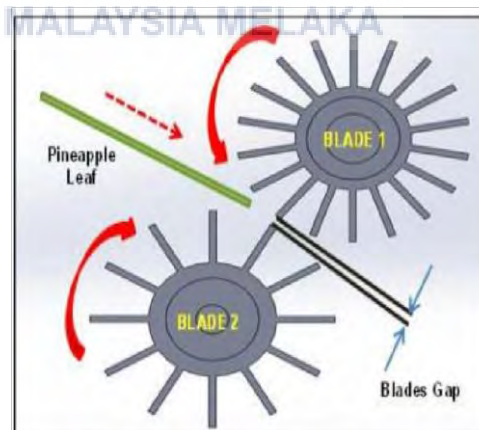


Figure 2.6: Mechanism of Pineapple Leaf Fibre Machine

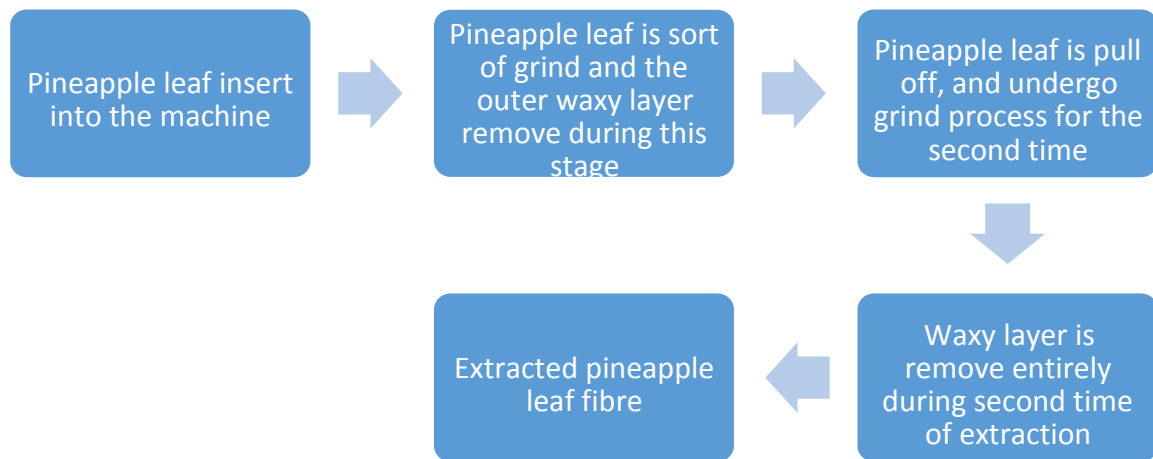


Figure 2.7: Mechanical Extracting Method

There are a few comparison can be made between these two methods; manual method and mechanical method. These two methods were tested by the previous researcher according to the performance of extracting pineapple leaf and the test was conducted for eight hour of working. The results of the performance between two methods is shown in Figure 2.8.

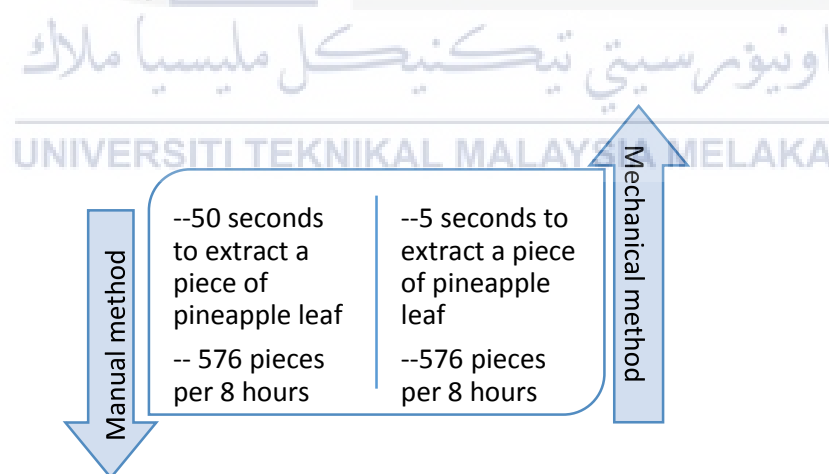


Figure 2.8: Comparison of Performance between Manual Method and Mechanical Method

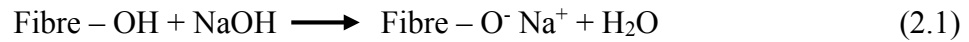
Based on the comparison above, mechanical method is more effective as the production rate of fibre is 10 times larger than manual method. It is important to consider about time efficiency as to enhance good labour management and control the production cost at once.

Since the Pineapple Leaf Fibre Machine has been introduced, the agriculture waste produced from pineapple leaf can be minimised. Hence, the sustainable of the environment can be thoroughly developed.

2.1.3 Alkaline Treatment of PALF

Alkali treatment is one of the commonly used to treat surface of pineapple leaf fibre and the other treatments that can be used such as silane, acrylation, benzene, peroxide and etc. Based on the previous research, alkali treatment can remove lignin, wax, oil covering part of pineapple leaf fibre and increase roughness of surface fibre (Sood and Dwivedi, 2017). Alkali treatment on pineapple leaf fibre is important since the water absorption can be reduce after that treatment process. The untreated pineapple leaf fibre has higher water absorption compared to the alkali treated pineapple leaf fibre. For instance, the outer layer of untreated pineapple leaf fibre has high potential to absorb more water and leads to gradually decrease into bulk of matrix. Nowadays, pineapple leaf fibre has demanded highly by composite industries because of their good properties. However, composite material performance can be affected regarding to the high water intake by the untreated pineapple leaf fibre (Supri and Lim, 2009). Several drawbacks of the untreated pineapple leaf fibre that related to composites such as the weight of wet profile increased, strength reduced, increment in deflection, swelling and caused pressure on nearby structure (Mohammed *et al.*, 2015). Besides, untreated pineapple leaf fibre can leads to wrapping, buckling, microbial inhabitation that can reduce mechanical properties of the fibre itself.

During alkali treatment, surface of fibre is cleaned as to ensure no impurities that can increase fibre surface roughness. Alkali treatment also prevent moisture absorption via removal of hydroxide, OH^- groups of fibre that refers to Eq. (2.1):



Furthermore, alkali treatment consist of major advantages such as can improve adhesive characteristics of fibre surface and provide large surface area that leads to fibre-matrix enhancing on interfacial bonding and stress transferability of the composite (Yusof, Yahya and Adam, 2014). Pineapple leaf fibre that treated by alkali tends to have good surface topography, thermal stability and tensile strength morphology. Moreover, as elongation of the treated pineapple leaf fibre decrease, composite become stiffer and stronger but less flexible. Based on the previous study, pineapple leaf fibre that has been treated using 6% of NaOH is 25% higher in term of flexural strength than untreated pineapple leaf fibre (Sood and Dwivedi, 2017). Alkali treatment also can improve compatibility, properties, dimensional stability and can avoid fungal decay. Previous preparation of alkali treatment for pineapple leaf fibre is shown in Figure 2.9 below (Nopparut and Amornsakchai, 2016):

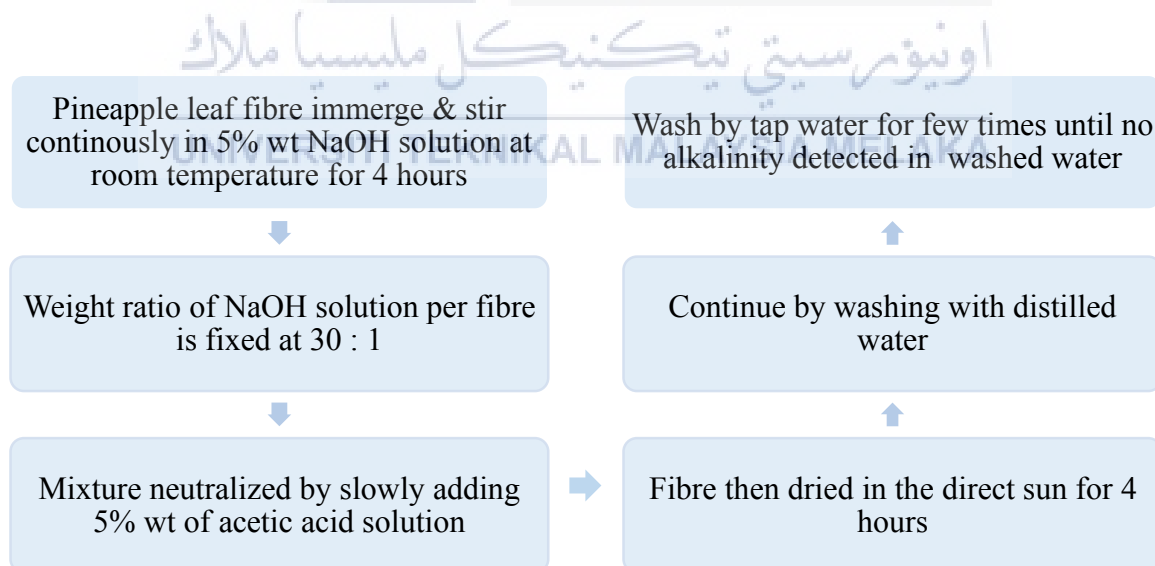


Figure 2.9: Alkaline Treatment Process for Pineapple Leaf Fibre (Nopparut and Amornsakchai, 2016)

2.2 Types of Composites for PALF

Pineapple leaf fibre has been as a great choice by composite industries compared to the synthetic fibre. Cultivation of pineapple can be anywhere with the hot and dry season and every year there are tons of pineapple waste produced. With the improvement of technology and researches, pineapple waste obtained from the leaves nowadays can be turn into fibre which is beneficial in common industries especially composites. Pineapple leaf fibre as a part of composite has showed many advantages over the other fibre materials in term of less weight, biodegradability and mostly low in price.

As for automotive industries, the natural fibre reinforced polymer composite are widely used as the material for mechanical parts. There are friction occurs all the time due to relative movement of plastics mechanical parts that leads to wear and affected their service life. Based on the previous research, the factors that related to the friction properties of polymers and wear are selection of material couple such as (polymer-polymer contacts or non-polymer-polymer contacts), crystallinity, surface treatment of part and surface roughness (Běhálek, Lenfeld and Seidl, 2012).

Polymer is one of the material that used extensively in natural fibre reinforced composite production. Polymer refers to long chain molecule that consist of large number of repeating units in identical structure. Polymeric composites can be produced by combining fibre and polymer resin which also known as fibre reinforced plastic (FRP) (Munirah, Abdul Razak and Hassan, 2007). Combination between these two materials also known as bio-composite. Polymer can be classified into two groups that related to thermal processing behaviour which are known as thermoset and thermoplastic. Traditional plastic material reinforced by glass fibre normally expensive and can give bad effect to the environment. Thermoset and thermoset are usually used in the natural composite industries and also used as matrices for pineapple leaf fibre composite. Some advantages of pineapple

leaf fibre reinforced polymer composites are low cost, low density and low energy consumption. Besides, pineapple fibre that consist in the composite can make the material partially biodegradable.

2.2.1 Thermoset

Thermoset can be classified into polymer with individual chain that is chemically link by covalent bonds during polymerization either by subsequent chemical and thermal treatment. After undergo the polymerization process, crosslink network will resist heat softening, creep and solvent attack. Thermoset has widely used in certain industries such as composites, aeronautic, automotive and so on. Thermoset at room temperature can be in either liquid form or solid form and that place into a mould and then heated to harden preform into desire shape and solid properties.

Thermoset is known as a polymer with good stability that is applicable for certain applications. Epoxy, polyester and formaldehyde are some examples of thermoset. Nowadays, thermoset has been used as matrix for certain reinforced composite and material. For instance, there are several study performed on the effect of properties for pineapple leaf fibre and polyester as reinforced composite. The result obtained is elastic modulus of polyester composite increase as volume fraction of pineapple leaf fibre increase. However, when tested with epoxy matrix, flexural strength decrease as pineapple leaf fibre cannot be combined together and leads to crack propagation (Glória *et al.*, 2017). Table 2.2 shows the relation between volume fraction of pineapple leaf fibre and flexural strength with polyester.

Table 2.2: Previous Flexural Strength Result based on Thermoset Matrix with PLF (Glória *et al.*, 2017)

Volume fraction (%)	Weibull modulus, β	Characteristic flexural strength, Θ (MPa)	Precision adjustment, R^2	Average flexural strength (MPa)	Statistical deviation (MPa)
0	2.394	26.93	0.9191	27.72	7.22
10	2.766	35.99	0.9635	36.22	7.13
20	3.382	77.99	0.9344	75.61	11.02
30	2.289	101.52	0.9430	103.25	13.31

Thermoset is made from polymer resin that have the ability to form chemical crosslink. Thus, as the number of crosslink increase, the stiffness will be increase and become less brittle. Besides, the impact toughness can be increased by adding reinforcement or filler. Thermoset have drawback which is cannot be reshape or in other word it cannot be recycle (Ourdjini, 2005). Even though thermoset can produce excellence adhesion and mechanical properties in term of chemical, electrical and resistance to heat, most of epoxy resin and hardeners used are derived from oil resource that can be harmful to environment (François *et al.*, 2017).

2.2.2 Thermoplastic

In this globalization era, thermoplastic composite has been widely used with the reinforcement of the natural fibre such as kenaf, jute, banana, pineapple and many more for several applications and industries. Atom bond by covalent bonding in thermoplastic gives results of high strength, high stiffness, retention of mechanical properties yet also can withstand to such environmental factor like water, solvent and chemical. Thermoplastic is one of the new class of material that have good potential in certain applications such as for consumer goods, furniture, low cost housing, structures and buildings (Munirah, Abdul

Razak and Hassan, 2007). Polyethylene (PP), Polypropylene (PE), Polystyrene (PS), Polyvinylchloride (PVC) are the examples of the fraction under polymer class which can be melt by variety of method such as extrusion and moulding.

Nevertheless, lack of good interfacial adhesion and poor resistance to moisture make thermoplastic become less attractive in the eyes of the researchers. In previous study, Polypropylene (PP) has chosen as matrix because it has low processing temperature which is 230°C and below, that will not degrade the fibre yet has low density, high crystalline and high stiffness and hardness. The benefit of this type of thermoplastic is own low processing temperature that can be recycle. Typically, thermoplastic act poorly in term of loading because linear polymer molecule require strong time and temperature dependent response (Farsi, 2000). With the development and improvement in composite field, continuous natural fibre reinforced thermoplastic material are expected to take place of inorganic fibre reinforce thermosetting materials.

Besides, one of the advantage of thermoplastic is can be reshape as it has low processing temperature and leads to easy to recycle. On the top of the advantage, thermoplastic also has several drawbacks. Thermoplastic has high hydrophilic nature of natural fibre that leads to similarity problem with hydrophobic thermoplastic in composite. Mechanical properties of composite is based on interfacial adhesion between composite. Since natural fibre has low strength, it is difficult to saturate with thermoplastic resin into reinforcement because of high melt viscosity ('Biodegradable Thermoplastics As Matrices for Natural', no date). Biodegradable polymer and matrix and natural fibre as reinforcement are the first step to be commercialized into common application in current industries.

2.3 Force Biodegradable Resin (Corn Starch)

Currently, rapid development is happening around the globe and there are a lot to do to maintain the green environment as to ensure safety and health of life being. Researchers are continuously study about material such as composites that can be used in applications and industries without increasing environmental issues. One of the attractive source of raw material that can be used for development of green composite is starch. Starch is a white substance that is odourless, tasteless and also namely as polysaccharide polymer of D-anhydro-glucose reiterate units that consists of two main element; amylose and amylopectin (Wattanakornsiri and Tongnunui, 2012).

Main properties of starch is it can be hydrolysed by microorganism and enzyme into glucose that will metabolized into carbon dioxide (CO_2) and water (H_2O). Next, starch which is classified into carbohydrate organic compound also naturally renewable which means it can totally biodegradable in wide variety of environment. According to the previous study, flexural strength of composite will increase as the starch content is increase, but decrease when mass fraction of starch is above 10% (Liu, Jia and He, 2012).

One of disadvantages of starch is composite that using starch as natural polymer cannot be melt because as it melts pyrolysis will occurs before crystalline melting point of starch is reached. Hence, conventional plastic equipment cannot be used for starch-composite material process. Next, starch contains hydroxyl groups which indicates hydrophilic properties that is low resistance to water and tends to absorb more water that lead to decline the mechanical properties. Starch can loses the good properties when exposed to water that caused by amylose which is dissolved in water while amylopectin is swelled in presence of water.

Nowadays, starch has been converted to thermoplastic starch (TPS) that is developed by addition of plasticizer. The function of thermoplastic starch (TPS) as matrix is better than

starch in term of properties such as better water resistance, acoustic insulation properties, high internal bonds strength, flexibility and flexural strength (Gironès *et al.*, 2013). Besides, the uses of formaldehyde resin and polymer as matrix of composite preparation can improve certain properties as mentioned above. However, matrix like that are difficult to degrade because it is not fully natural.

2.4 Plasticizer (Glycerol)

Plasticizer is a material that can be formed into plastic material with high flexibility and applicability of plastic material. The function of plasticizer such as glycerol is become as an additive that can increase plasticity and fluidity of material to which they are added. According to the previous study, by adding the glycerol to the starch matrix it can decrease absorption of water in starch and prevent material from become fragile (Nafchi *et al.*, 2013). Even though glycerol is added to starch matrix, the mechanical properties such as elongation and strength of composite are still remained the same.

Due to hydrogen bond formation with glycerol, the direct interaction between starch and chains were partly reduced and the rigidity of material (slope of elastic zone) is decreased as the glycerol content is increased. Figure 2.10 shows some stress- strain curves obtained for high amylose corn starch films plasticized with different glycerol concentrations (g glycerol/g high amylose corn starch) (Nafchi *et al.*, 2013).

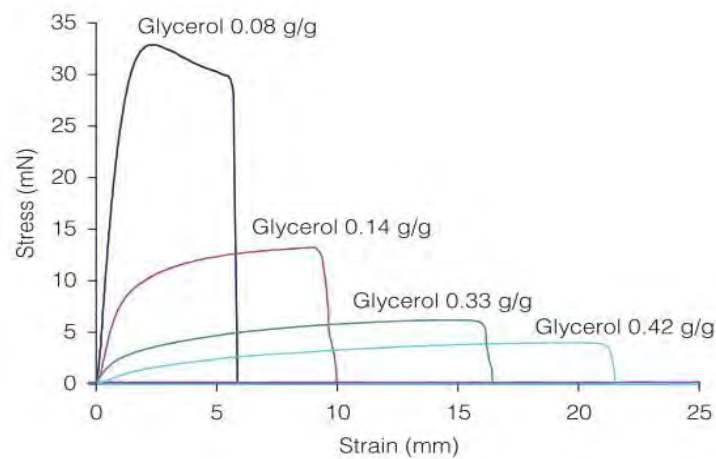


Figure 2.10: Stress-Strain Relation of Glycerol Content (Nafchi *et al.*, 2013)

For viscoelastic material, these curve seems to be typical. The stress increased rapidly and was proportional to the strain at the beginning; which is the elastic reversible strain area (elastic deformation). Plastic flow started as the yield point was obtained and the strain was no longer reversible. This part is called the plastic strain area.

CHAPTER 3

METHODOLOGY

3.0 Overview of Methodology

Several steps will be done in completing the research. Figure 3.1 shows the steps that need to be done as to accomplish the study and obtain the desire results.

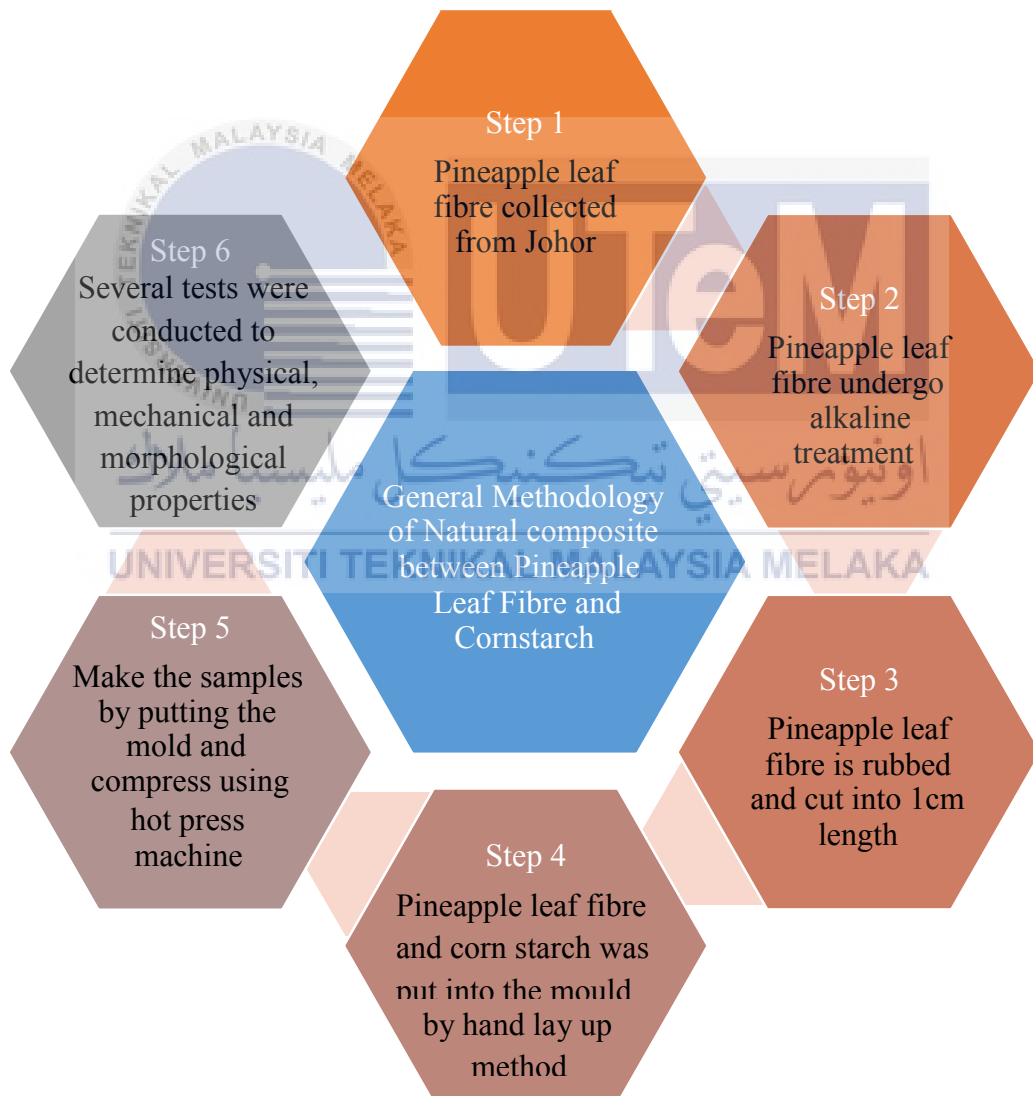


Figure 3.1: Overview of Methodology of Natural Composite between Corn Starch Reinforced Alkaline Treated PALF

3.1 Flowchart of Methodology

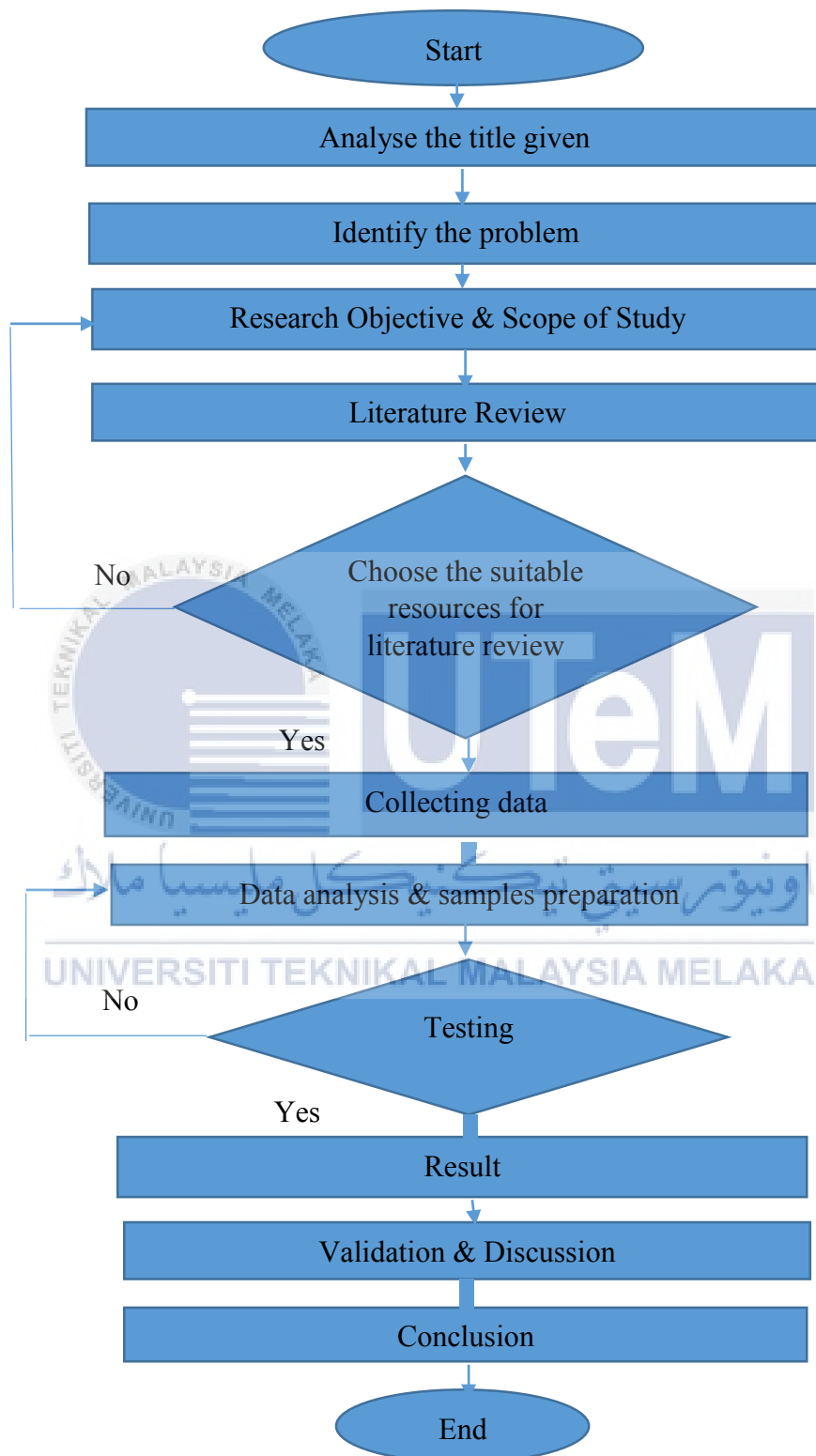


Figure 3.2: Flow Chart of Methodology

3.2 Sample preparation of Composites

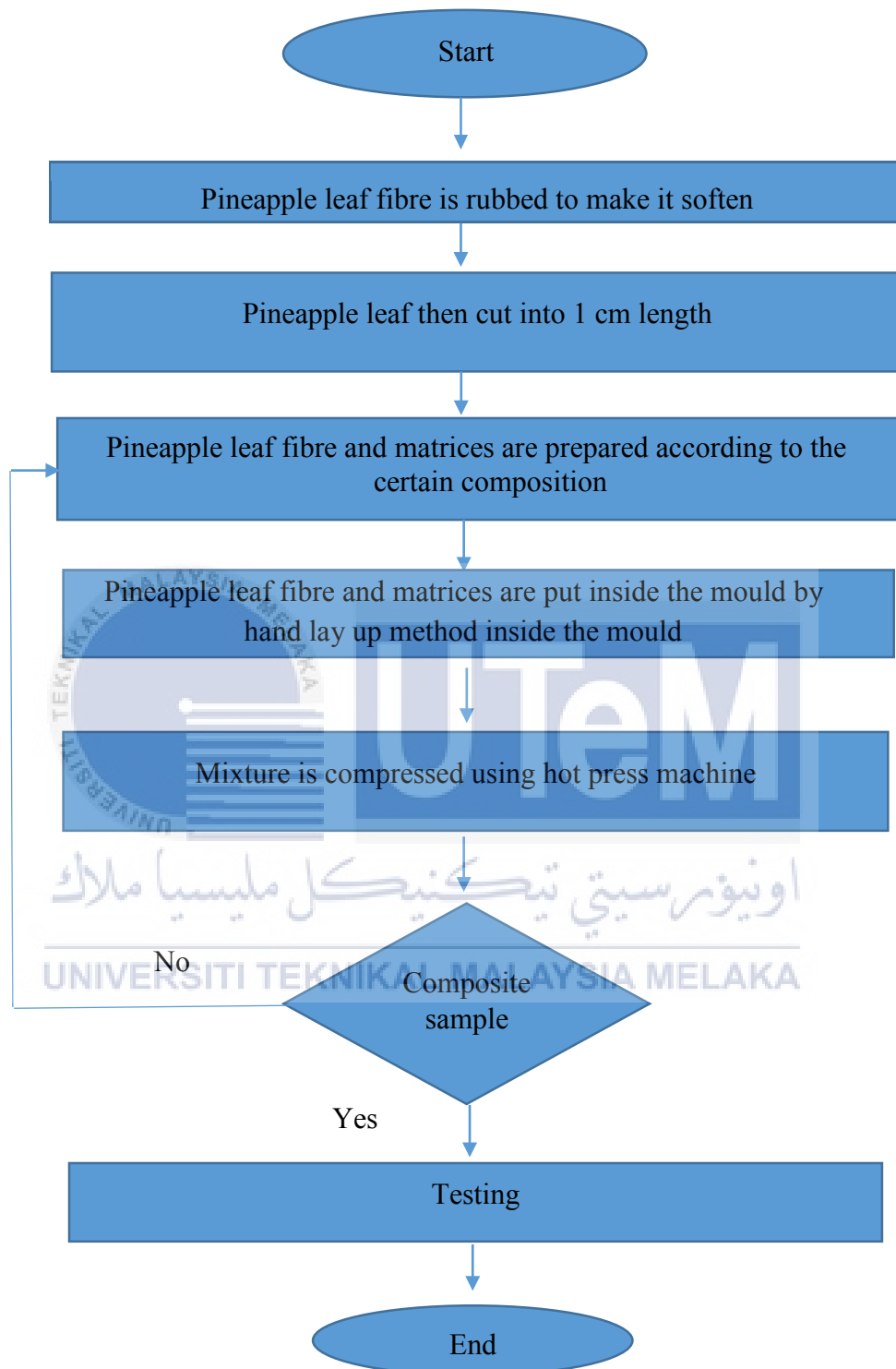
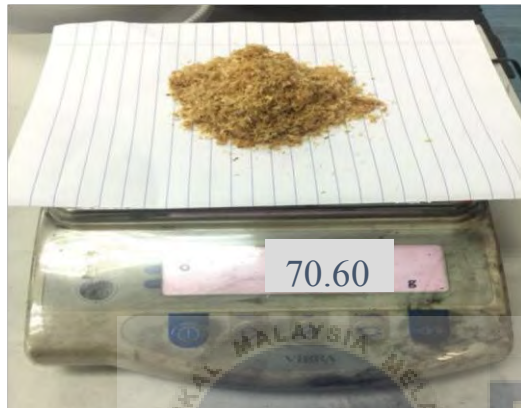


Figure 3.3: Flow Chart of Sample Preparation for Corn Starch Reinforced Alkaline Treated PALF Composites

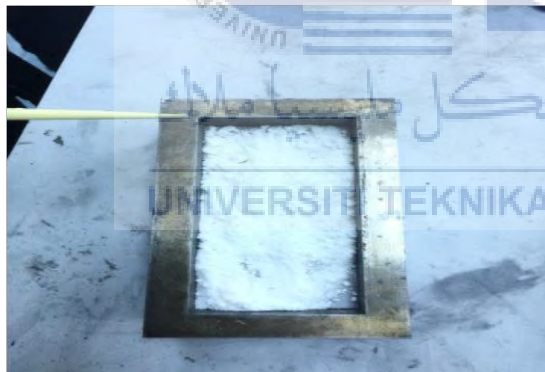
In this study, the sample of natural fibre reinforced composite is made from hand lay up method between the pineapple leaf fibre with corn starch. Pineapple leaf fibre and corn starch are prepared according to the composition that has been stated in the next sub-topic. Full process of the sample preparation of composite are shown in Figure 3.4 (a), (b), (c), (d), (e), (f), (g), (h) and (i).



(a)



(b)



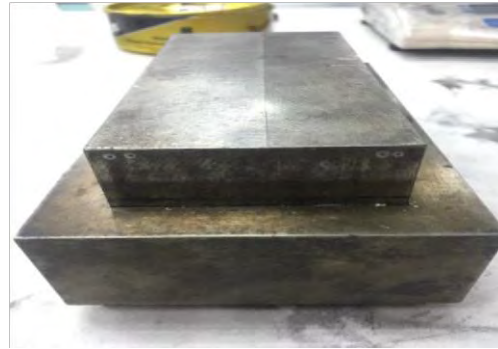
(c)



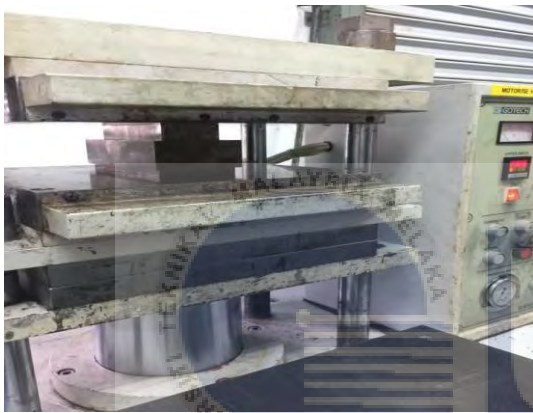
(d)



(e)



(f)



(g)



(h)

اونيورسيتي تيكنيكل مليسيا ملاك
UNIVERSITI TEKNIKAL MALAYSIA MELAKA



(i)

Figure 3.4 (a), (b), (c), (d), (e), (f), (g), (h) and (i): Process of Sample Preparation

3.2.1 PALF preparation

Pineapple leaf fibre (PALF) is the most important part in this research as it will be a part of the samples of natural fibre composite. Pineapple leaf fibre was collected in Johor and already undergo alkaline treatment and cured. Next, pineapple leaf fibre was scratched as to make the fibre soften for easy mixing with matrix during sample preparation and then will be cut into 1 cm of length. Figure 3.5 (a), (b), (c), (d) and (e) show the preparation of PALF:

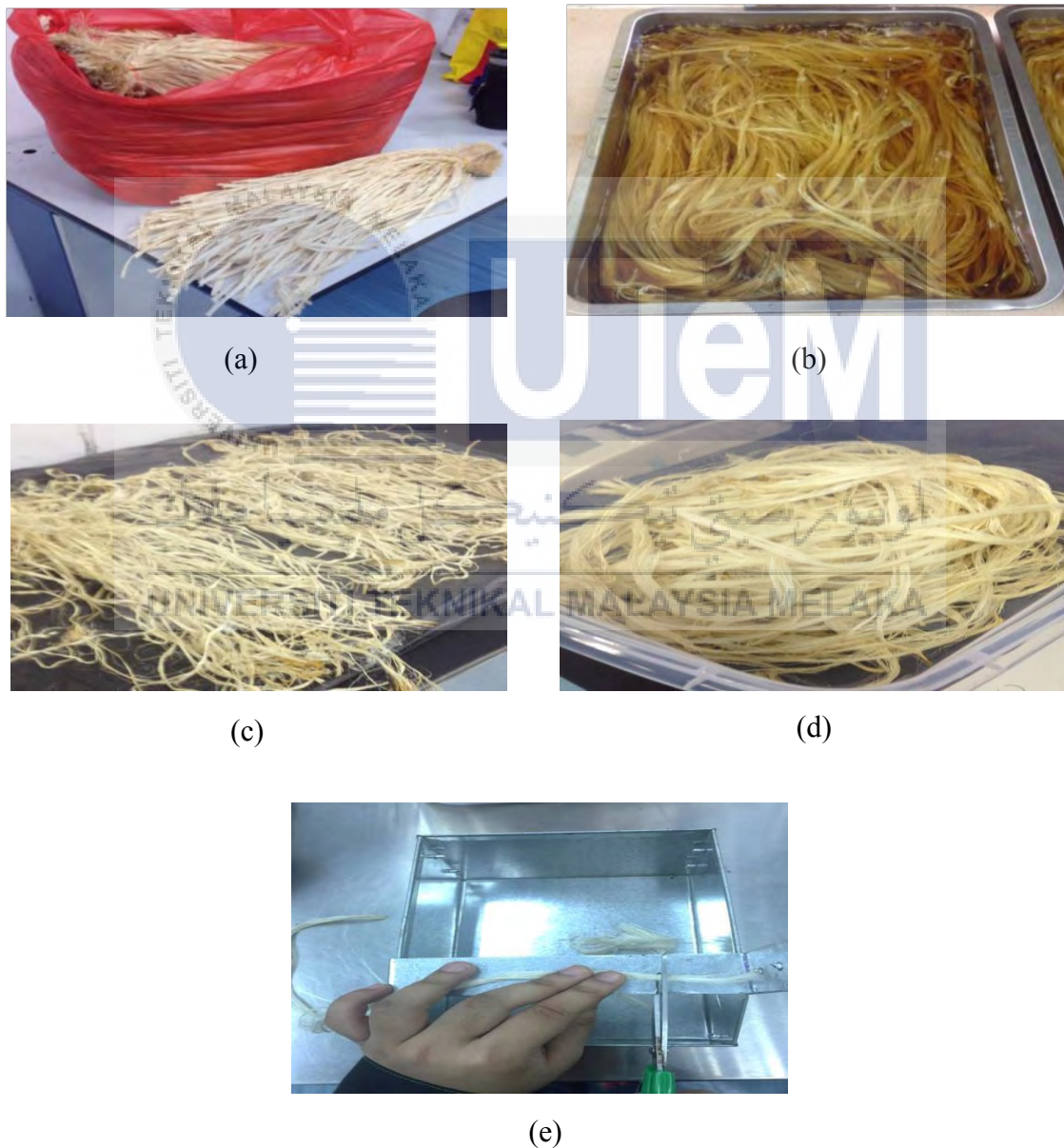


Figure 3.5 (a), (b), (c), (d) and (e): Preparation of PALF

3.2.2 Matrices Preparation

Corn starch is the matrix agent that will be used in the natural fibre reinforced composite making process. Corn starch has the tendency to absorb moisture based on the hydrophilic properties. Therefore, corn starch will be mixed with the additive that is known as glycerol to reduce the ability to be wetted by moisture. Matrices preparation are show in Figure 3.6 (a),(b), (c) and (d).



Figure 3.6 (a),(b), (c) and (d): Matrices Preparation

3.2.3 Composition of Sample

The composites samples will be produced according to the certain compositions. The sample is produced by mixing the pineapple leaf fibre with matrix that has been used in this research which is corn starch with the addition of glycerol additive. Next, the PALF will be lay up with corn starch into the mould based on the chosen compositions. The composition in term of fibre loading (wt%) of pineapple leaf fibre is shown in Table 3.1:

Table 3.1: Composition of Sample to be Tested

Fibre loading (weight percentage)	
	5
	10
	20
	30
	40
	50
	60
	70

3.3 Sample Testing

Testing is the key role in determining the properties and behaviour of the sample. In this research, there are several testing that need to be conducted as to obtain the properties of the samples in term of physical, mechanical, and morphological testing. Table 3.2 shows the detail of testing that will be conducted on the composite samples.

Table 3.2: Sample Testing Method

Physical Testing	Mechanical Testing	Morphological Testing
Density	Tensile	Scanning Electron Morphology (SEM)
Water Absorption	Flexural	
Moisture Content		

3.3.1 Determination of Moisture Content

Eight samples of each compositions were cut into 10mm (l) x 10mm (w) x 3mm (t) as to evaluate the moisture content. Natural fibre is known as hydrophilic in nature, so the moisture absorption in PALF is highly related to the humidity of air. For instance, higher humidity caused by the higher moisture content in those fibre. The standard that are referred is ASTM D5229 (Method, 1998). Moisture content in PALF were determine by the percentages according to the Eq. (3.1). The samples were heated in the oven at temperature of 105°C for 24 hours. The weight of the samples were taken before being heated in the oven is denoted as M_0 . After being heated for 24 hours in the oven, the samples were weighed again to get the mass and denoted as M_1 . Hence:

$$\text{Moisture Content (\%)} = \frac{M_1 - M_0}{M_0} \times 100 \quad (3.1)$$

where

M_0 is the mass of PALF sample before heated in the oven

M_1 is the mass of PALF sample after heated in the oven

Figure 3.7 shows the samples that is heated in the oven with the temperature of 105°C for 24 hours.



Figure 3.7: Samples that Heated at 105°C in the Oven for 24 Hours

3.3.2 Determination of Water Absorption

Eight samples from each compositions were prepared in the dimension of 10mm (l) x 10mm (w) x 3mm (t). The absorption of PALF samples were determined by using Eq. (3.2). Next, the value percentages of water absorption were calculated by taking the average values. The mass of the samples before being immersed in distilled water for two hours and also for half an hour were labelled as M_0 . After being immersed in the distilled water for two hours and also for half an hour, the samples were weighed again and denoted as M_1 . Therefore:

$$\text{Water Absorption (\%)} = \frac{M_1 - M_0}{M_0} \times 100 \quad (3.2)$$

where

M_0 is the mass of PALF sample before immersed in distilled water

M_1 is the mass of PALF sample after immersed in distilled water

Figure 3.8 shows the samples that being immersed in distilled water for two different times which are for two hours and also for half an hour.



Figure 3.8: Samples that Immersed in Distilled Water for Two Hours and for Half an Hour

3.3.3 Measurement of Density

The density of PALF was evaluated by using electronic densimeter. Firstly, PALF samples for each composition were cut into 10mm x 10mm x 3mm size. Then, the mass of samples before immersed into the water were weighed and denoted as (m). The amount of water before and after the immersion of the samples is stated as volume, (V) and these two parameter are used to determine the density of PALF samples as shown in Eq. (3.3).

$$p = m/V \quad (3.3)$$

where

p is the density of PALF

m is the mass of the PALF

V is the volume of water

Figure 3.9 represents the device use for measuring density of PALF samples which is electronic densimeter.



Figure 3.9: Electronic Densimeter for Measuring Density of PALF Samples

3.3.4 Tensile Test

Tensile test is one of the simple and common test that is used to determine the mechanical properties of natural fibre. There are certain properties in term of mechanical that can be obtained from tensile test such as tensile stress, tensile strain, maximum elongation, yield stress and Young's Modulus. The tensile properties of PALF samples were taken using Universal Testing Machine, Instron as shown in Figure 3.10 (a). For this tensile test, ASTM D3039 that stands for Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials was taken as reference for PALF samples (Jumaidin *et al.*, 2017). At least five samples were tested. The sample was held vertically by pneumatic holder as shown in Figure 3.10 (b), and the samples were marked at the centre as to indicate that the fracture occur within the line drawn as shown in Figure 3.10 (c). Load was applied until rupture occurred on the sample and the values of tensile strength were recorded in Bluehill 2 Software. 1mm/min of load speed was applied while conducting this test. The samples to be tested were set into dimension of 140mm (l) x 25mm (w) x 3mm (t).

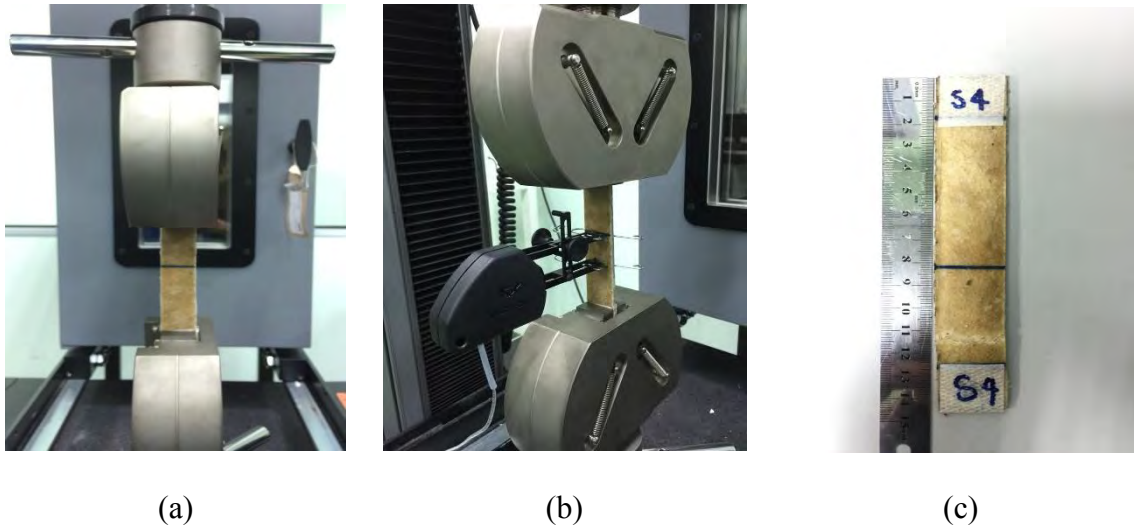


Figure 3.10 (a), (b) and (c): Tensile Test of PALF, Position of Pneumatic Holder and Sample Used

The failure area of the fibres were determine by considering the cross-sectional area and was calculated as Eq. (3.4) follows:

$$A = \Pi r^2 \quad (3.4)$$

where

A is the area of sample

r is radius of sample

Next, the tensile strength of the PALF can be calculated manually by using Eq. (3.5):

$$\sigma_f = F/A \quad (3.5)$$

where

σ_f is the tensile strength of the fibre (Pa)

F is the maximum force at break (N)

A is the area of fibre cross section (m²)

3.3.5 Flexural Test

In this study, Figure 3.11 has shown the flexural tests that were conducted by using a three-point bending setup according to the ASTM D7264 standard (ASTM, 2007). At least five specimens were tested for this flexural method. The dimension of the samples is 140 mm (l) x 13 mm (w) x 3mm (t) were tested using a Universal Testing Machine, Instron and the results were determined and recorded in Bluehill 2 Software. The load speed applied for this test is 1mm/min. The sample was placed on the gauge and the head was adjust to touch the surface of the sample and the load was applied onto the sample as shown in Figure 3.11 (a) and (b).

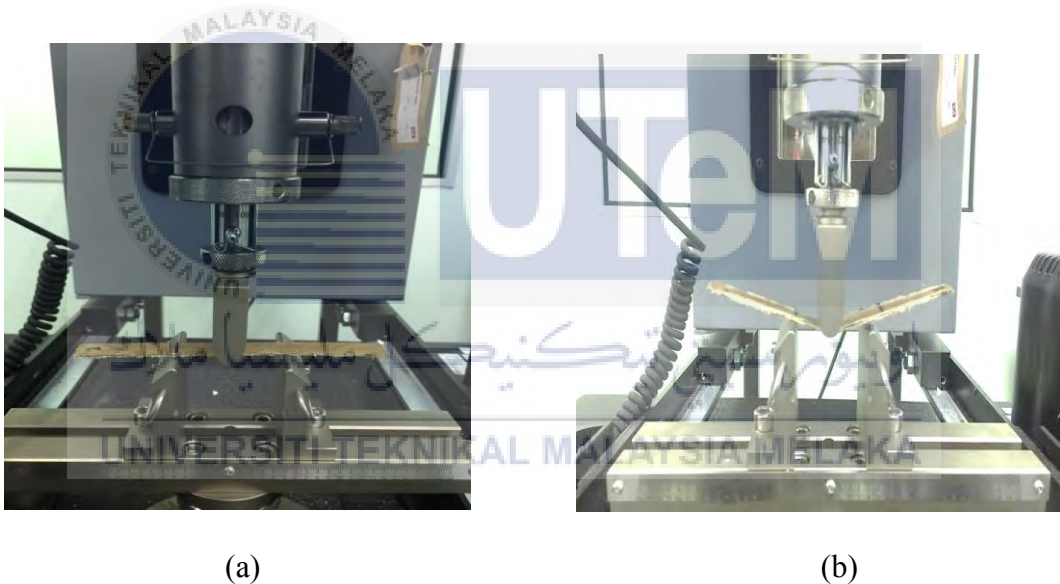


Figure 3.11 (a) and (b):Flexural Test Method

3.3.6 Morphological Test

Morphological studies is performed to determine the surface of sample after undergo tensile test which referred to the surface of fracture area. In this study, Scanning Electron Microscopy (SEM) was used to obtain the morphological properties under 30 times of magnification. Three samples that consist of 10 wt%, 60 wt% and 70 wt% of fibre loading were selected to undergo morphological test. Limited or non-conductive material sample need to be coated with conductive layer of metal which consist of electron microscopy as to enable and enhance the sample imaging resolutions. Figure 3.12 (a), (b) and (c) shown the Scanning Electron Microscopy, samples placement and sample coating machine, respectively.

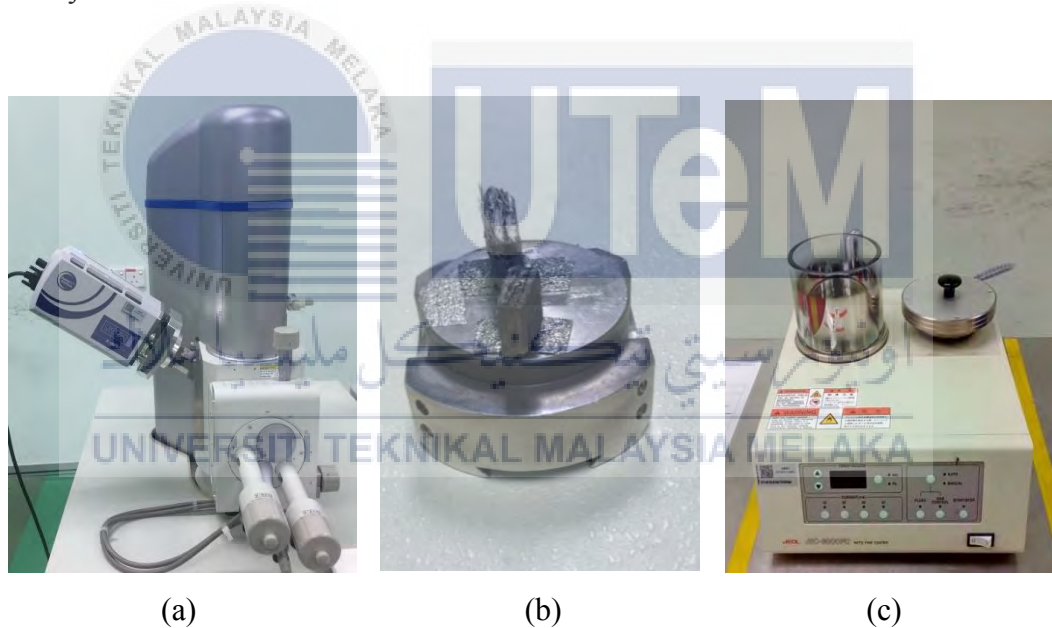


Figure 3.12 (a), (b) and (c): Scanning Electron Microscopy, Sample placement, and Sample Coating Machine

3.4 Equipment and Machine

There are several equipment and machine that have been used in this study to produce the sample of natural fibre reinforced composite. Figure 3.13 (a), (b), (c) and (d) show the equipment and machines used during sample preparation:



Figure 3.13 (a), (b), (c) and (d): Equipment and Machine Used

CHAPTER 4

RESULTS & DISCUSSION

4.0 Results of Samples

In this chapter, the results obtained will be discussed in detailed. The fibre composition of PALF is ranging from 5 to 70 with the average increment of 10 as shown in Table 4.1.

Table 4.1: Composition of Sample to be Tested for Tensile, Flexural and Morphological

Test	Fibre Loading (weight percentage)
	5
	10
	20
	30
	40
	50
	60
	70

There are several samples of composite that have been produce according to the compositions chosen. The examples of samples that have been produced are compositions of 95/5, 90/10, and 80/20 as shown in Figure 4.1 (a), (b), and (c).

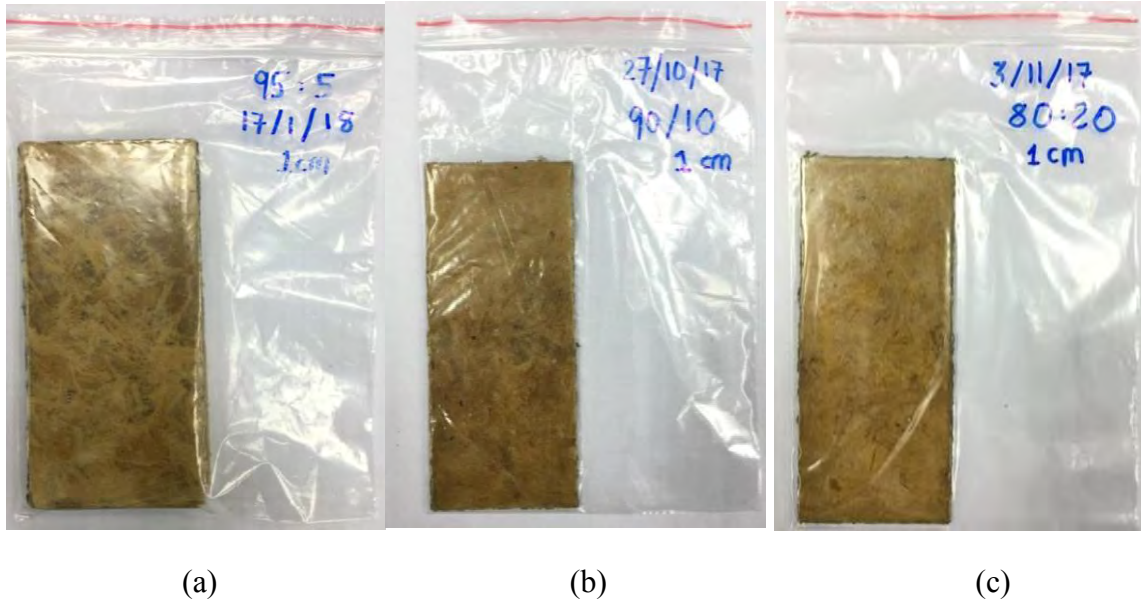


Figure 4.1 (a), (b) and (c): Examples of Compositions of the Samples to be Test

The sample of composite produce in this study has the fix dimension with length of 140 mm, wide of 60 mm and thickness of 3 mm. Figure 4.2 (a) and (b) show the length and wide of the sample

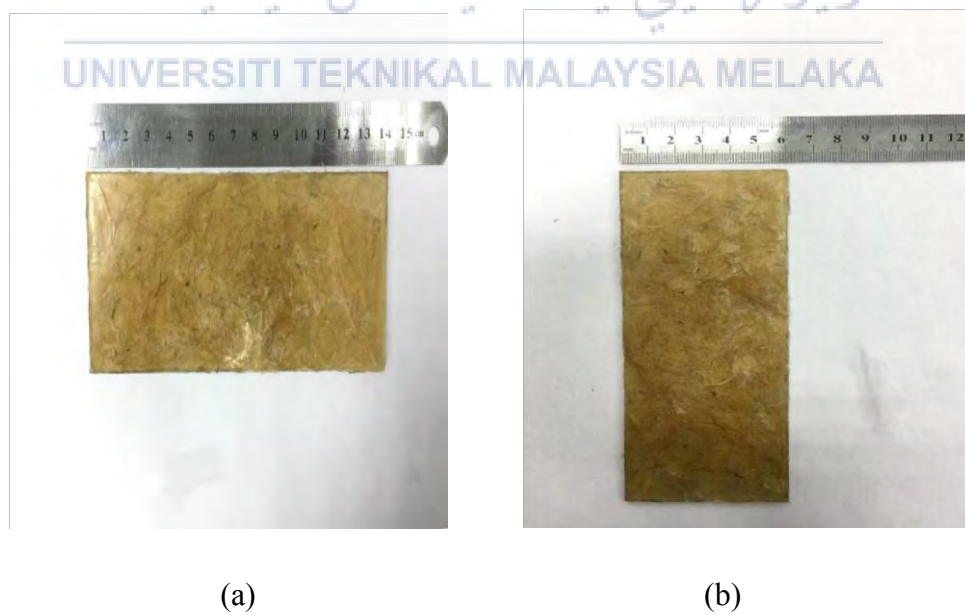


Figure 4.2 (a) and (b): Length and Wide of Sample

4.1 Physical Results

4.1.1 Moisture Content

Moisture content can be defined as the quantity of water contained in a material. Determination of moisture content is based on the principle of thermogravimetric process as the material loses weight after being heated. Surface treatment is one of the most crucial part as to enhance the properties existing natural fibre; PALF. Basically, PALF is categorized into natural fibres with hydrophilic behaviour which tends to have the problem in order to achieve good adhesion between reinforcement and matrix. Figure 4.3 shows the results of moisture content in corn starch reinforced alkaline treated PALF composites.

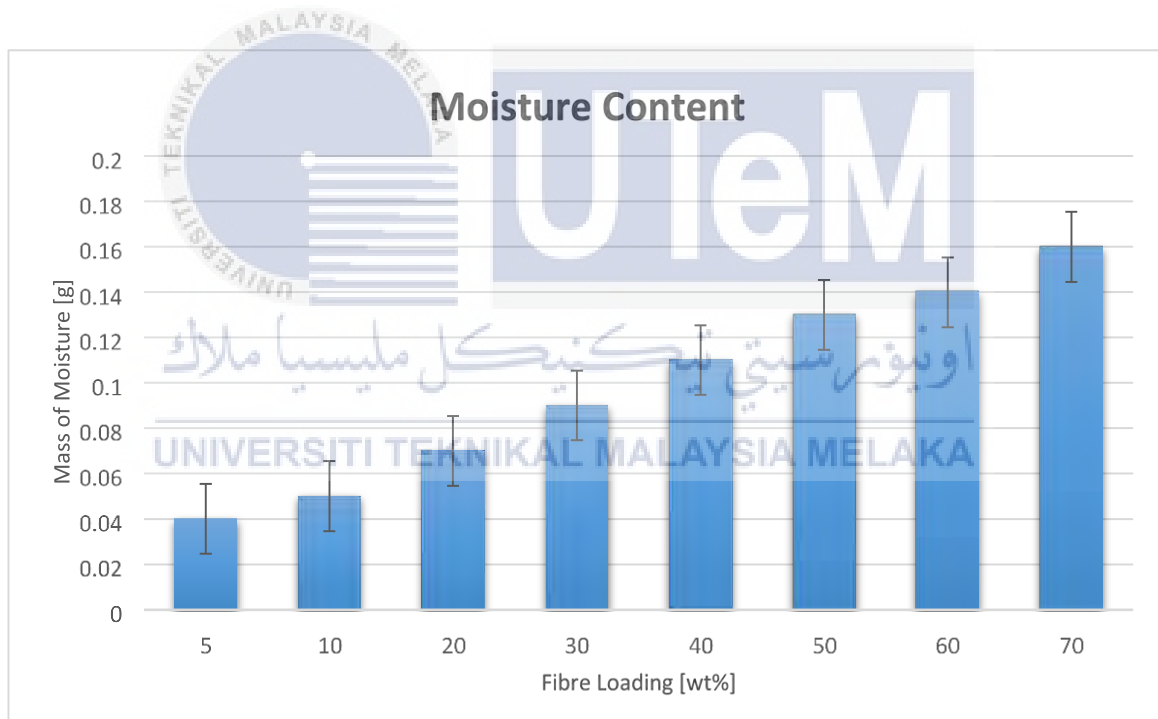


Figure 4.3: Moisture Content in Corn Starch Reinforced Alkaline Treated PALF Composite

Based on the results obtained, the values of moisture content are increased linearly as the fibre loading increase. 5 wt% of fibre loading has the lowest mass of moisture content which is 0.04 g and contradicted with the 70 wt% of fibre loading which is 0.16 g. The results

achieved are similar to the previous research which stated that the natural fibre is hydrophilic and the moisture content is higher with the presence of plenty of fibre loading (Ramesh *et al.*, 2016).

4.1.2 Water Absorption

There are many advantages of PALF because of their good properties such as not harmful to environment and also applicable as a reinforcement material. Instead of that, PALF also have certain flaws. According to the nature behaviour of PALF, high water absorption of the fibre has limit the product applications and this problem can be solved with surface treatment on the PALF. The biopolymer matrix which referred as corn starch in this study tends to have micro sized of crack. Hence, water molecules can easily penetrate and diffusion of water molecules towards the inside occur between micro gaps of polymer molecular chains. Next, water retention happens when there are inconsistent of hydrophobic matrix and the hydrophilic PALF inside the micro-void flaw. The capillary effect also give effect as the behaviour is to carry water through fibre and matrix interface which can leads to undesired mechanical properties changes. Figure 4.4 shows the water absorption of corn starch reinforced alkaline treated PALF sample.

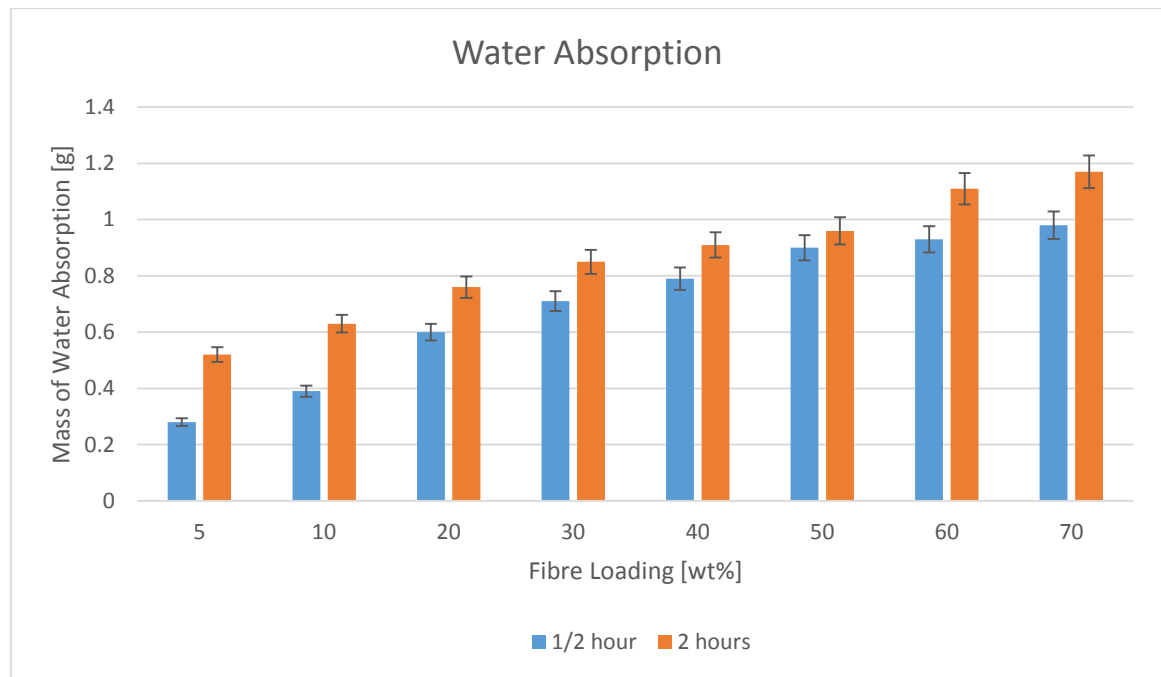


Figure 4.4: Water Absorption of Corn Starch Reinforced Alkaline Treated PALF Composite

In this study, corn starch reinforced alkaline treated PALF was investigated to evaluate the tendency of water absorption behaviour according to the fibre loading and the period of immersion. The study of water absorption was proceed by taking the fibre loading and time of immersion as the manipulated variables. According to the previous research, higher amount of water absorb can decrease the mechanical properties of natural reinforced composites (Tawakkal *et al.*, 2012).

Based on the graph shown in Figure 4.4, the mass of water absorption increase as the fibre loading increase. All sample show that the water absorption is increase linearly because the PALF is a plant based fibre which has hydrophilic behaviour. The results obtained is similar to previous study which stated that the level of water absorption is increased as the cellulose molecules in natural fibres consist of high quantity of hydroxyl groups (Rout *et al.*, 2016). In addition, the hydroxyl groups make the hydrogen bonds with water molecule, hence the rate of water absorption increased. Besides that, the results shows that the period of immersion of two hours give higher value of mass of water absorption rather than the

samples that being immersed for half an hour in water. This is because the PALF that immersed for two hours has longer time to absorb more water. The PALF was completely attached to the matrix and the sides of the specimen was exposed longer in time to the water and a significant amount of water was absorbed through capillary action (Ramesh *et al.*, 2016).

4.1.3 Density

Every substance/matter has their own density and natural fibre also has the density. Density can be referred as a measurement that differentiate the amount of matter of an object to its volume. A simple comparison can be made between the amount of water which are an object with plenty matter will has high density in a certain volume; and vice versa. Figure 4.5 shows the density of corn starch reinforced alkaline treated PALF composite.

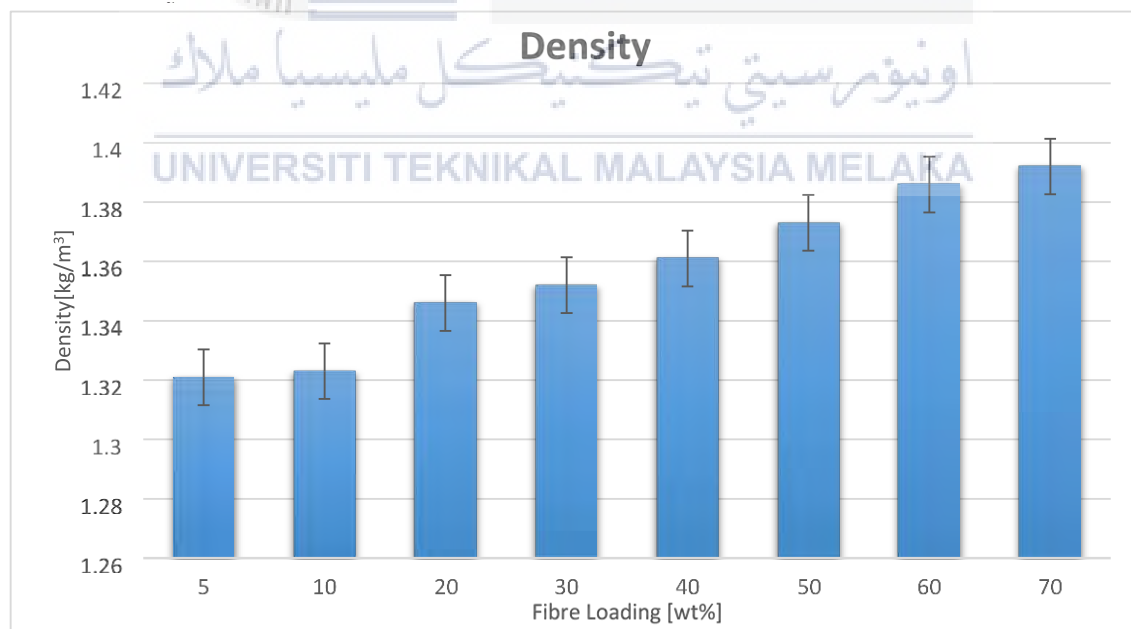


Figure 4.5: Density of Corn Starch Reinforced Alkaline Treated PALF Composite

The result obtained from the experiment gives the increasing value of density through the increasing fibre loading weight percentages. The lowest density is belonged to 5 wt% of fibre loading which is 1.321 kg/m^3 while the highest density is 1.392 kg/m^3 as for the 70 wt% of fibre loading. In theoretical study, higher fibre loading weight percentage makes the mass of the composite increases, thus resulting in higher density as well. To be conclude, the higher the fibre loading weight percentage, the higher the density of the composite.

4.2 Mechanical Results

4.2.1 Tensile Properties

Tensile test were conducted in this study to determine the mechanical properties of the corn starch reinforced alkaline treated PALF composites. Tensile test is performed by applying certain tension load and the specimens were being pulled apart. Ultimate tensile strength (UTS) is one of the important parameter in tensile test and used to evaluate the fibre resistance towards maximum force and stress while being stretched before rupture. Besides, stiffness of an elastic materials is measured by Young's Modulus and also become a quantity that has been used to classify materials.

In this study, ultimate tensile strength is determine by using the different of fibre loading weight percentages. Figure 4.6 shows the results of ultimate tensile strength of different fibre loading weight percentages.

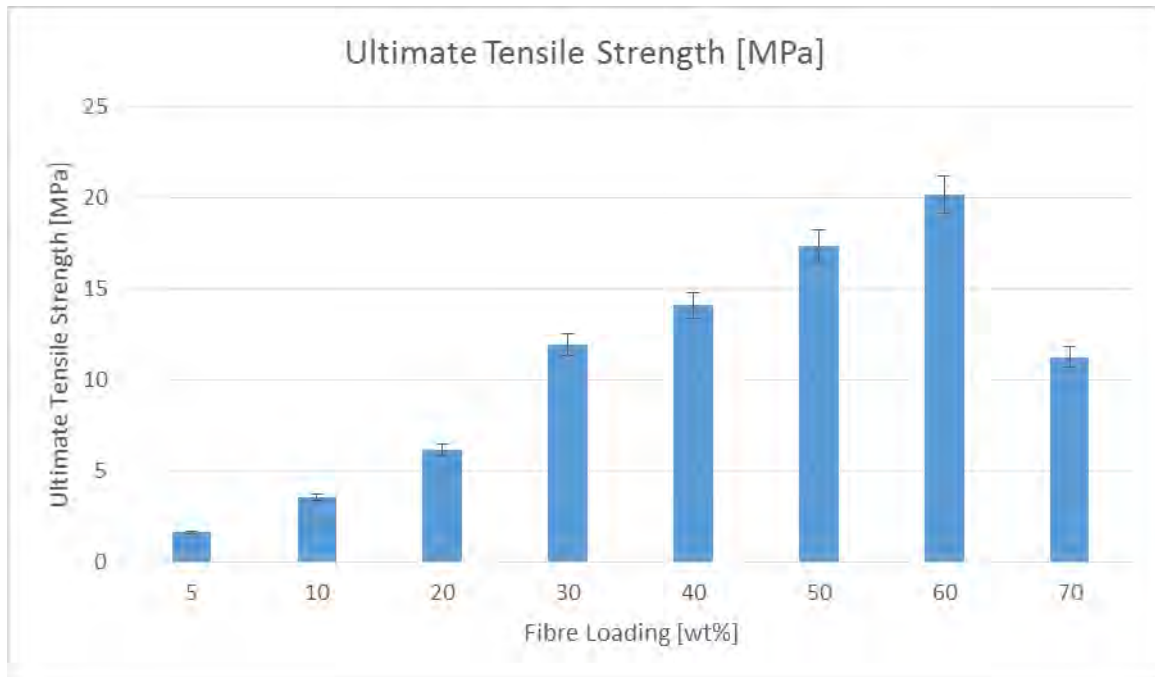


Figure 4.6: Ultimate Tensile Strength of Corn Starch Reinforced Alkaline Treated PALF Composite

From the results achieved in Figure 4.6, it can be observed that the value of ultimate tensile strength is increase linearly and the suddenly declined. Fibre loading with 60 wt% shows an optimum result with value of ultimate tensile strength of 20.1571 MPa, while at 70 wt% of fibre loading the value is reducing significantly with value of 11.2547 MPa. Normally, the value of tensile strength of fibres increase as the fibre loading increase because of the greater content of cellulose (Selamat *et al.*, 2016). However, the ultimate tensile strength has been decreased at 70 wt% of fibre loading because the matrix is not enough to cover up the PALF compared to the other fibre loading weight percentages. In the previous study, it has been stated that good adhesion between matrix and fibre leads to better mechanical properties; tensile strength (Ghasemlou *et al.*, 2013). This results proves that load transfer between the matrix and PALF is not effective. As expected, lack of matrix make it unable to support externally applied load when load fibres get transferred to matrix material (Kumar *et al.*,

2014). Thus, the composite become more brittle and eventually decreased the ultimate tensile strength.

4.2.2 Flexural Properties

The aim of flexural test is to evaluate the strength and the ability of the material to withstand deformation under loading before breaking point occurs. Basically, flexural test is conducted to determine the value of modulus elasticity in bending, flexural stress-strain, flexural stress and flexural strain response of the material. The ability of withstand the bending deformation is known as flexural modulus.

In this study, three point bending test were conducted. Three point bending test is easy to perform in term of specimen preparation and testing. Several important parameters such as flexural stress and flexural modulus have been evaluated from the results obtained. Figure 4.7 shows the results obtained after undergo the flexural test. As visualized in Figure 4.7, the values of flexural stress and flexural modulus is increased gradually until 40 wt% of fibre loading before started to decline at 50 wt% and continuously declined until 70 wt% of fibre loading. Besides, 5 wt% of fibre loading gives the lowest values of flexural stress and flexural modulus which are 2.47868 MPa and 88.40728 MPa, respectively. This is because, 5 wt% of fibre loading has the lowest fibre loading weight percentage compared to the others.

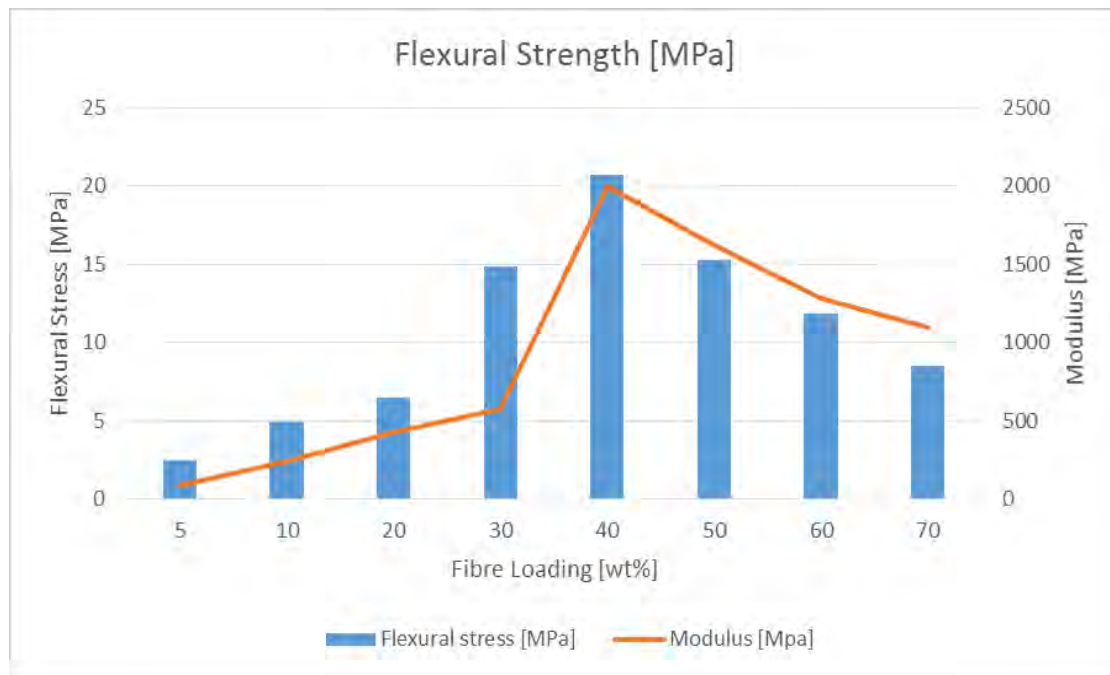


Figure 4.7: Flexural Stress and Modulus of Corn Starch Reinforced Alkaline Treated PALF Composite

In addition, fibres tend to have higher stiffness than the matrix. From literature study, the factor that leads to the lowest flexural strength is when the movement of polymer molecular segmental was limited due to the stiffness of natural fibre that presence in matrix (Heckadka *et al.*, 2014). Next, 40 wt% of fibre loading shows as the maximum flexural stress and flexural modulus which are 20.6998 MPa and 1997.9301 MPa, respectively. At this stage, the corn starch matrix and alkaline treated PLF are homogeneously combined and has the maximum level of orientation. Hence, when the load was applied, stress is uniformly distributed among the fibres and give the best value of flexural stress and flexural modulus. The factor that makes flexural stress and flexural modulus decreased at 50 wt% , 60 wt% and 70 wt% of fibre loading is due to the lack of matrix that can cover up the PLF. The values of flexural strength of 50 wt%, 60 wt% and 70 wt% are 15.2774 MPa, 11.8277 MPa and 8.553 MPa while the values of flexural modulus are 1620. 5112 MPa, 1279.9006 MPa and 1093. 3448 MPa, respectively. As expected, when the fibre loading weight percentage is too

high, the stress will not transferred uniformly due to the clustered fibres within the matrix and thus, overall flexural strength and flexural modulus of the composites will declined (Sood and Dwivedi, 2017).

4.3 Morphological Analysis

4.3.1 Scanning Electron Microscopy (SEM)

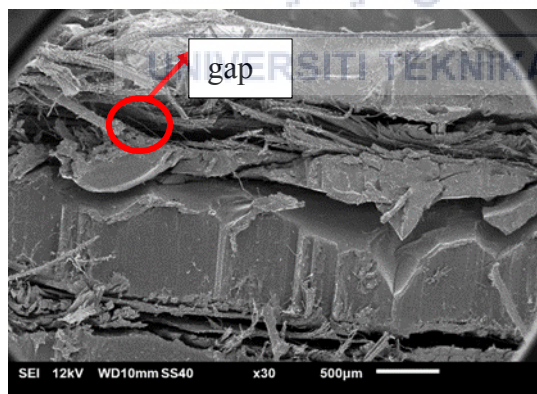
The characterization of morphological analysis on the natural fibre is one of the factor that affected the physical and mechanical properties of a composite with fibre as reinforcement. Morphological analysis is performed on the fracture surface of tensile test. The samples are coated with platinum as to provide electrical conductivity which can give good quality results.

In this study, one sample was taken from three different fibre loading which are 10 wt%, 60 wt% and 70 wt%. Instead of eight samples that have been produce, only three samples were taken for morphological analysis. Specimen with 10 wt%, 60 wt% and 70 wt% of fibre loading were chosen as to indicate the morphology characterization of initial, optimum and lowest in term of tensile strength, respectively. Figure 4.8(a) shows the morphological results of 10 wt% of fibre loading. As the result obtained from the (SEM) analysis, specimen with 10 wt% fibre loading shows a lot of gaps between the corn starch matrix and PALF. These gaps may be due to the insufficient alkaline treatment. Based on the result achieved from tensile test, specimen with 10 wt% of fibre loading has the low value of ultimate tensile strength because. According to the previous research, the further improvement in term of alkaline treatment can be considered as to improve the adhesion between the matrix and fibre (Nopparut and Amornsakchai, 2016).

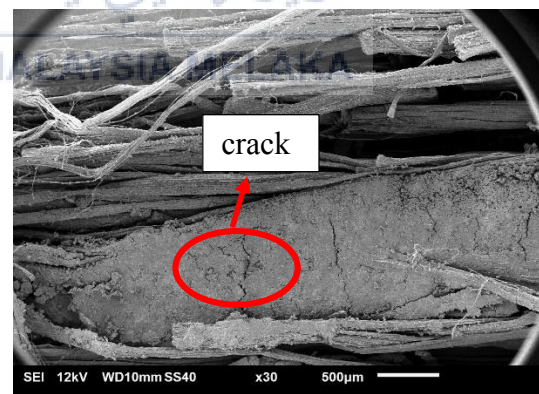
Next, Figure 4.8 (b) shows the morphology result for specimen with 60 wt% of fibre loading. The result of morphology in Figure 4.8 (b) shows that there are some cracks on the

matrix material and noticeable gaps. However, these gaps did not give significant effect to the corn starch and PALF as the bonding is still hold together. Theoretically, the specimen with 60 wt% will has the optimum value of ultimate tensile strength because the load is fully transfer to the bundle of reinforced material (Kasim *et al.*, 2016).

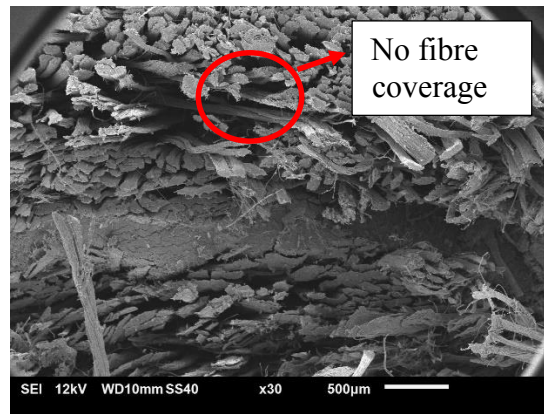
Besides, Figure 4.8 (c) shows the result of morphology for 70 wt% fibre loading that has a drastic decreased value in ultimate tensile strength. The specimen shows that the PALF are not fully covered by the corn starch matrix because of the highest value of fibre loading. Based on the previous study, the composite with low amount of starch will decreased the ultimate tensile strength because of the low of amylose and amylopectin (Wattanakornsiri and Tongnunui, 2012). The result also shows the large amount of fibre which are not cover up by the matrix. Regarding to this study, 70 wt% is the highest fibre loading which can decreased the bonding mechanism between the corn starch matrix and PALF. Due to the weak bonding between the corn starch matrix and PALF, the composite specimen become easy to fracture when the load is applied during tensile test.



(a)



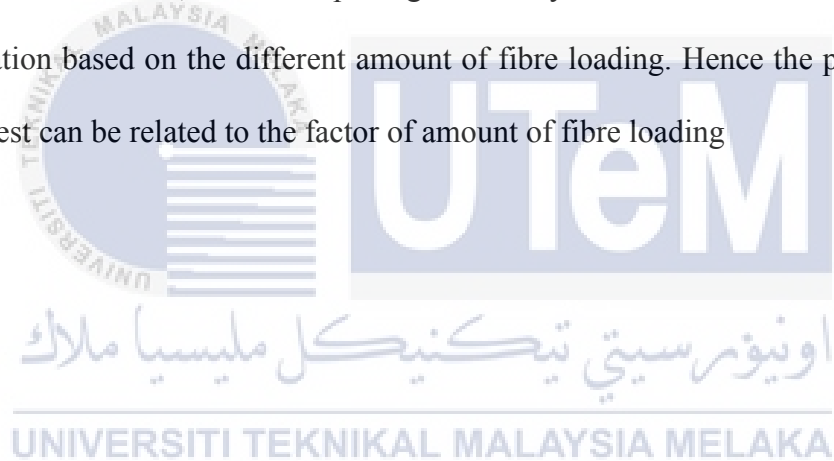
(b)



(c)

Figure 4.8 (a), (b) and (c): Morphological Analysis Results

To be conclude, the morphological analysis results shows the different characterization based on the different amount of fibre loading. Hence the performance of the tensile test can be related to the factor of amount of fibre loading



CHAPTER 5

CONCLUSION AND RECOMMENDATION FOR FUTURE WORKS

In conclusion, this report consists of the study about mechanical behaviour of natural composite between alkaline treated pineapple leaf fibre and corn starch. Composites can be defined as material with multifunctional system that have certain characteristic which cannot be found in other discrete material. Based on the previous study, composite materials have better heat resistance compared to the other material with single component (Milanese, Cioffi and Voorwald, 2011). Natural fibre reinforce polymer composite (NFPC) is knowingly as attentive source and have potential replacement over common synthetic fibres in several applications that commonly related to automotive and construction industry. Previously, pineapple plant is not fully utilised and has become as the agriculture waste until the researchers found that pineapple leaf can be turned into fibre that is beneficial to certain industries and applications. Nowadays, pineapple leaf fibre has been used widely for composite production because the fibre own good mechanical properties and shows superior properties based on the high cellulose content and low microfibrillar angle (Kengkhetkit and Amornsakchai, 2012). Next, there are several steps that will be done in completing the research and determining the mechanical behaviour of natural composite between pineapple leaf fibre and corn starch. Raw pineapple leaf fibre is collected from Johor and undergo alkaline treatment by immersing in NaOH solution and distilled water. Next, pineapple leaf fibre is cured and rubbed to make it soften. Pineapple leaf fibre then cut into 1cm of length. To produce the sample of composite, pineapple leaf fibre is put with corn starch into the mould by lay up method according to the certain compositions. The mould then preheated,

compressed and cooled using hot press machine. Once the samples are produced, then several testing were conducted to determine the physical properties such as moisture content, water absorption and density. Based on the results obtained, all of these physical properties is increase linearly with the increasing fibre loading wt%. Besides, mechanical properties such as tensile, flexural strength and hardness also determined by mechanical tests. The results show that tensile properties is optimum at 60 wt% while for the flexural strength is maximum at 40 wt% fibre loading. Regarding to this study, the physical and mechanical properties are depending on the fibre loading wt%. The characterization of the fracture tensile also figured out by conducting Scanning Electron Method (SEM). Next, many researchers are interested in the field of biodegradable composite development, thus the responsible of disposal of waste and conservation of space are important elements to be considered. Natural composites containing natural fibres and starch tend to have adhesion problems. According to the previous study, the additional starch content thermoplastic blends increases flexibility but give the drawback which is decrease mechanical strength (Kolybaba *et al.*, 2003). ASTM standard of polymer matrix composite have been referred in this study. As for future work recommendation, a need for review and improvement of these test have to be done because in particular, non-homogenities are occurred in biodegradable composites by the clamp used for tensile test (Singh, Afrin and Karim, 2017). Besides, the nature of natural fibre used in composites needs different consideration compared to the synthetic materials. This study can be improved by determining the aspects that can increase the mechanical properties as to give best performance for certain application in the future work. For instance, nanocomposites can be one of the advancement in the composites development in the future as it has many benefits compared to the conventional fibres in term of size. The improvement if their hydrophobic properties, surface modification and advanced processing techniques can also took into account.

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APPENDIX A

ASTM D3039



Designation: D 3039/D 3039M – 00^{ε1}

Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials¹

This standard is issued under the fixed designation D 3039/D 3039M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

^{ε1} Note—Eq 5 was revised editorially in December 2002.

1. Scope

1.1 This test method determines the in-plane tensile properties of polymer matrix composite materials reinforced by high-modulus fibers. The composite material forms are limited to continuous fiber or discontinuous fiber-reinforced composites in which the laminate is balanced and symmetric with respect to the test direction.

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement²
- D 883 Terminology Relating to Plastics²
- D 2584 Test Method for Ignition Loss of Cured Reinforced Resins³
- D 2734 Test Method for Void Content of Reinforced Plastics³
- D 3171 Test Methods for Constituent Content of Composites Materials⁴
- D 3878 Terminology for Composite Materials⁴
- D 5229/D 5229M Test Method for Moisture Absorption

Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials⁴

- E 4 Practices for Force Verification of Testing Machines⁵
- E 6 Terminology Relating to Methods of Mechanical Testing⁵
- E 83 Practice for Verification and Classification of Extensometers⁵
- E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus⁵
- E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process⁶
- E 132 Test Method for Poisson's Ratio at Room Temperature⁵
- E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁶
- E 251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages⁵
- E 456 Terminology Relating to Quality and Statistics⁶
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method⁶
- E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading⁵
- E 1237 Guide for Installing Bonded Resistance Strain Gages⁵

3. Terminology

3.1 **Definitions**—Terminology D 3878 defines terms relating to high-modulus fibers and their composites. Terminology D 883 defines terms relating to plastics. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other standards.

3.2 Definitions of Terms Specific to This Standard:

NOTE—If the term represents a physical quantity, its analytical dimensions are stated immediately following the term (or letter symbol) in fundamental dimension form, using the following ASTM standard symbology for fundamental

¹ This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

Current edition approved April 10, 2000. Published July 2000. Originally published as D 3039 – 71T. Last previous edition D 3039 – 95a.

² Annual Book of ASTM Standards, Vol 08.01.

³ Annual Book of ASTM Standards, Vol 08.02.

⁴ Annual Book of ASTM Standards, Vol 15.03.

⁵ Annual Book of ASTM Standards, Vol 03.01.

⁶ Annual Book of ASTM Standards, Vol 14.02.

APPENDIX B

ASTM D7264



Designation: D 7264/D 7264M – 07

Standard Test Method for Flexural Properties of Polymer Matrix Composite Materials¹

This standard is issued under the fixed designation D 7264/D 7264M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method determines the flexural stiffness and strength properties of polymer matrix composites.

1.1.1 *Procedure A*—A three-point loading system utilizing center loading on a simply supported beam.

1.1.2 *Procedure B*—A four-point loading system utilizing two load points equally spaced from their adjacent support points, with a distance between load points of one-half of the support span.

NOTE 1—Unlike Test Method D 6272, which allows loading at both one-third and one-half of the support span, in order to standardize geometry and simplify calculations this standard permits loading at only one-half the support span.

1.2 For comparison purposes, tests may be conducted according to either test procedure, provided that the same procedure is used for all tests, since the two procedures generally give slightly different property values.

1.3 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text, the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

- D 790 Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials
- D 2344/D 2344M Test Method for Short-Beam Strength of

Polymer Matrix Composite Materials and Their Laminates

D 3878 Terminology for Composite Materials

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials

D 5687/D 5687M Guide for Preparation of Flat Composite Panels with Processing Guidelines for Specimen Preparation

D 6272 Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending

D 6856 Guide for Testing Fabric-Reinforced “Textile” Composite Materials

E 4 Practices for Force Verification of Testing Machines

E 6 Terminology Relating to Methods of Mechanical Testing

E 18 Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials

E 422 Practice for Calculating Sample Size to Estimate, With a Specified Tolerable Error, the Average for a Characteristic of a Lot or Process

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods

E 456 Terminology Relating to Quality and Statistics

E 1309 Guide for Identification of Fiber-Reinforced Polymer-Matrix Composite Materials in Databases

E 1434 Guide for Recording Mechanical Test Data of Fiber-Reinforced Composite Materials in Databases

2.2 Other Documents:

ANSI Y14.5-1999 Dimensioning and Tolerancing—Includes Inch and Metric³

ANSI B46.1-1995 Surface Texture (Surface Roughness, Waviness and Lay)³

3. Terminology

3.1 *Definitions*—Terminology D 3878 defines the terms relating to high-modulus fibers and their composites. Terminology E 6 defines terms relating to mechanical testing. Terminology E 456 and Practice E 177 define terms relating to statistics. In the event of a conflict between terms, Terminology D 3878 shall have precedence over the other documents.

¹ This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

APPENDIX C

ASTM D5229



Designation: D 5229/D 5229M – 92 (Reapproved 2004)

Standard Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials¹

This standard is issued under the fixed designation D 5229/D 5229M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the Department of Defense.

INTRODUCTION

Consistent evaluation and comparison of the response of polymer matrix composites to moisture absorption can only be performed when the material has been brought to a uniform through-the-thickness moisture profile. The procedures described in Test Method D 570 and Practices D 618 do not guarantee moisture equilibrium of the material. A similar, but more rigorous, procedure for conditioning to equilibrium is described by this test method, which can also be used with fluid moisture other than water, and which, additionally, can provide the moisture absorption properties necessary for the analysis of single-phase Fickian moisture diffusion within such materials.

1. Scope

1.1 This test method covers a procedure (Procedure A) for the determination of moisture absorption or desorption properties in the through-the-thickness direction for single-phase Fickian solid materials in flat or curved panel form. Also covered are procedures for conditioning test coupons prior to use in other test methods; either to equilibrium in a non-laboratory environment (Procedure B), to equilibrium in a standard laboratory atmosphere environment (Procedure C), or to an essentially moisture-free state (Procedure D). While intended primarily for laminated polymer matrix composite materials, these procedures are also applicable to other materials that satisfy the assumptions of 1.2.

1.2 The calculation of the through-the-thickness moisture diffusivity constant in Procedure A assumes a single-phase Fickian material with constant moisture absorption properties through the thickness of the specimen. The validity of the equations used in Procedure A for evaluating the moisture diffusivity constant in a material of previously unknown moisture absorption behavior is uncertain prior to the test, as the test results themselves determine if the material follows the single-phase Fickian diffusion model. A reinforced polymer matrix composite material tested below its glass-transition temperature typically meets this requirement, although two-phase matrices such as toughened epoxies may require a

multi-phase moisture absorption model. While the test procedures themselves may be used for multi-phase materials, the calculations used to determine the moisture diffusivity constant in Procedure A are applicable only to single-phase materials. Other examples of materials and test conditions that may not meet the requirements are discussed in Section 1.4.

1.3 The evaluation by Procedure A of the moisture equilibrium content material property does not assume, and is therefore not limited to, single-phase Fickian diffusion behavior.

1.4 The procedures used by this test method may be performed, and the resulting data reduced, by suitable automatic equipment.

1.5 This test method is consistent with the recommendations of MIL-HDBK-17B (1),² which describes the desirable attributes of a conditioning and moisture property determination procedure.

1.6 The values stated in either SI units or inch-pound units are to be regarded separately as standard. Within the text the inch-pound units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system must be used independently of the other. Combining values from the two systems may result in nonconformance with the standard.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ This test method is under the jurisdiction of ASTM Committee D30 on Composite Materials and is the direct responsibility of Subcommittee D30.04 on Lamina and Laminate Test Methods.

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² The boldface numbers in parentheses refer to the list of references at the end of this standard.