THE EFFECT OF SURFACE TREATMENT ON PALF REINFORCED VINYL ESTER COMPOSITES

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DECLARATION

I declare that this project report entitled "The effect of surface treatment on pineapple leaf fibre reinforced vinyl ester composites" is the result of my own work except as cited in the references

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APPROVAL

I hereby declare that I have read this project report and in my opinion this report is enough in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Plant & Maintenance).

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ABSTRACT

Nowadays, the growing of the science and technology is the main problem to the environmental and become very serious all over the world. Researchers are working together to produce the environment-friendly material or also known as a green material in order to solve this critical problem. Researchers have focused the usage of natural fibre to replace synthetic fibre. Natural fibre have a lot of advantages compare with synthetic fibre such as low cost, eco-friendly product and can reduce waste of pineapple leaf. In this research, the objective is to investigate the effect of surface treatment PALF to the vinyl ester reinforcement in terms of mechanical, physical and morphological properties. The parameter use in this research is 5wt% of the fibre loading for untreated and treated of PALF. The chemical treatment use is alkaline treatment which is 6% of NaOH. The sample will be characterized in term of their physical, mechanical and morphological properties. Based on the physical properties, the density of the treated composites was higher due to increase the amount of the fibre in the composites, the treated composites also show good result for moisture content and water absorption. For the mechanical properties, treated composites showed an improvement for tensile and flexural strength. This is because alkaline treatment on the fibre improve the interfacial bonding between fibre and polymer. For morphological analysis, treated composites also has good surface structure because there is no gap between fibre and matrix and less fibre pull-out compared with the untreated composites sample. As the conclusion, alkali treatment on the natural fibre improve the interfacial bonding between fibre and polymer.

ABSTRAK

Pada masa kini, perkembangan sains dan teknologi merupakan masalah utama kepada alam sekitar dan menjadi sangat serius di seluruh dunia. Penyelidik bekerjasama untuk menghasilkan bahan mesra alam atau juga dikenali sebagai bahan hijau untuk menyelesaikan masalah kritikal ini. Penyelidik menumpukan penggunaan serat semula jadi untuk menggantikan serat sintetik. Serat semulajadi mempunyai banyak kelebihan berbanding dengan serat sintetik seperti kos rendah, produk mesra alam dan dapat mengurangkan pembaziran daun nanas. Dalam penyelidikan ini, objektifnya adalah untuk mengkaji kesan rawatan permukaan serat nanas bercampur dengan vinyl ester dari segi sifat mekanikal, fizikal dan morfologi. Parameter yang digunakan dalam kajian ini ialah 5wt% daripada beban serat untuk PALF yang tidak dirawat dan dirawat. Penggunaan rawatan kimia adalah rawatan alkali iaitu 6% NaOH. Ujian sampel dibahagikan kepada tiga fasa iaitu ujian fizikal, ujian mekanikal dan analisis morfologi. Berdasarkan ujian fizikal, ketumpatan komposit terawat adalah lebih tinggi kerana meningkatkan jumlah serat dalam komposit, komposit yang dirawat juga menunjukkan hasil yang baik untuk kandungan lembapan dan penyerapan air. Untuk ujian mekanikal, komposit yang dirawat mempunyai bacaan yang lebih tinggi untuk kekuatan tegangan dan lentur. Ini adalah kerana rawatan alkali pada serat meningkatkan ikatan antara antara serat dan polimer. Untuk analisis morfologi, komposit terawat juga mempunyai struktur permukaan yang baik kerana tiada jurang antara serat dan matriks dan kurang jumlah serat yang teertarik berbanding dengan sampel komposit yang tidak dirawat. Sebagai kesimpulan, rawatan alkali pada serat semulajadi adalah kaedah yang sangat berkesan untuk menghasilkan produk serat semulajadi berkualiti tinggi.

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LIST OF ABBREVIATION

Abbreviation	
PALF	Pineapple Leaf Fibre
CFRP	Carbon Fibre Reinforced Polymer
PLA	Polylactic Acid
PP	Polypropylene
PC	Polycarbonate
HIPS	High Impact Polystyrene
VE	Vinyl Esters
SEM	Scanning Electron Microscope.

LIST OF SYMBOLS

Symbol	
NaOH	Sodium Hydroxide
ρ	Density
m	Mass
V	Volume

CHAPTER 1

INTRODUCTION

1.1 Overview of the project

Nowadays, the growing of the science and technology is the main problem to the environmental and become very serious all over the world. Unsafe modern material that can influence an individual's wellbeing, the transfer of the unbiodegradable material, for example, plastic and an Earth-wide temperature boost cause by the nursery impact. Researchers are working together to producing the environment-friendly material or also known as a green material in order to solve this critical problem. Researchers have focused the usage of natural fibre to replace synthetic fibre.

The use of natural fibre can reduce the addiction on non-renewable energy and decrease pollutant emissions. The other advantages is can reduced greenhouse gas emissions and improve the energy recovery(Sanjay and Yogesha, 2017). In term of mechanical properties, natural fibre has low in density and high specific strength and stiffness. Natural fibre has lower cost compared with the synthetic fibre. It is also renewable resources and has low abrasive damage while processing equipment process(K.L. Pickering, M.G. Aruan Efendy, 2016).

Several types of natural fibre has possibilities to become reinforcement materials in composites and replacement for glass(Sgriccia, Hawley and Misra, 2008). Sisal, coir, jute, ramie, pineapple leaf (PALF), and kenaf is the most used of natural fibre in industry.

Pineapple leaf (PALF) has to be selected as a reinforcing material in natural composite products because Malaysia is one of the main world producers of pineapple. However, only the fruit is employed whereas the leaf, whose main content is fibre, is burnt or thrown away. The use of pineapple leave as natural fibre can reduce environmental pollution and can generate extra income for the country especially Malaysia.

Some of the pineapple leaves has been used in the manufacture of yarn, woven fabrics and handmade products. Fibres extracted from pineapple leaves suitable for industrial applications because it is one of the finest fineness indices among the vegetable fibres(Leão *et al.*, 2014). Even the PALF fibre has a lot of advantages in term of their physical properties it still has some flaws. However, there has a separate method to improve the fibres surface to make it suitable for interfacial fibre/matrix bonding. The amount of cellulose in the PALF is about 70-80%wt which is making it having specific modulus and strength(Panyasart *et al.*, 2014).

Vinyl ester is one of the thermosets and resin will be used in this research. Short fibre will be used as reinforced material in this research. To assess the growth of PALF-vinyl ester composites, there are many criteria can be used which is mechanical properties, physical properties and thermal stability.

This study discusses the PALF mechanical and physical properties characteristic after doing a surface treatment of PALF reinforced with thermoset materials. In this research, the PALF was treated using alkali treatment to produce PALF composites that have better physical, mechanical and morphological properties.

1.2 Problem statement

Natural fibre is found abundantly in Malaysia. There are several types of fibre typically used in Malaysia. For example, is kenaf, abaca, oil, pineapple leaves, sugarcane bagasse, and banana. PALF is one of the potential fibre that can replace the synthetic fibre.

In Malaysia, pineapple leaves are one of the available waste materials. Generally, the agriculture industries only utilized pineapple fruit while the leaf is currently being wasted. The most critical part is when the pineapple leaves are treated as agricultural wastes where it either being let composted or burned by farmers. Basically, natural fibre has a lot of advantages over the disadvantages, but it still needs to modify some of the mechanical and physical properties of the natural fibres. The surface modification is important to restructure the hydrophilic characteristic of the natural fibres fibres and become more effective compared with the synthetic fibre.

1.3 Objective

The objective of the project:

1. To investigate the effect of surface treatment on the pineapple leaf fibre reinforced vinyl ester in terms of mechanical, physical and morphological properties.

1.4 Scope of product

In this project, there have four phases need will be carried out in order to have better understanding about pineapple fibre composites in term of physical, mechanical and morphological properties. The first phase this research is PALF is treated with alkali solution and blended with vinyl ester form the composite sample.

The second phase of this research, the composites were prepared. The composites are being fabricated by place the PALF and vinyl ester into the mould and handle using hand lay-up method. Then, insert the samples into the oven to obey the cure cycle of vinyl ester.

The third phase of this project covers the physical and mechanical properties. Physical properties will be characterized by density analysis, water absorption test and moisture content. For mechanical testing covers two types of testing which is a tensile test and flexural test.

The last phase is the most important part of the research where the physical and mechanical properties of PALF were discussed and observed. The chemical treatment was deliberated to investigate which treatment lead to the major improvements in physical and mechanical properties of PALF reinforced vinyl ester. The result of the untreated and treated was compared. All these testing is being done by following the standards involved according to each method.

CHAPTER 2

LITERATURE REVIEW

2.1 Natural fibre composites

In days gone by few decades, the use of natural fibres in composites applications has been increasing. The use of natural fibres is not a modern technology because scientist already used in China and Korea to build a wall in the village by a combination of the straws and mud(Lau *et al.*, 2018). Natural fibres have been chosen by the researchers and scientist because of their eco-friendly and sustainable. Natural fibres convey benefit to many engineering applications, but the worst issues regularly condemned by people in general which is serious environmental problems after disposal of advanced composites as it is hard to be recycled. The used of the carbon fibre reinforced polymer composites (CFRP) create strong strength structure from advanced composites but very high cost of materials for domestic products(Lau *et al.*, 2018). The density and eco-friendly properties of the natural fibre attract many engineering sectors to studied on the improvement of the natural fibre because it can reduce pollution and save the environment.

Natural fibres can be derived from plant, animal, or mineral. Biomedical applications such as implants usually used animal-based fibres as example cocoon silk and spider silks. New bone fixator has been created by Lau et al after done research on the cocoon silk fibre-reinforced Polylactic acid (PLA) to formulated bio-resorbable polymer composites.(Lau *et al.*, 2018).

The next type of natural fibres is plant fibre. Bast fibres, leaf fibres, fruit, seed, wood, and cereal straw is the most used of natural plant in application(Webber, Bhardwaj and Bledsoe, 2002). Among natural fibre, plant fibres are widely used as reinforcement in fibre reinforced composites. It's is do not harm the environment because natural fibres extracted from nature and can be classify as renewable resources. Natural fibres has better specific strength, specific modulus, and lower cost compared with glass fibres. Nowadays, many major components of bio-composite materials like paper, boards, and many structures come from fibres composites.

For the plant-based natural fibres sources, depending on the utilization, two types of fibres class which primary class and second class where the primary class are those plant which grown for their fibre. Sisal, jute, kenaf and hemp classify as primary type of plant but pineapple, oil palm and coir in the second type of plant. The secondary type of plant is where the fibre is producing as a by-product. Figure 2.1 shown that the classification of the natural fibre divided depending upon their region(Thakur, Thakur and Gupta, 2014).

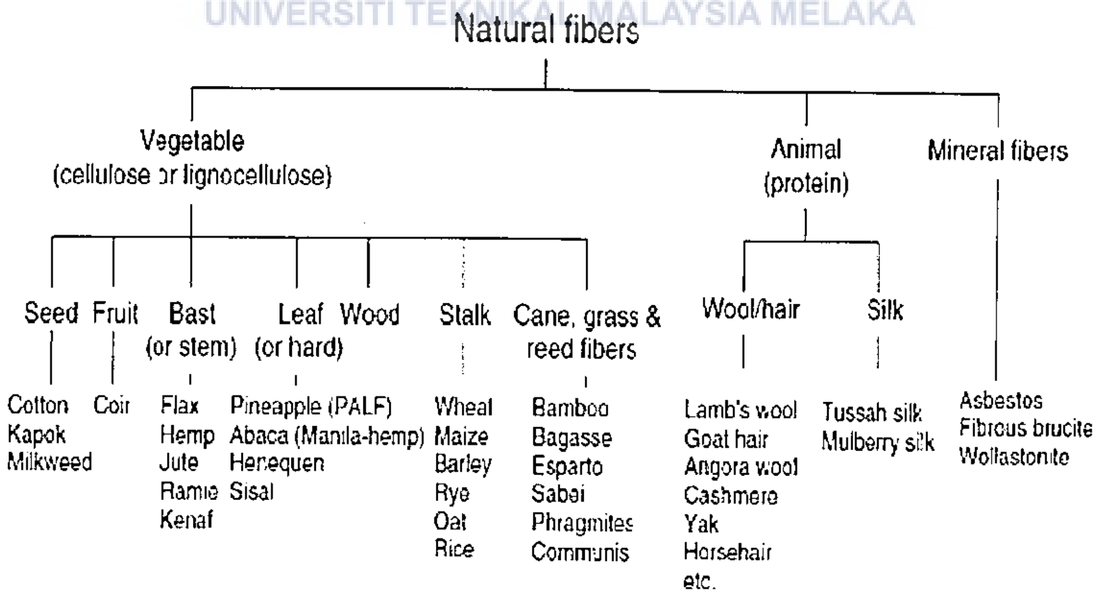

Figure 2.1: Groups of natural fibres (Leão et al., 2014)

Contrasted with synthetic fibres, common natural fibres bring a large number of points of interest for example, low cost, low density, fewer equipment abrasion, and respiratory irritation, improved energy recovery and biodegradability(Sgriccia, Hawley and Misra, 2008). Basically, natural fibres have high moisture and weak adhesion to hydrophobic matrices. The addition of hydrophilic in natural plant will create weak mechanical properties because of the non-uniform fibre dispersion in matrix. The way to improve the adhesion to matrix material is natural fibres need to be treated. Other than that, low degradation temperature of natural fibre makes them inharmonious with thermoset that have large curing temperature. There have other disadvantages of natural fibre such as large variability of mechanical properties, lower elongation, poor resistance to weather and lower ultimate strength.

The structure of the natural fibre consists of the hemicelluloses, lignin, cellulose, waxy and pectin substances. Weak bindings between the fibres and polymer because of the moisture absorption from the surrounding. The modification of natural fibres can change the composition and fibres structures by utilize of the reagent functional group. As a result, the moisture absorption of the natural fibre has been reduced which bring to the great improvement incompatibility between fibre and polymer matrix.

Natural fibres treatment was important factor to measured when processing natural fibres (Mohammed *et al.*, 2015). By performing different type of chemical treatment, natural fibres will lose hydroxyl group, thus reducing the hydrophilic behaviour of the fibres and causing improvement in mechanical strength and dimensional stability of natural fibres reinforced polymer composites. As the result, chemical treatment can enhancement of the natural fibres reinforced composites.

2.2 Pineapple plant

The pineapple is a tropical plant with a comestible multiple fruit consisting of coalesced berries. The pineapple plant in the family Bromeliaceous, subclass monocotyledons and species Ananas comosus. The word Ananas comosus is coming from Tupi word of 'nanas', means excellence fruit. All the world's pineapple cultivars are grouped in several classes which is Smooth Cayenne, Red Spanish, Queen and Abacaxi. The shape of the fruit look like pinecone make the plant is called "pineapple".

Brazil, Costa Rica, and China accounted as one of the world's production of pineapples, producing over 700,000 tone of fruit(Leão *et al.*, 2014). In India, it is refined on about 2250 000 acres of land and is continuously increasing its production. One of the top 3 largest country that produce pineapple in 60 and early 70 in Malaysia. Unfortunately, almost half of the 12 thousand hectares farm of the pineapple plant in Malaysia has been reduced. In Malaysia, the plant of the pineapple is unique because 90% plants in the peat soil which is considered marginal for most other agriculture crop(Y.K.Chan, 2000).People in Malaysia usually known pineapple as Nanas. Pineapple has been used in different varieties for different purpose which is red pineapple and green pineapple used for commercial purpose and Sarawak and Morris pineapple for edible purpose. Figure 2.2 show the red pineapple from Malaysia.

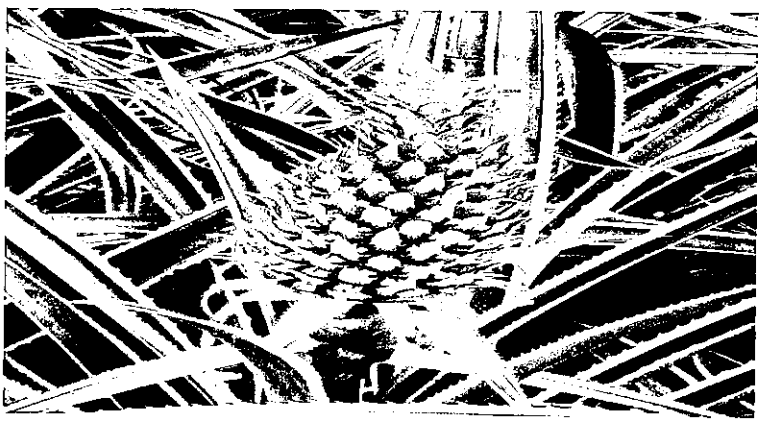

Figure 2.2: Red pineapple

Pineapple is ironic source of bromelain and other cysteine proteases are present in different part of pineapple. Many food industries, cosmetics, and dietary supplements usage bromelain from pineapple but some users need to evade from taking bromelain such as pregnancy, allergies or anticoagulation. The sore mouth feeling after eating pineapple due to the bromelain content of raw pineapple because enzymes react with the proteins.

New fruit will still growth continues through its base even after harvest. 200 days or 6-9 months on average the time need by the plant to allow fruit and propagated vegetative by cutting grown from the plant itself. The pineapple is a short and thick stem where the leaves grow in narrow, rigid and axillary roots. The plant can be categorizing as mature plant when the height of the plant reaches 1 to 1.2 meters, 0.80 width to 1.5 meters and can hold 80 leaves in every shape, with length of up to 1.3 meter. Old leaves place at base of the stem and younger leaves are elongated, conversing a wider baseline. Pineapple plant with detailed view of the leaf shown in figure 2.3.

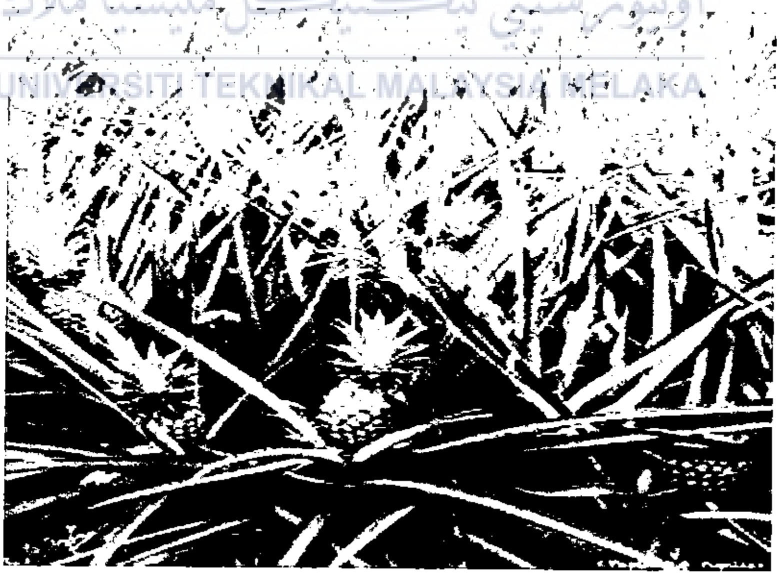

Figure 2.3: Pineapple plant

2.3 Pineapple leaf fibres

Pineapple, banana and citrus is one of the significant tropical fruit in the world. Three type of species locally grown-up in Malaysia which is Moris Gajah, Smooth Cayenne and Josephine. Josephine is one of the suitable species for PALF extraction in term of the fibre quantity, fibre fineness, easy to extraction, mechanical and thermal properties(Fadzullah and Mustafa, 2016). However, the main centre of the plantation of pineapple industry in our country is just on the fruit and other corresponding foodstuff. The leaf normally become wasted in many countries. The pineapple leaf can be evaluated for industrial application to reduce the amount of waste that can harm the environment cause the environmental pollution. This pollution occurs because only fruit will be utilized, and the leaf will become wasted and burn by the farmer will be led to the release of the carbon dioxide.

To solve this problem, multiple researches and studies has been conducted related to the possibility of finding other applications to these pineapple leaf(Mohammed *et al.*, 2015). This will change the pineapple leaves from waste into wealth and save the environment from the pollution. There are several researches has been done by researcher on several aspect of PALF to studies the properties of PALF including physical, mechanical, and chemical.

Most of the previous researcher only focusing on PALF physical and mechanical properties. Chemical treatment of PALF will improve their fitness, resulted in degradation of their tensile properties. Chemical treatment can be done by using silane, acids or alkaline. The mechanical properties of the PALF is outstanding compared with other natural fibres associated with their high cellulose content and low microfibril angle(Leão *et al.*, 2014).

Pineapple leaves comes in shaped like a sword which ascend from a stem with an overall dimension from between 0.9 m to 1.5 m in length, while the width between 2.54 m and 5.1 m respectively. According to the research(Mohamed *et al.*, 2009), PALF have highest tensile strength and middle elongation at break compared with the betel fruits and barks. The extracted of the PALF will deliver advantage in manufacture of yarn, woven fabrics, woven knitted, and handmade products(Leão *et al.*, 2014).

The mechanical properties of PALF can improve by perform surface treatment and PALF can replacing synthetic fibres in many applications. Natural fibres can transformed into nanocellulose, all the defect can be removed and resulting in the fibres with high specific modulus(Leão *et al.*, 2014). PALF is extracted by retting process and classify as multi-cellular and lignocelluloses. The colour of the leaves could be purely green or with a red spot, yellow and ivory(Fadzullah and Mustafa, 2016).

IBRAHIM, (2013) investigates the growth of PALF reinforced polymer composites in term of mechanical properties. The type of matrices has been used in this investigation is the thermosets and thermoplastic resins. The research proposed the major study can focus on long PALF that can be used in manufacturing such as vacuum bag moulding and autoclave moulding.

2.3.1 Extraction method of PALF

There have two method to extracted leaf fibres either manually or mechanically. In Philippines, they manually scraped by using a piece of broken porcelain or a small knife throughout the leaf. Figure 2.4 shown the manual process that started with scraping, beating, and husking the leaves. After that, fibres are soaked in water and added of chemical to improve the activity of the microorganisms.

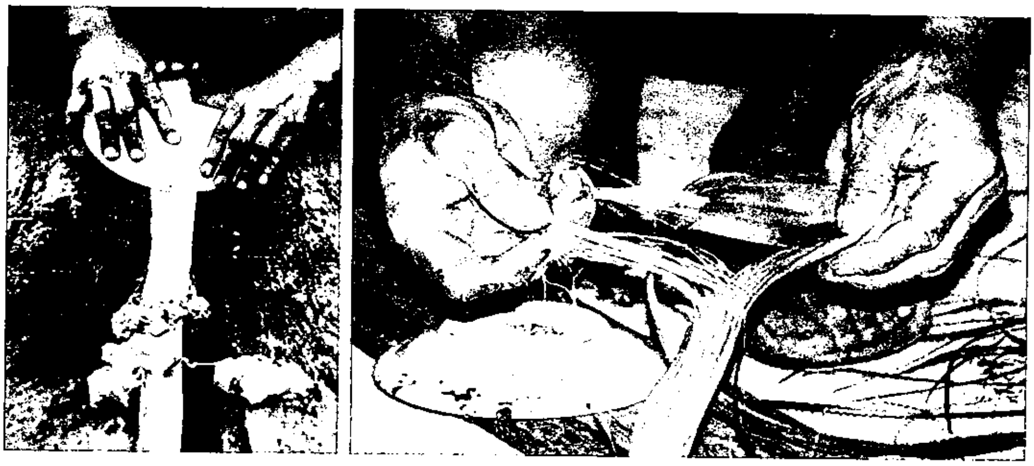

Figure 2.4: Manual shredding of pineapple leaf (Leão et al., 2014)

The second method is mechanical process which is by using decorticator machine. However, mechanical process will bring damage to the PALF through breaking, hackling and scorching that led to the minimise of the tensile strength properties of PALF. This machine has a cylinder and there are some plates that can beatings on the pineapple leaf when the cylinder is rotating(Strategia *et al.*, 2016). Figure 2.5 show that the cylinder was turning by using an electric motor. The leaves fed to the machine and follow the cylinder to rotate, then pulled back out of the machine. After that, the extracted leaves are washed and dried by sun drying method. By using this machine, the extraction process of pineapple become more faster and easier.

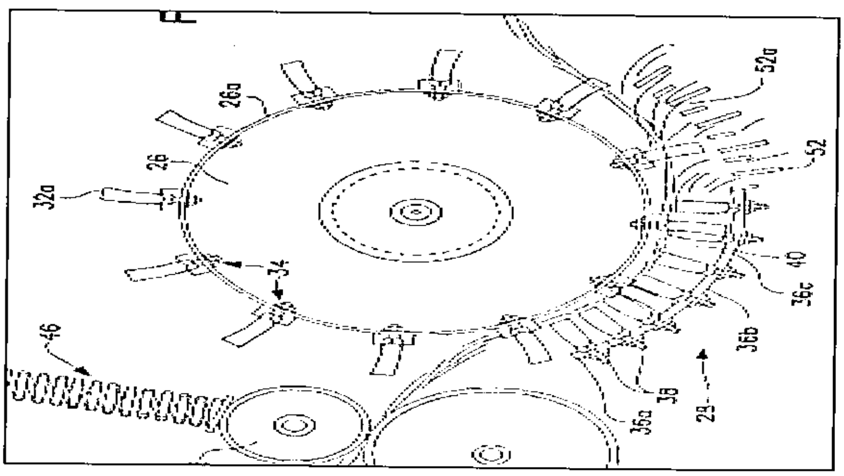

Figure 2.5:Decorticator machine.

In Malaysia, Pineapple Leaf Fiber Machine 1 (PALF M1) in Universiti Tun Hussein Onn (UTHM), Batu Pahat, Johore is one of the machines that can extracted pineapple leaf fibres(Fadzullah and Mustafa, 2016). According to the research (Fadzullah and Mustafa, 2016) extraction process of pineapple leaves including scrapping, retting, and mechanical separation of fibres to drying in air. The pineapple leaves are fed by a feed roller and the second roller scratching the top leaf and the wax layer is removed in the scrapping process. Final part of this process which is the leaver will be crushed by serrated roller and create an entrance path for the retting microbes. Figure 2.6 show the pineapple leaf fibres machine 1 in UTHM.

This machine technically does not used crusher technology but use blade in removing the wax layer in pineapple leaf. The leaf will not be attached at the blades during the extraction process because the size and angle of the blades used in this machine have specific condition.

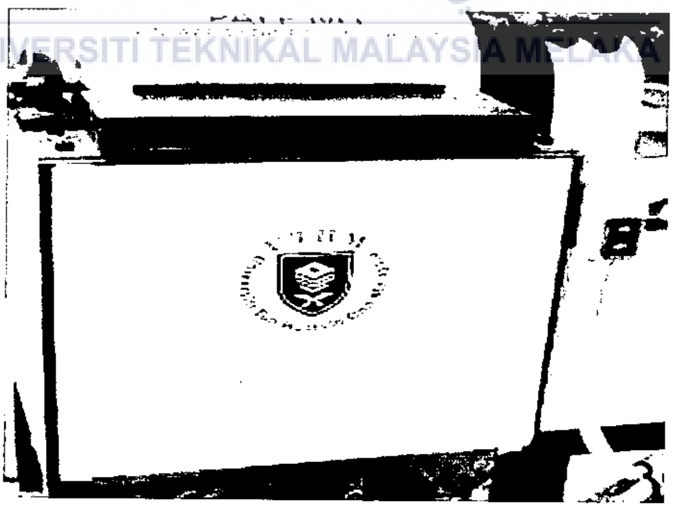

Figure 2.6:PALF machine (Yusof, Yahya and Adam, 2014)

2.4 Pineapple leaf fibres (PALF) reinforced polymer composites

The studies about PALF reinforced polymer composites basically have been done by some researcher because of the replacement glass fibre with the natural fibre in the industry. Lately, PALF is being used successfully in polymer matrix to created composites with improved mechanical strength. The exceptionally good mechanical properties of individual PALF are reflected in its ultimate product. Most of the previous research has been done in order to studies the mechanical, and physical properties of natural fibre reinforced with thermosetting and thermoplastic.

Table 2.1 indicate the physical and mechanical properties of the PALF obtained from previous research. Matrices used for PALF composites is thermosetting and thermoplastic resins. The increasing of the pineapple plantation area has impact with the market price of the pineapple, so the development of the PALF reinforced polymer composites should be increasing to utilize the use of PALF in industry.

Table 2.1: Physical and mechanical properties of PALF.

Mechanical and physical properties.	Source			
	Mohanty et al. (2000)	George et al. (1993)	Arib et al. (2006)	Mohamed et al. (2009)
Tensile strength (MPa)	413–1,627	170	126.6	293.08
Young modulus (GPa)	34.5–82.5	6.26	4.405	18.934
Elongation break (%)	1.6	0.8–1.6	2.2	1.41
Density gcm^{-3}	-	1.44	1.36	-
Moisture content (%)	11.8	-	-	-

2.4.1 PALF reinforced thermoplastic composites

A thermoplastic is a plastic material, a polymer that can be folded or moulded above a certain temperature and solidifies when cooled. The shape can be changed or reshaped by heating if we used thermoplastic and typically produced parts using different polymer processing techniques such as moulding injection, compression moulding and extrusion. Several type of thermoplastic that can be used as reinforced agent with natural fibre which is polypropylene, polystyrene , polycarbonate and polyethene (Mohammed *et al.*, 2015).

Thermoplastic do not melt when heated and will no change the shape upon cooling but only can be melt by heating above melting point. However, thermoplastic also have advantages which is recyclable, tough structure and easy to repair by welding.

George, Bhagawan and Thomas (1996) has studied on the thermal behaviour of pineapple leaf fibre (PALF) reinforced polyethene composites. The purpose of treatment to increase the interfacial adhesion between fibre and matrix by using silane, peroxide and isocyanate. Both treated and untreated fibre was used to compare the properties of the PALF. The result found that treated fibre has better properties of PALF at high-temperature degradation before the polyethene matrix. The cache modulus decreased with increment of temperature, but the increase of fibre loading will increase the storage modulus.

The study of the pineapple leaf fibre reinforced polypropylene composites was report by (Rungsima Chollakup, Rattana Tantatherdtam, Suchada Ujjin, 2010). The results showed that the development of the PALF contents will increase the tensile strength of the composites. The experiment used two different length of fibre which short and long fibre to observe the mechanical properties of PALF reinforced PP. The long fibre PALF were stronger than short fibre because of the greater tensile strength.

Rungsima Chollakup, Rattana Tantatherdtam, and Suchada Ujjin, (2010) investigated the properties of pineapple leaves fibre reinforced polycarbonate composites. The result of PALF treated with alkaline pre-treatment using sodium hydroxide (NaOH) and silane treatment using two different functionalities treatment which is γ-methacryloxy propyl trimethoxy silane and γ-aminopropyl trimethoxy silane was reported. The tensile strength and impact strength of the PALF reinforced PC treated with alkaline treatment is higher compared with the silane treatment. The result show that NaOH treatment has better mechanical properties than silanized composites.

Siregar *et al.*, (2010) carried out investigation on the mechanical properties of PALF reinforced high impact polystyrene (HIPS) composites by performing alkali treatment. The purpose is to studies the result of alkali treatment on PALF reinforced HIPs with different concentration of NaOH. The concentration use in the experiment is 0%, 2% and 4%. All the mechanical properties of PALF reinforced HIPS was observed and compared with other different concentration. The result is alkali treatment with 4% NaOH showed highest mechanical properties compared with others concentration.

2.4.2 PALF reinforced thermosetting composites

A polymer that is irreversibly hardened when healed is called a thermoset polymer. Curing occur because of heat that may be helped by high pressure or use of a catalyst. Thermoset is a cured thermosetting polymer. Usually, thermoset is been used as adhesive because the thermoset resin cannot re-shaped once hardened and cannot be recycles. However, thermoset also have advantages which is low resin viscosity, have great thermal stability, excellent fibre wetting and chemically resistant(S.M. Sapuan, A.R. Mohamed, J.P. Siregar, 2011). There are several types of thermoset resin most widely used which is epoxy, polyester, vinyl ester and phenol formaldehyde (PF).

There is slightly less literature regarding to PALF reinforced vinyl ester. Basically, vinyl esters are strong, flexible and less hydrophilic in nature. Natural fibre-vinyl ester can produce high interfacial shear stress of the fibre-matrix adhesion compared with other matrices(Mohammed *et al.*, 2015). Most of the preparation PALF reinforced VE used wet hand lay-up method compared with the use of the liquid compression moulding process. The availability of PALF reinforced vinyl ester composites can be measured in term of mechanical, physical and thermal properties.

Mishra *et al.*, (2001) investigated the PALF reinforced polyester composites for the mechanical properties in term of fibre loading and fibre surface modification. The mechanical properties including flexural, flexural dan impact has been observed in this experiment. From all modification, 10 % of the PALF composite grafted AN showed maximum tensile strength (48.36 MPa), while the cyanoethylated PALF composite showed improved bending and impact strength. Figure 2.7 shows that the tensile strength of PALF reinforced polyester increase with increasing of the fibre loading. However, the tensile strength started to decrease about 31%wt when the fibre loading at 40%wt.

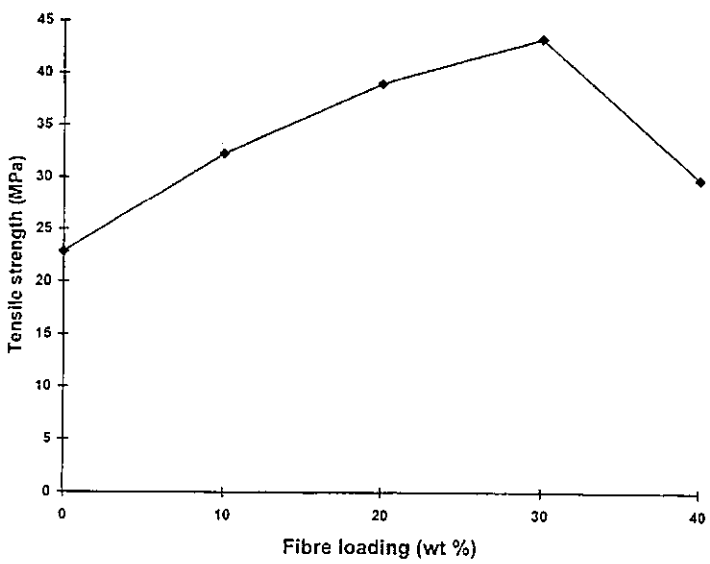

Figure 2.7: Tensile strength of the composites (Mishra et al., 2001)

Recently, the studies about the surface treatment of PALF reinforced epoxy composites has been done to observe the improved mechanical and thermal properties. The improvement of mechanical properties because of the increase the fibre loading in epoxy. Nagarajan *et al.*, (2016) studies the PALF reinforced epoxy using long fibre in unidirectional to produce the composites using hand lay-up process. The result showed that the tensile and flexural modulus increased with fibres content due to the formation of a better bond between fibres and epoxy matrix, which is the mechanical properties PALF reinforced epoxy improved up to 20 %wt fibres loading, but the thermal properties do not change considerably.(Nagarajan *et al.*, 2016). The faster decomposition rate of natural fibre causes the neat epoxy were more than the thermal stability. The decent adhesion with the matrix shows in the surface morphology of the fractured surface because of the fibre deboned and pulled out due to brittle nature of epoxy materials.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The flowchart process shows that all the activity related in this research. The process started with materials gaining for processing, testing and result analysis. Figure 3.1 shows the flow process of the methodology.

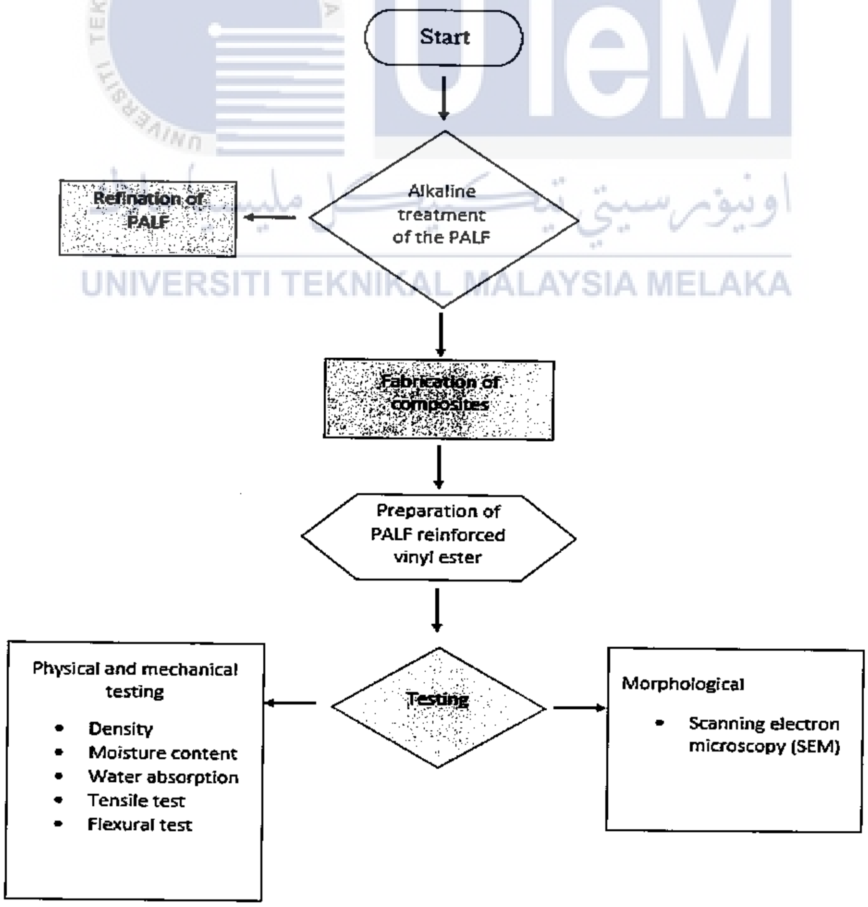

Figure 3.1:Flow process methodology

Based on the flow chart in figure 3.2 and figure 3.3, all the activities and job scope write in the Gantt chart to provides a schedule of earliest possible start and finish time of activities and help in plan the flow of the task. It is also helpful for managing the reliance between tasks.

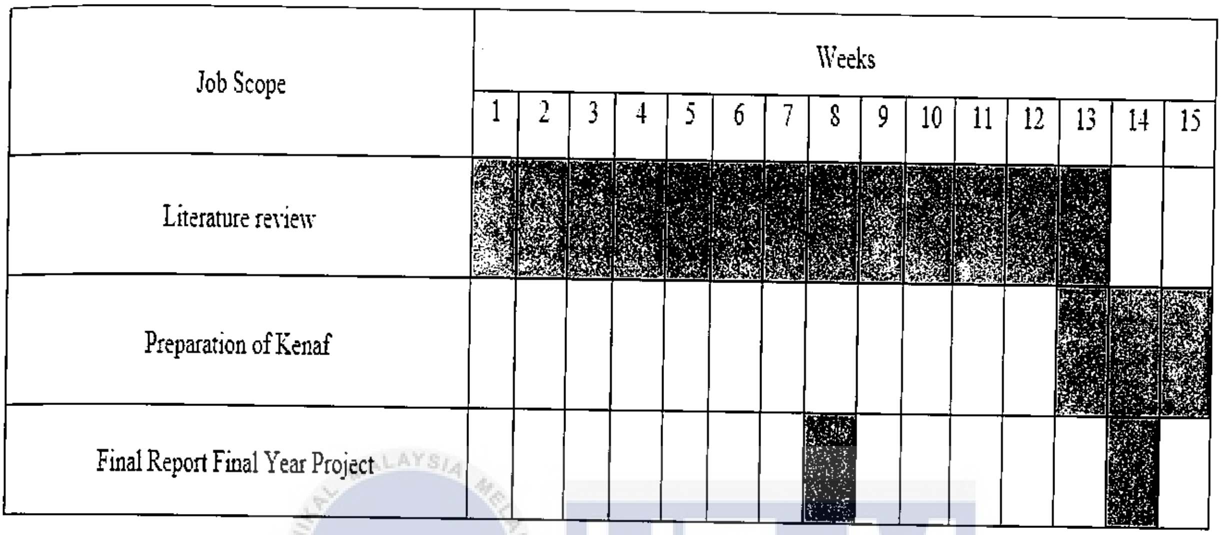

Figure 3.2: Gantt chart FYP 1

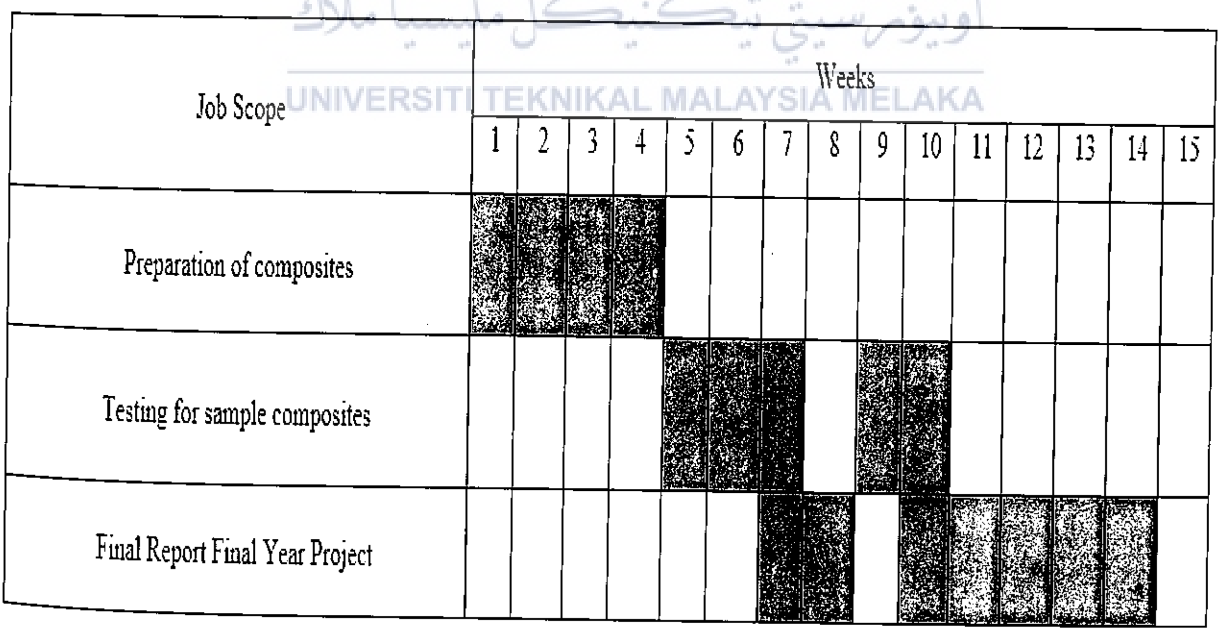

Figure 3.3: Gantt chart FYP 2

3.2 Materials

3.2.1 Pineapple leaves fibres

Pineapple leaves fibre were extracted from pineapple plant leaves, of which were obtained from Mersing, Johor. These pineapple fibres were extracted through a water retting process. The fibres were removed manually by retted the leaves in running water. Then, the fibres were then purified and dried in the sunlight. All fibres extraction processes were the same about using water retting process. Finally, the pineapple leaf fibres were prepared for testing to observe the potential as reinforcement materials.

3.2.2 Vinyl Ester

In this study, thermosetting resin has been chosen because of its specific properties. The type of resin used was vinyl ester (VE). The sources of the vinyl ester obtained from Polymer Technology Pte Ltd (Singapore). The properties of the vinyl ester which is the density is 1.6g/cc, viscosity is 400 cP, 120 °C temperature of heat distortion, and 104.44 to 143.33 °C temperature of glass transition. Vinyl ester is frequently found in industrial applications as anti-corrosion parts as example pipes, containers, tank and floor coverings.

3.2.3 Sodium hydroxide (NaOH) pellets

The type of alkali used was sodium hydroxide (NaOH) pellets. The sodium hydroxide pellets obtained from QRec (Asia) Sdn. Bhd., Malaysia. The NaOH solution were prepare for the alkaline treatment process in this study. The NaOH pellets are inorganic compounds that are highly water soluble and some polar solvents like ethanol and methanol. The NaOH solution obtained when the NaOH pellets are dissolved is colourless and odourless.

3.3 Composites preparation

3.3.1 Preparation of fibre and matrix

Before started the experiment, the first step is to process all the solution and fibre which is weight all the fibre and solution needed to create PALF. Figure 3.4, 3.5 and 3.6 shown weight of pineapple fibre, hardener and vinyl ester solution.

Figure 3.4: Weighing vinyl ester

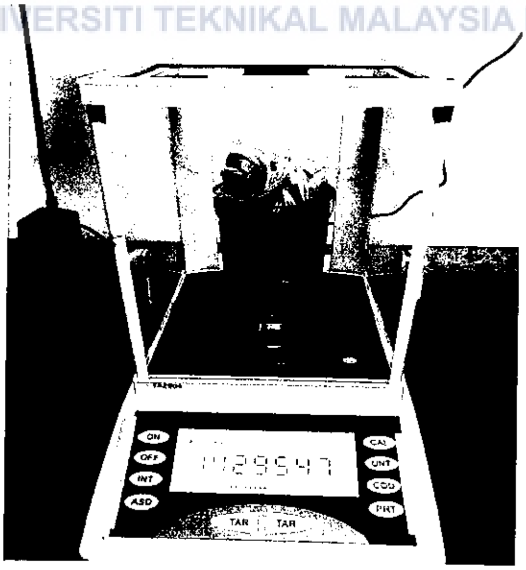

Figure 3.5: Weighing pineapple fibre

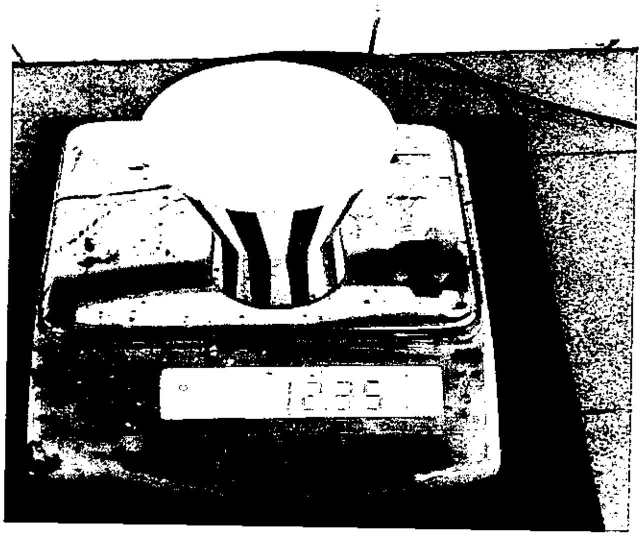

Figure 3.6:Weighing hardener

Table 3.1: Parameter of the samples

Element	Weight of composites (g)	Weight of fibre 5%(g)	Weight of resin (g)	MekP (Methyl ethyl ketone peroxide)
Neat polymer	100±5	-	98±5	2±0.5
Untreated	120±5	6±5	111.6±5	2.4±0.5
Treated	150±5	7.5±5	139.5±5	3±0.5

3.3.2 Preparation pineapple leaves fibre with treatment.

The PALF need to treated using alkali solution to remove impurities on the surface of PALF used to improve the surface properties of the fibres. At room temperature, distilled water was used to dissolved sodium hydroxide (NaOH) pellets to prepare the 6% NaOH solutions. Mettler Toledo. In order to achieve 6% NaOH solution, 94ml of distilled water mix with 6g of NaOH pellets and stir until all the pellet dissolved in the water.

After the NaOH pellets completely dissolved in the water, PALF was soaked into the NaOH solution for 24 hours. Then, clean the PALF using distilled water and let the fibre to dry at room temperature for 24 hours. Figure 3.7 show the complete cycle of the process to prepare the treated fibre.

Figure 3.7: Process treat fibre with NaOH

3.3.3 Fabrication of the composites.

The treated PALF need to clean up after drying process. This is because the fibres are tangled up together during the drying process after the alkaline treatment. Then, the treated PALF are being cut into 3cm long by using scissors before mix with the resin which is vinyl esters. The sample composition in this research is 5 wt % of the fibre loading which is optimum value for PALF. The percentage of the fibre loading calculate by using Eq 3.1.

$$Fibre\ weight\ for\ loading = \frac{Fibre\ loading\ wt\%}{100} \times fibre\ samples$$

Eq.3-1

After cleaning process, the treated and untreated PALF has been mix and stir using mechanical stirrer in the beaker. PALF fibre and vinyl ester was placed into the rectangular mould and using chopstick to level the surface and remove small bubble in the mixture. Then, using mylar sheet to cover the bottom and top of the samples before placed into the oven to obtain smooth surface finish. Figure 3.8 shows the process to put the PALF into mould start from cutting the fibre until put the PALF into the mould.

After that, the sample heated in the oven at temperature 150°C. After the heated has finished, the mould is then being cooled for about 15 minutes. The samples were heated again until vinyl ester is curing but cooled the sample 15 minutes before the sample can be taken out from the mould.

Figure 3.8: Process of putting the fibre into mould

3.4 Characterization and testing method.

3.4.1 Characteristic of Physical Properties

3.4.1.1 Density

The density of the PALF reinforced vinyl ester composites is determined by using the electronic densimeter. Figure 3.9 show the electronic densimeter at Universiti Teknikal Malaysia Melaka (UTeM).

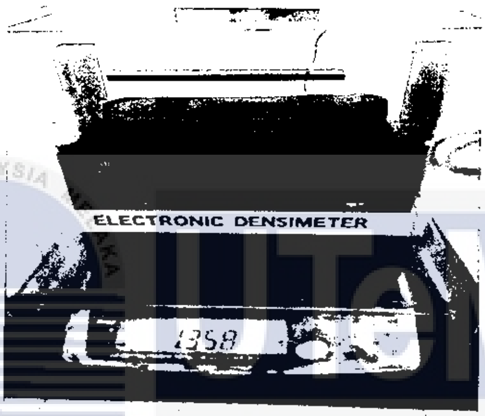

Figure 3.9:Electronic densimeter

The density(ρ) of PALF reinforced vinyl ester was determined by using 15 samples. At first, PALF reinforced vinyl ester was weighed (m) before being soaked into water. The volume of water (V) was get by measured amount of water before and after immersed. The value of m and V was used to calculate the density of PALF reinforced vinyl ester.

$$\rho = \frac{m}{v}$$

Eq. 3-2

Where

ρ is the density of fibre

m is the mass of fibre

V is the volume of water

3.4.1.2 Moisture content

15 samples were prepared for the moisture content evaluation. Before weight the fibres, the fibres were placed at room temperature (270°C + 20°C) for 24 hours with 65% relative humidity of air. The samples were heated in the oven at 105C for 24 hours. Before the samples were placed in the oven, the fibres weight was measured as *Mi.* After 24 hours in the oven, the fibres were then weighed again as *Mf.* Moisture content of the sample is determined through Eq.3-2.

$$Moisture\ content\ (\%) = \frac{Mi - Mf}{Mi} \times 100$$

Eq. 3-3

3.4.1.3 Water absorption

The average of percentage water absorption was calculated by using five samples for each parameter. By using Eq. 3-3, percentage of water absorption of PALF was determined. Firstly, the weight of the sample was weighed as *Wi* before immersed for 24 hours at room temperature in the fresh water. The samples were weighted again after 24 hours to get W_f value.

$$Water\ content\ \% = \frac{W_f - W_i}{W_i} \times 100$$

Eq. 3-4

3.4.2 Mechanical Testing

3.4.2.1 Tensile Test

The simple way to know the mechanical properties of the natural fibre by perform the tensile test. Mechanical properties can be gained from tensile test such as tensile stress, Young's modulus, maximum elongation, yield stress and tensile strain. For this research, the testing was carried out by following to ASTM-D3039(Nagarajan *et al.*, 2016). The tensile test of PALF reinforced vinyl ester determined by using the Instron Universal Testing Machine, 5980 Series in figure 3.10. The result is then obtained through the Bluehill 2 software.

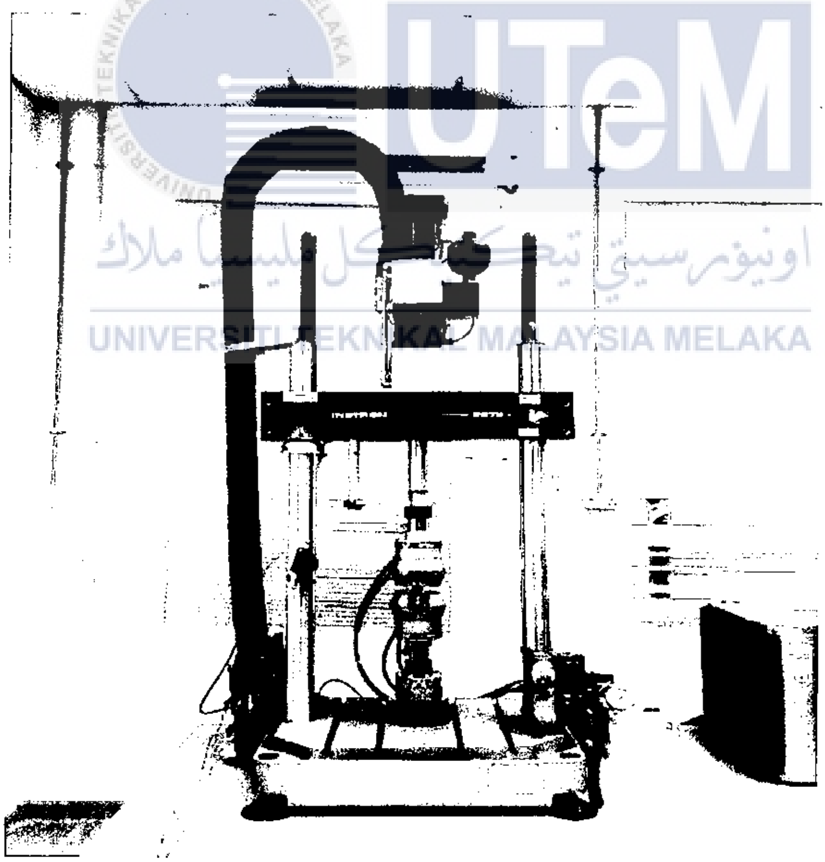

Figure 3.10: Instron Universal Testing Machine, Model 5980 Series

The tensile strength of the single fibre can be calculated using equation 3-4.

$$\sigma = \frac{F}{A}$$

Eq. 3-4

Where,

σ = tensile strength of the fibre (Pa)

F = maximum force at break (N)

A = area of fibre cross-section (m^2)

3.4.2.2 Flexural test

Flexural test was conducted by using a three-point bending set-up as stated by the ASTM-D790(Mishra *et al.*, 2001). Five sample from 0 wt%, untreated and treated fibre is tested using Instron Universal Testing Machine, 5585 Series with a load speed of 1mm/min. The reading of the flexural strength and modulus is recorded through the Bluehill 2 software. Figure 3.11 shows the Universal Testing Machine Model Instron 5585 at Universiti Teknikal Malaysia Melaka.

Figure 3.11: Universal Testing Machine model Instron 5585

3.5 Morphological Analysis

Morphological studies of the impact test samples have been carried out in detail on the fractured surface. The test was performed at accelerating voltage of 15kV using Scanning Electron Microscope (SEM). The sample selected are the sample from tensile test which is one samples for each parameter. Platinum were used to coat the samples to provide electrical conductivity, which did not affect the resolution significantly, allowing high-quality results.

CHAPTER 4

RESULT AND DISCUSSIONS

4.1 Introduction

In this section, the physical and mechanical properties will be discussing and evaluate according to the result from the testing. Physical characterization can be divided into 3 which are density, moisture content and water absorption and mechanical characterization includes tensile and flexural test.

4.2 Physical properties

Density, moisture content and water absorption one of the main factors that can affect the properties of the composite's product. In this section, the result for the three physical testing that has been performed on the PALF reinforced vinyl ester composites will be analysed.

4.2.1 Density

Density of an object can be measure by knowing the mass and volume of the object. The mass of the object can be determining from density so it is important to know the density of materials because it can affect the weight of the object. Table 4.1 show the value of the specific gravity from the electronic densimeter machine. The density of the fibre can be calculated using Eq.4-1.

$$Specific\ gravity = \frac{Density\ of\ the\ object}{Density\ of\ the\ water}$$

Eq. 4-1

Table 4.1: Specific gravity data

No	Vinyl ester	Untreated	Treated
1	1.158	1.128	1.187
2	1.104	1.172	1.125
3	1.102	1.157	1.179
4	1.103	1.162	1.191
5	1.139	1.163	1.199

From the figure 4.1, the density of the composites will increase when the amount of the fibre increases in the composites. Density of the composites keep increasing from neat polymer to treated composites which is for treated is 1.1762 g/cm^3, untreated composites is 1.1562 g/cm^3 and neat polymer is 1.1212 g/cm^3. Based on the table 3.1, the amount of the composites is different for the neat polymer, untreated and treated composites. It can be concluded, the different amount of fibre in the composites can affect the density of the composites.

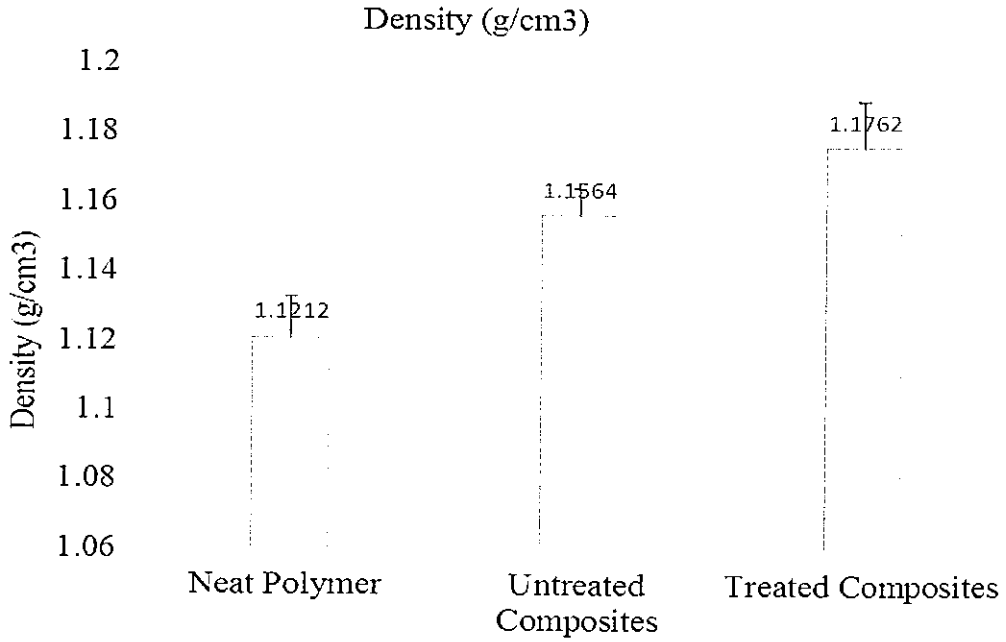

Figure 4.1: Density of the samples

4.2.2 Moisture content

One of the important aspects to consider before choosing the natural fibre as reinforced materials is moisture content of the natural fibre. The stability, tensile strength, porosity and swelling behaviour of the natural fibre affects by the moisture content. Natural fibre that has lower moisture content that composites combined with high moisture content are less likely to declined due to characteristic of the fibre to absorb water(Razali *et al.*, 2015).

There have two main part of chemical content related with absorption of water in fibre which is lignin and hemicellulose. Lignin has lower attraction to water but different for hemicellulose which is high attraction to water and hold water molecules in the fibre. This is because of the hydroxyl bonding or OH group in the cellulose structure(Razali *et al.*, 2015).

The result of moisture content of PALF indicated that, treated composites has low moisture content compared with the untreated composites which is 9 % and 19.98 %, as shown in figure 4.2. Neat polymer has lower percentages of moisture content because vinyl ester has hydrophobic characteristic which is will never absorb water but there has value because of the poor surface finish. Therefore, treated composites is ready for fabricating composites product with high quality and stability. This is because the higher percentage of moisture content in untreated composites indicate critical weight changes of the composites after place in oven for 24 hours. If the weight of the composites become lighter after 24 hours, it indicates that water in the composites is fully dry. This is proved that highly amount of water hold inside the fibre for untreated composites. The percentages of moisture content for treated composites is low because the weight of the samples not changing much due to the alkaline treatment has been perform on the fibre that reduce the tendency of the cellulose to absorb water.

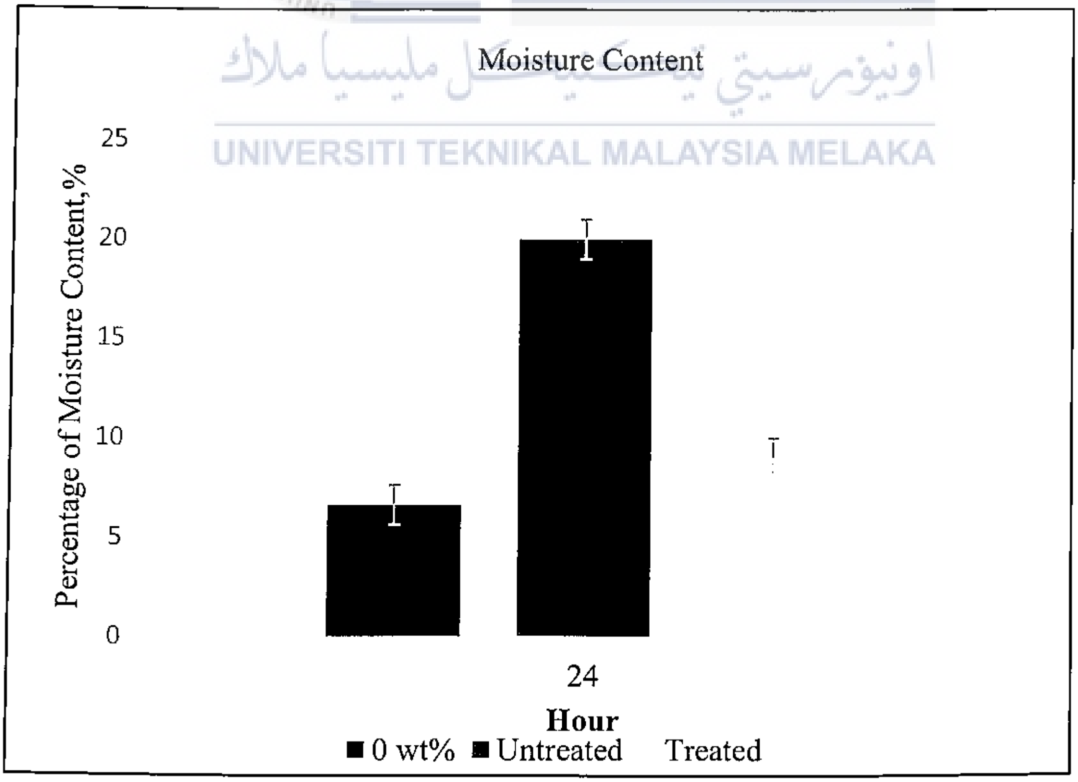

Figure 4.2 : Moisture content of composites and vinyl ester

4.2.3 Water absorption

Hydrophilic is characteristic of fibres to absorb moisture from environment until equilibrium is established. The moisture absorption can change dimensional variations in fibre and affecting the mechanical and interface of composites material. Composites without chemical treatment have poor fibre -matrix interface and no practicable for transfer of load due to poor interface.

Hydrophilic is derived from lignocellulose which strongly polarised with hydroxyl groups. If the wetting of the fibre-matrix occurs, the weak interfaces between two phases will occurred. Chemical treatment on fibre can help fibres to have interfacial bonding and reduce its moisture regain.

In this experiment, the tendency of PALF to absorb water was tested by using moisture content technique which is place the sample inside water for 24 hours. The reading weight of sample is taken every 2 hours in order to get accurate data. The last weight of the sample will indicate amount of water enter the composites and swell. The presence of the water inside the composites due to the micro crack on the surface of samples.

The sample dimension for this testing is 10mm × 10mm × 3mm and the percentages of moisture content was calculated by using Eq 3-3. Figure 4.3 shows the process of the water absorption testing for vinyl ester, untreated fibre and treated fibre. Blue cup is for vinyl ester sample, green cup is for untreated fibre and white cup is for treated fibre.

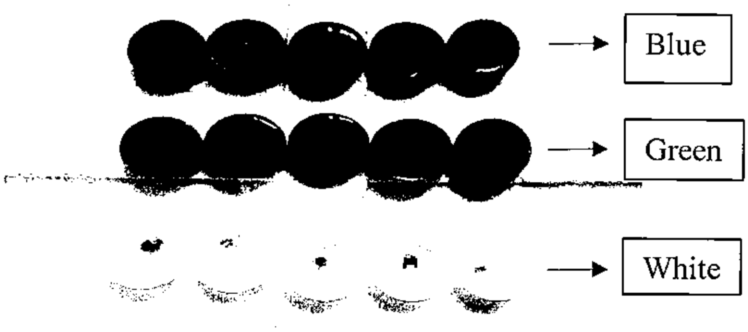

Figure 4.3: Water absorption sample

Based on the result from figure 4.4, untreated composites have high percentage of water absorption compare with the treated composites and neat polymer. This is because the untreated fibres contain of cellulose which is have higher chemical content that cannot resist water and will absorb more water than treated. More water will penetrate to the fibre-matrix interphase due to higher content of cellulose which can cause to crack in microstructure and swelling. Small cracking will because large cracking due to the higher amount of water.

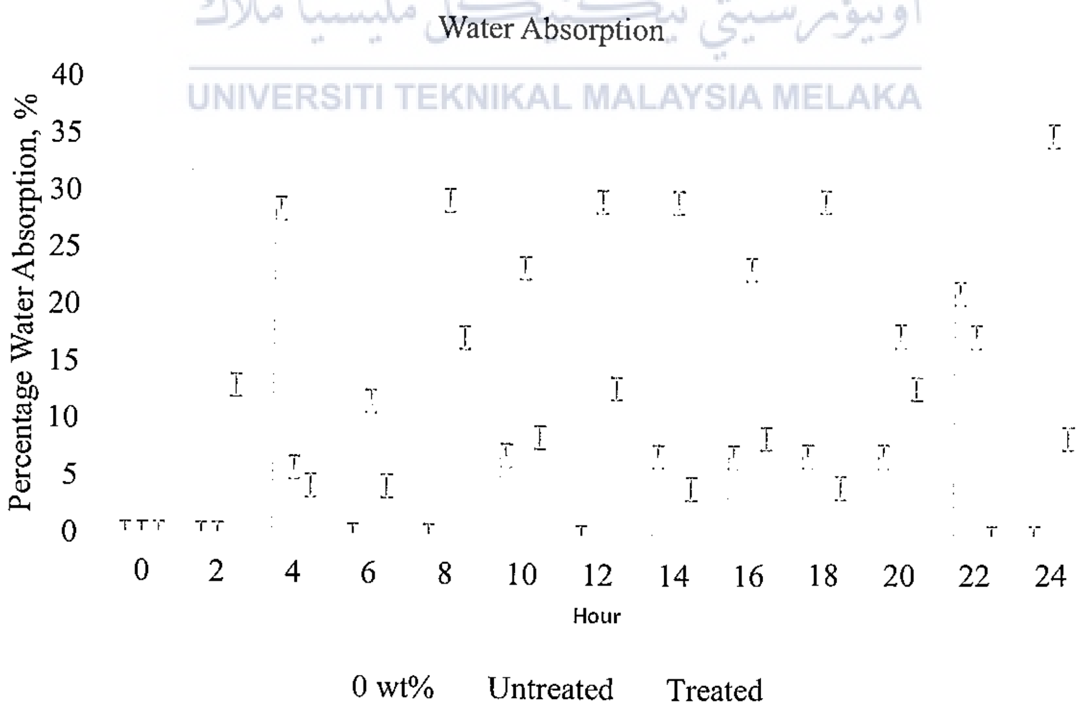

Figure 4.4: Bar graph of the water absorption testing

However, the adhesion bonding between fibre and vinyl ester can be improve with surface treated of natural fibre. Based on the result, treated composites has low water absorption percentage because the surface of the fibre has been treated with 6% NaOH solution. Alkaline treatment on the natural fibre will reduce the tendency of fibre to absorb water by remove lignin and hemicellulose components from natural fibre and increase the mechanical properties of the fibre.

As the result form bar graph, the water absorption percentage increase with time. The longer the fibre expose to the water, the higher water it will absorb. Other than that, chemical treatment also will reduce the amount of water absorb by the natural fibre via improve the surface of the fibre and remove all the impurities. The neat polymer characteristic is different from the untreated fibre because neat polymer can resist water because polymer is hydrophobic characteristic.

4.3 Mechanical properties

Mechanical testing is performed in order to study the behaviour of composites under different loads. For this research, the involve mechanical testing are tensile testing and flexural testing

4.3.1 Tensile testing

Tensile test is used to evaluate the behaviour of the fibre sample under tension load and to determine whether the sample have good in interfacial bonding. The value of ultimate tensile strength of material was determined by pulled the sample to breaking point. This test can measure the amount of force and elongation applied to the sample. Figure 4.5 shows that the position of the sample clamp on the tensile machine.

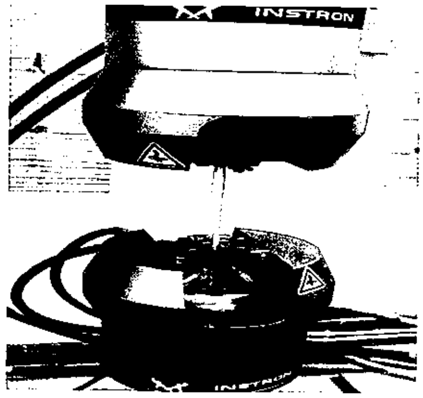

Figure 4.5: Sample position on the machine

Tensile test was carried out according to ASTM D3039 standard using Universal Testing Machine model Instron 8872, equipped with load cell of 5kN, with crosshead 1mm/min. For each composite, five samples from each parameter which is total sample is fifteen with dimensions 150×15×3 mm were tested.

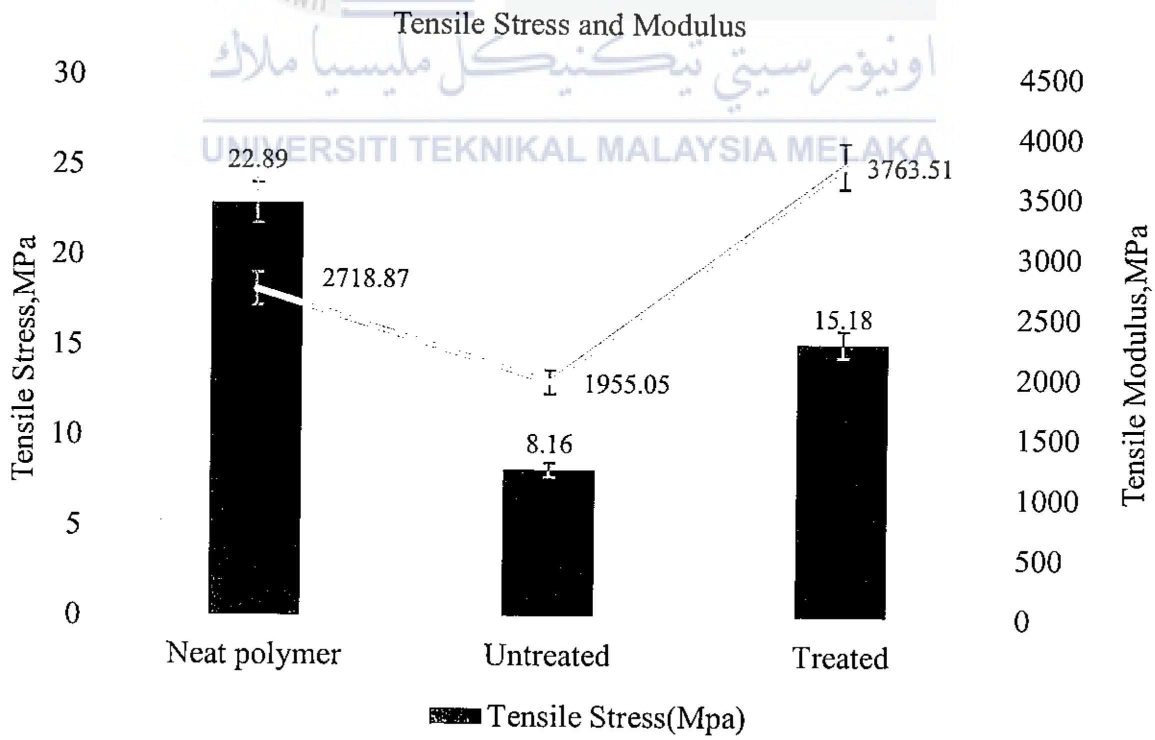

Figure 4.6:Tensile Stress and Tensile Modulus bar graph

The bar graph in figure 4.6 show that the tensile stress of the treated composites higher than untreated composites which is 15MPa compared with the 8MPa. The result between treated and untreated of tensile stress and modulus show highest result on the treated composites. This is because alkaline treatment on fibre can remove hemicelluloses and lignin and enhance surface area for fibre/matrix adhesion(M Asim1, M. Jawaid1, 2018). The tendency of the fibre to absorb water also reduce due to alkaline treatment but at certain concentration it can increase the tensile properties of the fibre.

The composites become more brittle when increasing the pineapple fibre as reinforcement and will show brittle behaviour and will decrease overall strength. The impurities efficiently can be removing by using high chemical concentration but will reduce the tensile strength cause to lignocelluloses degradation and rupture of fibre surface(M Asim1, M. Jawaid1, 2018). However, the data show that treated fibre gives better strength compared with the untreated fibre.

Strong interfacial bonding between fibre and polymer and good stress dispersion of load the towards the composites because of the increase in tensile stress and modulus due to alkaline treatment. The increase of mechanical interlocking bonding because of the strong fibre and thermoset bonding. The efficiency of a fibre reinforced composited depends on the fibre-matrix interface and the ability to transfer stress from the matrix to the fibre(Siregar *et al.*, 2015). The good adhesion between two phases due to clear surface of treated fibre with enhancement of mechanical properties.

Table 4.2: Comparison value of tensile strength for theoretical and experiment

No	Theoretical value of tensile strength (MPa)	Experimental value of tensile strength (MPa)
1	100	15

There has different value for theoretical and experimental which is experimental value is lower than the theoretical value. This is because the fibres were not perfectly aligned, and composites were poorly prepared(Kasim *et al.*, 2016). Other than that, the different value also due to bad surface finishing on the sample because of the not proper surface finishing during fabrication process.

4.3.2 Flexural testing

The sample was tested to evaluate the flexural properties of fibre via strength and modulus value of the samples according to ASTM D790(Sood and Dwivedi, 2018). The sample is test by using universal testing machine (SEM) by three-point bending technique. The three-point position of the sample during the testing was shown in figure 4.7. This three-point technique can evaluate flexural stress, modulus of elasticity and flexural strain. The crosshead speed for this testing is 1mm/min.

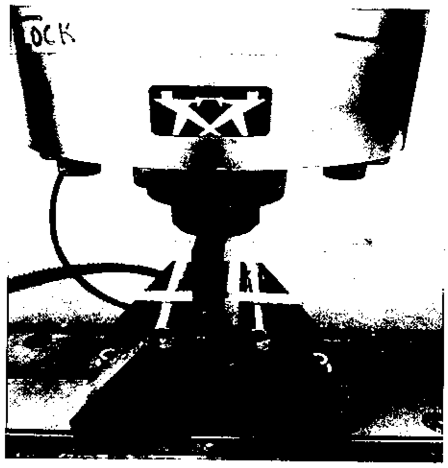

Figure 4.7: Sample position for three-point testing

Flexural testing was performed to determine the strength and the ability of the material to resist the deformation under loading before reach the break point. Figure 4.8 show the flexural testing value of flexural stress and flexural modulus for zero wt%, untreated and treated PALF.

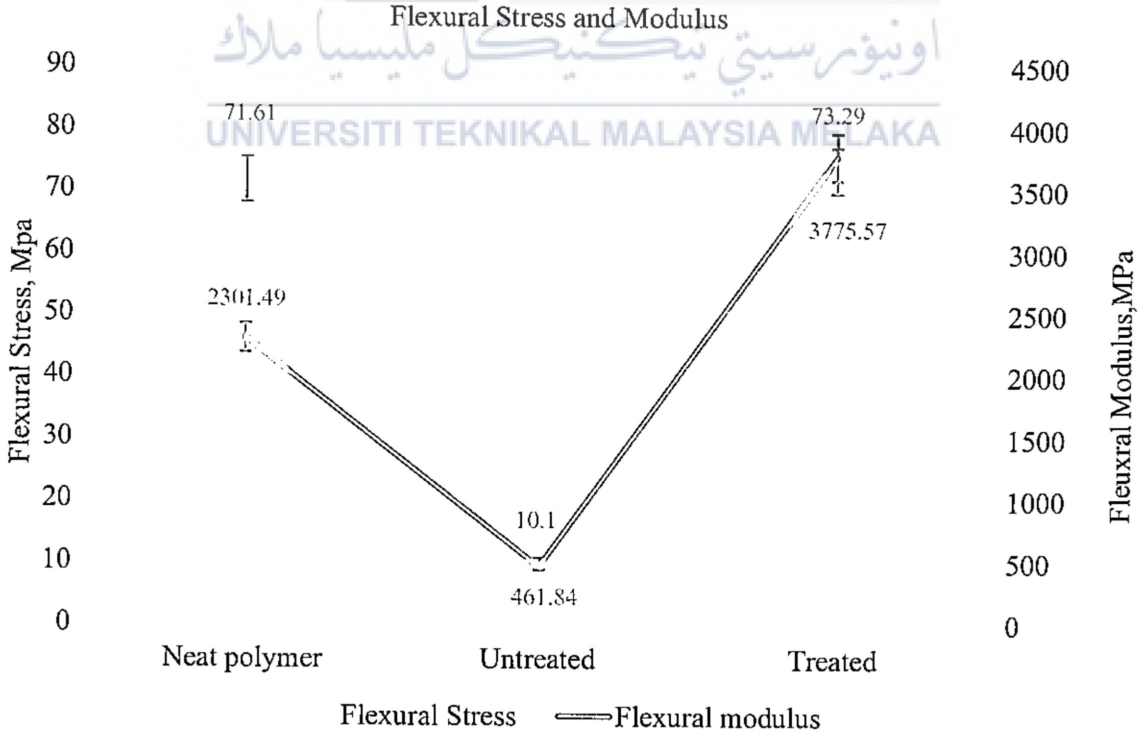

Figure 4.8: Flexural strength and modulus of pineapple leaf reinforced vinyl ester

Based on the graph in figure 4.6, flexural strength and modulus of treated fibre sample has higher value compared with the untreated and vinyl ester sample. This is proved that alkaline treatment can improved the flexural strength and modulus of the composites. The treatment will remove the external fibre on surface and improve fibre matrix bonding by exposed more cellulose content to the surface, resulting in higher strength of composites treated than untreated fibre composites. The difference value between treated and untreated is 86.22 % which is 73.28 MPa and 10.10 MPa. For flexural modulus the different is about 87% which is 3775.57 MPa and 461.84 MPa. However, the flexural stress and modulus of the vinyl ester still higher than untreated fibre due to the gap between the fibre and vinyl ester. The gap normally occurs due the wetting problem of the untreated fibre that led to the weak bonding between them.

The bonding of interaction linkage between fibre and matrix for untreated composites weak due to the untreated fibre with alkaline so the strength of fibre is lower than treated. The good mechanical interlocking can obtain when the surface modification is done towards the fibre surface. The treatment can improve the compatibility between reinforcement and matrix. Positive effects can be seen from alkaline treatment more to flexural strength compared to the flexural modulus of fibre reinforced vinyl ester(Sood and Dwivedi, 2018). As conclusion, the alkaline treatment of natural fibre will increase the strength of the natural fibre.

4.4 Morphological

4.4.1 Scanning Electron Microscope (SEM)

Scanning Electron Microscope (SEM) was used to investigate the effect on the surface morphology of the alkaline treatment and the interfacial bonding between fibre and matrix(Nadlene *et al.*, 2016). The sample use in this analysis is from tensile test sample which is one sample for one parameter. From the result, the surface fracture of the vinyl ester was much smoother than the addition of pineapple leaf fibre in the matrix. The characteristic of the vinyl ester was in brittle manner and smooth. Figure 4.9(a) shows that there is no gap or any hole in the vinyl ester fracture surface because there is no presence of bubble in the solution during fabrication process.

From figure 4.9(b), it can be observed the presence of the fibre pull-out and gap between PALF and vinyl ester. This situation can attribute to poor adhesion between fibre and vinyl ester resin. Therefore, the tensile strength of the untreated composites is lower than the treated composite. The gap occur because of the fibre has been pull out from the matrix during the tensile test. Untreated fibre has impurities, wax and there are many burrs on the fibre surfaces(Nadlene *et al.*, 2016). All the impurities and wax on the surface of the fibre can be remove using alkaline treatment and increase the adhesion of the matrix.

Other than that, the surface of the untreated fibre also rougher than treated fibre with alkaline treatment. A group of the PALF was separate from the vinyl ester also can be observed. This is due to the wetting problem of the untreated fibre because natural fibre is hydrophilic characteristics which is the tendency of to absorb water is high for natural fibre. This will reduce the interfacial bonding between fibre and resin.

Figure 4.9(c) shows the result of SEM from treated of the PALF with 6 % NaOH to remove all the impurities and wax at the surface of the fibre. From the figure, the treated fibre has better dispersion of PALF in the matrix compared with the untreated fibre. Other than that, the dewaxing process of fibre can reduce fibre pull-out because the fibre and matrix adhesion has been improved, which can be seen in figure 4.9(c). The fracture surface results indicate that the fibre and matrices are distributed equally that can led to the highest in value of flexural and tensile strength. There is no presence of gap in the treated sample so treated fibre has higher interfacial adhesion between fibre and matrix, resulting in good mechanical properties such as no fibre pull-out, less gap between fibre and matrix and less crack on the composites surface.

As conclusion, alkaline treatment can clean the fibre surface from impurities because remove the impurities can increase the interfacial binding between fibre and matrix that can cause cracking on the surface.

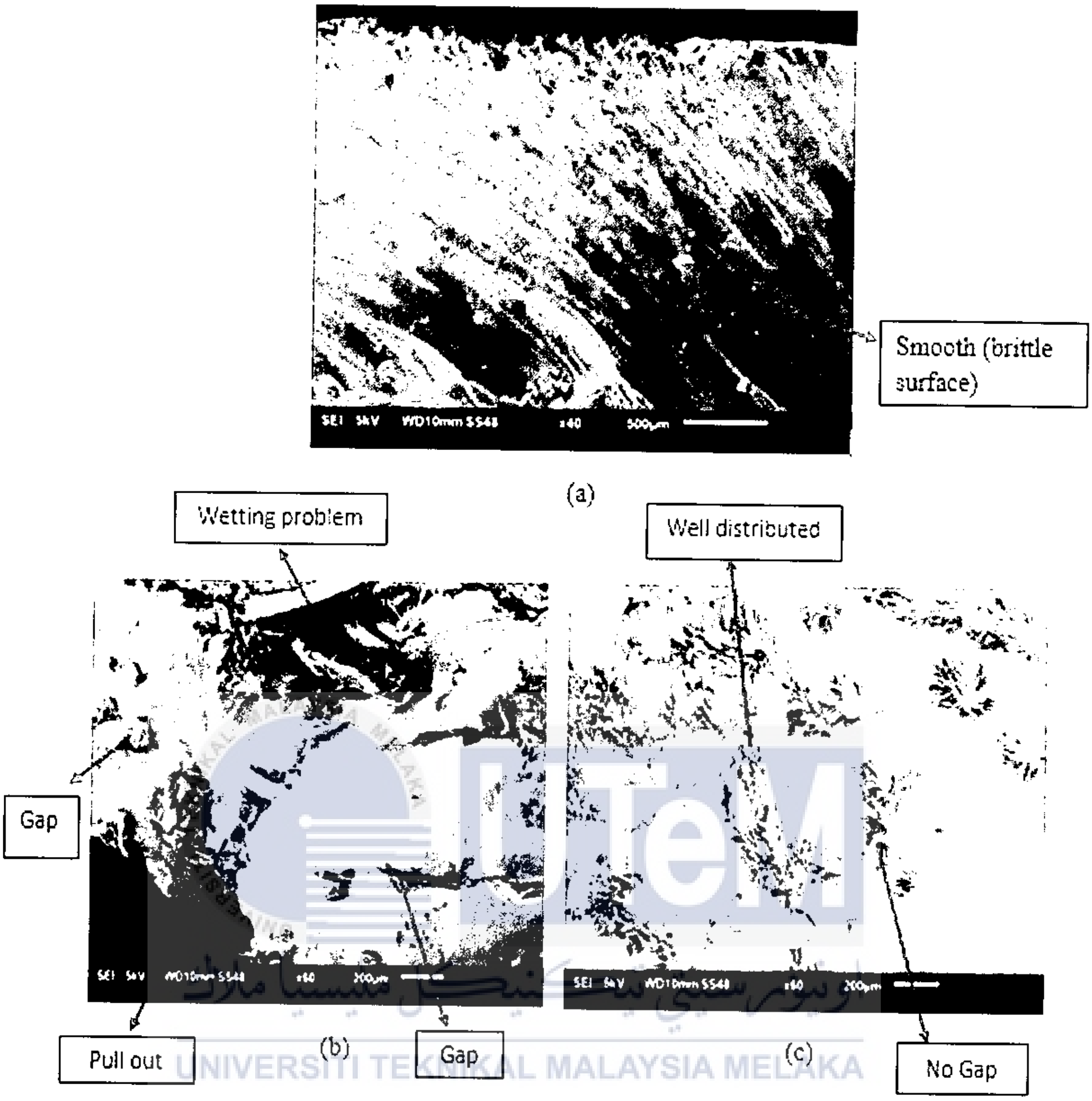

Figure 4.9 :Scanning Electron Micrographs of PALF subjected to different parameter :
a) vinyl ester b) untreated fibre c) treated fibre

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

There have few researches has been conducted related with alkaline treatment on the pineapple leaf fibre but focusing on the concentration of the NaOH solution. In this research, it is more focusing on the optimum parameter of the alkaline treatment which is 6% of the NaOH. The effects of alkaline treatment of the PALF reinforced vinyl ester on physical, mechanical and morphological properties were analysed.

In this research, the fibre loading for the PALF is 5 wt% has been chosen for untreated and treated composites samples. For physical testing, the samples been investigated through the density, moisture content and water absorption properties of the composites. In mechanical testing, the samples been analysed through tensile and flexural testing. Based on previous researches, treated composites has better mechanical and physical properties compared with the untreated composites due to the strong adhesion between fibre and matrix.

Based on the physical properties result, treated composites has better properties compared with the untreated composites. Treated composites has lower percentages of the water absorption and moisture content. This is show that the alkaline treatment on the natural fibre will reduce the tendency of fibre to absorb water by remove lignin and hemicellulose components from natural fibre and increase the mechanical properties of the fibre.

There has been a positive enhancement in the tensile strength and flexural properties for the mechanical properties of the treated PALF reinforced vinyl ester. Tensile test result show that treated composites has better strength compare with the untreated composites. This is show that the alkaline treatment on the fibre enhanced interfacial bonding between fibre and polymer and good stress dispersion of load the towards the composites. This is support by SEM result, treated fibre show less fibre pull-out, no gap between fibre and all the fibre well distributed in the samples.

For future study, the parameter of the fibre loading of PALF reinforced vinyl ester should be increase such as 5 wt%, 10wt%, 20 wt% and 50wt% in order to analyses which fibre loading has better properties in term of physical and mechanical. Other than that, further study about the other chemical treatment that can be perform on the PALF in order to produce high quality and stability of fibre for future use in industry production.

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APPENDICES

Designation: D 3039/D 3039M – 00ε1

Standard Test Method for Tensile Properties of Polymer Matrix Composite Materials[1]

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 Displacement[2]

D 883 Terminology Relating to Plastics[2]

D 2584 Test Method for Ignition Loss of Cured Reinforced Resins[3]

D 2734 Test Method for Void Content of Reinforced Plastics[3]

D 3171 Test Methods for Constituent Content of Composites Materials[4]

D 3878 Terminology for Composite Materials[4]

D 5229/D 5229M Test Method for Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[4]

E 4 Practices for Force Verification of Testing Machines[5]

E 6 Terminology Relating to Methods of Mechanical Testing[5]

E 83 Practice for Verification and Classification of Extensometers[5]

E 111 Test Method for Young's Modulus, Tangent Modulus, and Chord Modulus[5]

E 122 Practice for Choice of Sample Size to Estimate a Measure of Quality for a Lot or Process[6]

E 132 Test Method for Poisson's Ratio at Room Temperature[5]

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[6]

E 251 Test Methods for Performance Characteristics of Metallic Bonded Resistance Strain Gages[5]

E 456 Terminology Relating to Quality and Statistics[6]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

E 1012 Practice for Verification of Specimen Alignment Under Tensile Loading[5]

E 1237 Guide for Installing Bonded Resistance Strain Gages[5]

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.

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[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.

1

Designation: D 790 – 03

Standard Test Methods for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials[1]

This standard is issued under the fixed designation D 790; 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.

1. Scope*

1.1 These test methods cover the determination of flexural properties of unreinforced and reinforced plastics, including high-modulus composites and electrical insulating materials in the form of rectangular bars molded directly or cut from sheets, plates, or molded shapes. These test methods are generally applicable to both rigid and semirigid materials. However, flexural strength cannot be determined for those materials that do not break or that do not fail in the outer surface of the test specimen within the 5.0 % strain limit of these test methods. These test methods utilize a three-point loading system applied to a simply supported beam. A four-point loading system method can be found in Test Method D 6272.

1.1.1 *Procedure A*, designed principally for materials that break at comparatively small deflections.

1.1.2 *Procedure B*, designed particularly for those materials that undergo large deflections during testing.

1.1.3 Procedure A shall be used for measurement of flexural properties, particularly flexural modulus, unless the material specification states otherwise. Procedure B may be used for measurement of flexural strength only. Tangent modulus data obtained by Procedure A tends to exhibit lower standard deviations than comparable data obtained by means of Procedure B.

1.2 Comparative tests may be run in accordance with either procedure, provided that the procedure is found satisfactory for the material being tested.

1.3 The values stated in SI units are to be regarded as the standard. The values provided in parentheses are for information only.

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.*

NOTE 1—These test methods are not technically equivalent to ISO 178.

2. Referenced Documents

2.1 *ASTM Standards:*

D 618 Practice for Conditioning Plastics for Testing[2]

D 638 Test Method for Tensile Properties of Plastics[2]

D 883 Terminology Relating to Plastics[2]

D 4000 Classification System for Specifying Plastic Materials[3]

D 5947 Test Methods for Physical Dimensions of Solid Plastic Specimens[4]

D 6272 Test Method for Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials by Four-Point Bending[4]

E 4 Practices for Force Verification of Testing Machines[5]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[6]

3. Terminology

3.1 *Definitions*—Definitions of terms applying to these test methods appear in Terminology D 883 and Annex A1 of Test Method D 638.

4. Summary of Test Method

4.1 A bar of rectangular cross section rests on two supports and is loaded by means of a loading nose midway between the supports (see Fig. 1). A support span-to-depth ratio of 16:1 shall be used unless there is reason to suspect that a larger span-to-depth ratio may be required, as may be the case for certain laminated materials (see Section 7 and Note 8 for guidance).

4.2 The specimen is deflected until rupture occurs in the outer surface of the test specimen or until a maximum strain (see 12.7) of 5.0 % is reached, whichever occurs first.

4.3 Procedure A employs a strain rate of 0.01 mm/mm/min [0.01 in./in./min] and is the preferred procedure for this test method, while Procedure B employs a strain rate of 0.10 mm/mm/min [0.10 in./in./min].

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.10 on Mechanical Properties.

Current edition approved March 10, 2003. Published April 2003. Originally approved in 1970. Last previous edition approved in 2002 as D 790 – 02.

[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 08.03.

[5] *Annual Book of ASTM Standards*, Vol 03.01.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

*A Summary of Changes section appears at the end of this standard.

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

Standard Test Method for
Moisture Absorption Properties and Equilibrium Conditioning of Polymer Matrix Composite Materials[1]

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.*

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[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.

Designation: D 570 – 98

Standard Test Method for
Water Absorption of Plastics[1]

This standard is issued under the fixed designation D 570; 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 covers the determination of the relative rate of absorption of water by plastics when immersed. This test method is intended to apply to the testing of all types of plastics, including cast, hot-molded, and cold-molded resinous products, and both homogeneous and laminated plastics in rod and tube form and in sheets 0.13 mm (0.005 in.) or greater in thickness.

1.2 The values given in SI units are to be regarded as the standard. The values stated in parentheses are for information only.

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.*

NOTE 1—ISO 62 is technically equivalent to this test method.

2. Referenced Documents

2.1 *ASTM Standards:*

D 647 Practice for Design of Molds for Test Specimens of Plastic Molding Materials[2]

2.2 *ISO Standard*

ISO 62 Plastics—Determination of Water Absorption[3]

3. Significance and Use

3.1 This test method for rate of water absorption has two chief functions: first, as a guide to the proportion of water absorbed by a material and consequently, in those cases where the relationships between moisture and electrical or mechanical properties, dimensions, or appearance have been determined, as a guide to the effects of exposure to water or humid conditions on such properties; and second, as a control test on the uniformity of a product. This second function is particularly applicable to sheet, rod, and tube arms when the test is made on the finished product.

3.2 Comparison of water absorption values of various plastics can be made on the basis of values obtained in accordance with 7.1 and 7.4.

3.3 Ideal diffusion of liquids[4] into polymers is a function of the square root of immersion time. Time to saturation is strongly dependent on specimen thickness. For example, Table 1 shows the time to approximate time saturation for various thickness of nylon-6.

3.4 The moisture content of a plastic is very intimately related to such properties as electrical insulation resistance, dielectric losses, mechanical strength, appearance, and dimensions. The effect upon these properties of change in moisture content due to water absorption depends largely on the type of exposure (by immersion in water or by exposure to high humidity), shape of the part, and inherent properties of the plastic. With nonhomogeneous materials, such as laminated forms, the rate of water absorption may be widely different through each edge and surface. Even for otherwise homogeneous materials, it may be slightly greater through cut edges than through molded surfaces. Consequently, attempts to correlate water absorption with the surface area must generally be limited to closely related materials and to similarly shaped specimens. For materials of widely varying density, relation between water-absorption values on a volume as well as a weight basis may need to be considered.

4. Apparatus

4.1 *Balance*—An analytical balance capable of reading 0.0001 g.

4.2 *Oven*, capable of maintaining uniform temperatures of 50 ± 3°C (122 ± 5.4°F) and of 105 to 110°C (221 to 230°F).

5. Test Specimen

5.1 The test specimen for molded plastics shall be in the form of a disk 50.8 mm (2 in.) in diameter and 3.2 mm (1/8 in.) in thickness (see Note 2). Permissible variations in thickness are ±0.18 mm (±0.007 in.) for hot-molded and ±0.30 mm (±0.012 in.) for cold-molded or cast materials.

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[1] This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D 20.50 on Permanence Properties.

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[2] Discontinued 1994; replaced by D 1896, D 3419, D 3641, D 4703, and D 5227. See 1994 *Annual Book of ASTM Standards*, Vol 08.01.

[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[4] Additional information regarding diffusion of liquids in polymers can be found in the following references: (1) *Diffusion, Mass Transfer in Fluid Systems*, E. L. Cussler, Cambridge University Press, 1984, ISBN 0-521-29846-6, (2) *Diffusion in Polymers*, J. Crank and G. S. Park, Academic Press, 1968, and (3) "Permeation, Diffusion, and Sorption of Gases and Vapors," R. M. Felder and G. S. Huvard, in *Methods of Experimental Physics*, Vol 16C, 1980, Academic Press.