THE EFFECT OF ALKALINE TREATMENT ON THE MECHANICAL AND PHYSICAL PROPERTIES OF KENAF FIBRE REINFORCED VINYL ESTER COMPOSITES

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This report is submitted
in fulfillment of the requirement for the degree of
Bachelor of Mechanical Engineering with Honors (Structure & Materials)

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Faculty of Mechanical Engineering

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2019

DECLARATION

I declare that this thesis entitled “The Effect of Alkaline Treatment on The Mechanical and Physical Properties of Kenaf Fibre Reinforced Vinyl Ester Composites” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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APPROVAL

I hereby declare that I have read this dissertation/report and in my opinion this dissertation/report is sufficient in terms of scope and quality as a partial fulfilment of Bachelor of Mechanical Engineering with Honors (Structure & Materials).

Signature : ....

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ABSTRACT

Natural fibre nowadays having a high demand in any application development. The purpose in usage natural fibre in worldwide application is one of alternative way in order to replace the manufacturing using synthetic fibre. Natural fibres have many advantages including having low cost and easy to process. Next, the greenhouse effect, pollution percentages that occur and health hazard of human, flora and fauna should be reconsidering in any production requirement and become the main reasons natural fibre demand is rising dramatically. Despite that, the terms of polymer matric composites or known as PMCs, using natural fibre as reinforce agent are trending in aeronautic nor automotive industries. Hence, this research is focusing on the effect of chemical treatment kenaf fibre reinforced vinyl ester composites. As the experiment is done, the result data show that treated natural fibre have well in strength, modulus and also physical properties such as water absorption. From flexural experiment data by comparing with untreated, the strength of treated natural fibre improve at 55.46% while for modulus increase at 83.87%. Other than that, the strength distribution of fibre also has a good improvement after conduct tensile test and giving tensile stress of treated is better compared with untreated composites with increasing around 10.93%. For water absorption, the result shows that the treated kenaf fibre can resist water better than untreated one. The surface modification towards natural fibre using alkaline treatment really prove that it has good result compared to untreated natural fibre in terms of enhancement mechanical interlocking between natural fibre and matrix and reduction of chemical content of natural fibre that can reduce the natural characteristic which is hydrophilic.

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ABSTRAK

Serat semulajadi pada masa kini mempunyai permintaan yang tinggi dalam pembangunan ekonomi. Tujuan penggunaan serat semulajadi dalam aplikasi di seluruh dunia adalah salah satu cara alternatif untuk menggantikan pembuatan menggunakan serat sintetik iaitu serat yang diperbuat dan digubal oleh manusia. Serat semulajadi mempunyai banyak kelebihan termasuk kos yang rendah dan bahannya juga yang mudah diproses. Seterusnya, kesan rumah hijau, kadar peratusan pencemarah yang berlaku serta tahap kesihatan manusia, flora dan fauna juga harus dipertimbangkan dalam setiap pengeluaran yang telah diproses dan secara tidak langsung menjadikan serat semulajadi salah satu bahan yang menjadi permintaan yang tinggi. Walaubagaimanapun, terma komposit matrik polimer (PMC) yang menggunakan serat semulajadi sebagai agen pengukuhan sedang giat berjalan dalam industri aeronatik mahupun automotif. Oleh itu, kajian kesan rawatan permukaan gentian serat kenaf ini telah mendapat tumpuan yang lebih terperinci. Apabila percubaan dilakukan, hasil data menunjukkan bahawa gentian asli yang dirawat mempunyai kekuatan fizikal, kekuatan modulus dan sifat fizikal seperti penyerapan air yang baik berbanding yang tidak dirawat. Daripada eksperimen lenturan jika dibandingkan dengan yang tidak dirawat, kekuatan serat semulajadi yang dirawat meningkat pada 55.46% manakala peningkatan modulus pada 83.87%. Selain itu pengagihan kekuatan terhadap serat juga mempunyai peningkatan yang baik selepas menjalankan ujian tegangan dengan penambahan sebanyak 10.93%. Untuk eksperimen penyerapan air, hasil menunjukkan bahawa serat kenaf yang dirawat dapat mengahalang penyerapan air dengan lebih baik. Rawatan permukaan ke serat semulajadi menggunakan rawatan alkali benar-benar mebuktikan bahawa ia boleh mendapat hasil yang baik berbanding permukaan yang tidak dirawat dari segi mekanisme hubungkait antara rangkai serat dan juga matriks selain dapat mengurangkan juga kadar kimia dalam serat semulajadi dimana salah satunya adalah hidrofilik.

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LIST OF ABBREVIATIONS

Abbreviation	
CMCs	Ceramic Matrix Composites
MMCs	Metal Matrix Composites
PMCs	Polymer Matrix Composites
FRP	Fibre Reinforced Polymer
UV	Ultraviolet
PVC	Polyvinyl Chloride
PP	Polypropylene
PE	Polyester
VE	Vinyl Ester
VARTM	Vacuum Assisted Resin Transfer Molding
NaOH	Sodium Hydroxide
ASTM	American Society for Testing Materials
SEM	Scanning Electron Microscope

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LIST OF SYMBOLS

Symbol	
σ	Tensile Strength (MPa)
F	Force (N)
V	Volume (m^3)
m	Mass (g)
A	Area (m^3)
ρ	Density (g/m^3)

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CHAPTER 1

INTRODUCTION

1.0 Overview

In this modernization of modernity, Malaysia is trying to rise up in parallel with developed countries by way of applying green technology in every sector of marketing and production. Green technology is known as technology that can be recycled or reversed and also to develop products, equipment and system used to conserve the natural environment and resources which minimize and reduce the negative impact of human activities. This way is to reduce the percentage of pollutions in Malaysia in considering the green house effects in every aspect that had been taken. Green technology subject areas covered energy, green building, environmentally preferred purchasing, green chemistry and green nanotechnology.

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The famous case in Malaysia which to convert the usage of metals and synthetic fibre to natural fibres. Nowadays, synthetic fibres like glass, carbon and aramid are widely being used as polymer-based composites as the properties is strong and the stiffness tendency is high. However, these fibres have serious weakness in terms of their biodegradability, initial processing costs, recyclability, energy consumption, machine abrasion, health hazards and etc. Despite these, most significantly, environmental negative impacts change the attention from synthetic fibres to natural/renewable fibres. The introduction of natural fibres usage from annually renewable resources are now popularly used as reinforcements in polymer matrix. These provide benefits to the environment with obey the terms of the degradability and utilization of natural materials. The successful use of these fibres is dependent on their best structural and mechanical properties (Kabir *et al.*, 2012).

In industry mostly is looking for “greener material (renewable sources) to use in its products to reduce the burden and as a way to a growing environmentally conscious market. Particularly, in automotive production are looking for products that are much lighter, eco-friendly and low cost. An advantage for using natural fibre compared to glass fibre is their renewable nature and inherent biodegradability, rendering the issue of recyclability meaningless. The leading of using natural fibre reinforced plastics in automotive construction are become legal in many countries (Furtado, Silva and Alves, 2012).

Kenaf is the most comparatively commercially available and economically cheap amongst other natural fibre reinforcing material. Customarily kenaf symbolized as industrial due to of its great interest for the production of industrial raw materials. Kenaf fibre or known as Hibiscus Cannabinus species where genius is Hibiscus and the family Malvaceae obtained from stems of the plants. Kenaf is wild dicotyledons plant of subtropical and tropical parts of Asia. The Persian origin explaining that kenaf the plant having short day, warm season and annually herbaceous plant, with the average diameter of fibre is 67.6 μm. The properties of kenaf are hard, strong and tough plant with a fibrous stalk, resistant to insect damage and requires relatively fewer amount of or no pesticides (Saba, Paridah and Jawaid, 2015).

This study discusses the mechanical, chemical and physical properties characteristic of kenaf fibre composites comparing it with other type of natural fibres as an example PALF (Pineapple Leaf Fibres). In this research which kenaf fibres was treated using alkali solution, NaOH (Sodium Hydroxide) to get the better result in mechanical and physical properties of the fibres.

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1.1 Problem Statement

Natural fibre composites are being the top main of research nowadays. As researchers and scientists trying to overcome the disadvantages of kenaf fibres.

Recently, the usage of natural fibres in sector of automotive, construction, sports and leisure, and other mass production industries composite component was focusing on sustainable and renewable reinforced composites. The addition of reinforcements, such as fibres and fillers into polymer composites meaning that by extending the usage of fibres and improvise the properties of component of the composites parallel with the requirements of engineering application. The effects of these improvements will be associated with economic advantages, such as low production costs and low resin consumption. As a result, for the past few years, various commercial applications in the industrial sector was demanding for natural fibres in their usage components production. (Akil *et al.*, 2011).

Natural fibres have a lot of advantages which one of the advantages is can protect green house of earth and directly prevent the pollutions that often happen. However, one of the disadvantages is the dimension of the kenaf are not stable due to the hydrophilic properties.

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Regarding the disadvantages of the fibres, based on literature review it can be solved by using chemical treatment. Examples of chemical treatment are alkaline treatment, silane treatment, acetylation treatment, benzoylation treatment, peroxide treatment, maleated coupling agents, sodium chloride treatment, acrylation and acrylonitrile grafting, isocyanate treatment, stearic acid treatment, permanganate treatment, triazine treatment and fatty acid derivate (oleoyl chloride) treatment but in this experiment was been focusing on alkaline treatment using sodium hydroxide (NaOH) as the solution is cheapest and effective among others.

1.2 Objective

The objective of this experiment is to evaluate the effect of the alkalization on mechanical test and physical properties of kenaf fibres reinforced vinyl ester and to investigate the effect of kenaf reinforce vinyl ester in terms of morphological properties.

1.3 Scopes of Study

The composites samples were prepared by using hand lay-up process. Sample then been subjected with fibres loading 5 wt%(Razali, 2016).

The treatment process that emphasize the chemical treatment by using 6% concentration of sodium hydroxide solution to analyze the mechanical and physical properties of the composites. The properties of mechanical and physical properties covered tensile strength, flexural strength, water absorption and moisture content.

The fibres were categorized with two different part which is fibres that were treated and fibres that untreated. The results then been recorded.

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CHAPTER 2

LITERATURE REVIEW

2.0 Introduction

In the last few years, the reduction of the petroleum resources have increase the awareness towards the environment and greenhouse effect have given a big impact towards the industries in example automotive, building and nautical and one of method that had been discussed to reduce the effects by replacing the usage of synthetic fibre with the sustainable ones (Krishna and Kanny, 2016). It been stated by (Kabir *et al.*, 2012) which the terrible impact towards the environment decides that the attention from synthetic fibres to renewable/natural fibres.

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Natural fibres have been discovered as material in reinforcement agent for more than 3000 years. Based on matrix phase, composites can be classified into three classes which are metal matrix composites (MMCs), ceramic matrix composites (CMCs) and polymer matrix composites (PMCs) (Saba, Paridah and Jawaid, 2015). With development in technology, natural/renewable fibres have been combined with polymer matrix (Razali, Sapuan and Razali, 2018). In (Mohamed *et al.*, 2018) research stated that there are many advantages regarding usage of fibre reinforced polymer (FRP) compared to conventional substances for a certain property. A natural fibre reinforced polymer (FRP) is a composite material that consist of thermoset and thermoplastic resins such as epoxies, polypropylenes, vinyl group and elastomer. (Kabir *et al.*, 2012) said that the introduction of the natural fibre yearly as renewable resources is now popular used as reinforcements in polymer matrix as a result, (Akil *et al.*, 2011) prove that the demands of natural fibre are fluctuated over the few years for various applications in industrial sectors.

Natural fibre reinforced polymer composites is found to be an alternative way in replacing the petroleum sources that currently are decreasing as it receive attention from manufacturer and scientists as the properties of the natural fibre-based composites are biodegradability, light in weight, nontoxic and directly can prevent the pollution to the environment (Saba, Paridah and Jawaid, 2015). These type of composites presents many advantages compared to synthetic nor to carbon fibres as examples low tool wear, low density, cheaper cost, availability, high strength, high stiffness, good corrosion resistivity and low coefficient friction properties (Razali, Sapuan and Razali, 2018) (Mohamed *et al.*, 2018). Regarding that, natural fibres also have their own limitation as (Maslinda *et al.*, 2017) stated in her research that natural fibres have limit in their application such as variability in quality, high moisture absorption, limited processing temperature and a lesser durability and incompatibility between fibres and polymer matrices.

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In a way to overcome the limitation of the natural fibre’s properties, many treatments had been endured in a way to improve the performance of the natural fibre itself. In order to improvise the strength of the natural fibre can be achieved by reinforcing it using various chemical treatment on the fibres. (Krishna and Kanny, 2016)

2.1 Natural Fibre

Due to environmental concern, the replacement of using synthetic fibre with natural fibre has been investigated (Maslinda *et al.*, 2017) and in (Krishna and Kanny, 2016) research also been stated that environment was one of the high concern issue and added by financial issue and thus nowadays natural fibres have become interesting and fascinating. Fibres had been stated are look like hair continuous filament materials that act as the spreading phase (Mohamed *et al.*, 2018) and plant was known as renewable resources, biodegradable and energy that required to produce is small (Akil *et al.*, 2011). Humans have continued to domesticate these crops over time as the natural fibre crops are the earliest fertilized plants. The worldwide now availability in using natural fibres and abundantly agrowaste in engineering research and the search for a sustainable technology in combining polymer science (Mahjoub *et al.*, 2014).

Exterior underfloor paneling of cars, sports equipment and marine structures are example in widely usage of natural fibres composites (Maslinda *et al.*, 2017). The application of natural fibres had been used widely in many sectors of industries such as furniture, construction, automobiles and packing due to its low cost, low weight and less damage as compares to synthetic fibres (Krishna and Kanny, 2016). This two sources above are also been proved in (Mahjoub *et al.*, 2014) research that industry now are focusing in natural fibres as industrial material such as sport equipment, automotive application and construction material for structural and non-structural elements.

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There are a few types of fibres that can be obtained which are natural (plant and animal) and man-made (synthetic and regenerated fibres) and can be categorized into three parts which from lignocellulosic materials, animal and mineral (Mohamed *et al.*, 2018). (Akil *et al.*, 2011) also wrote in their journal that natural fibres had been divided into three categories.

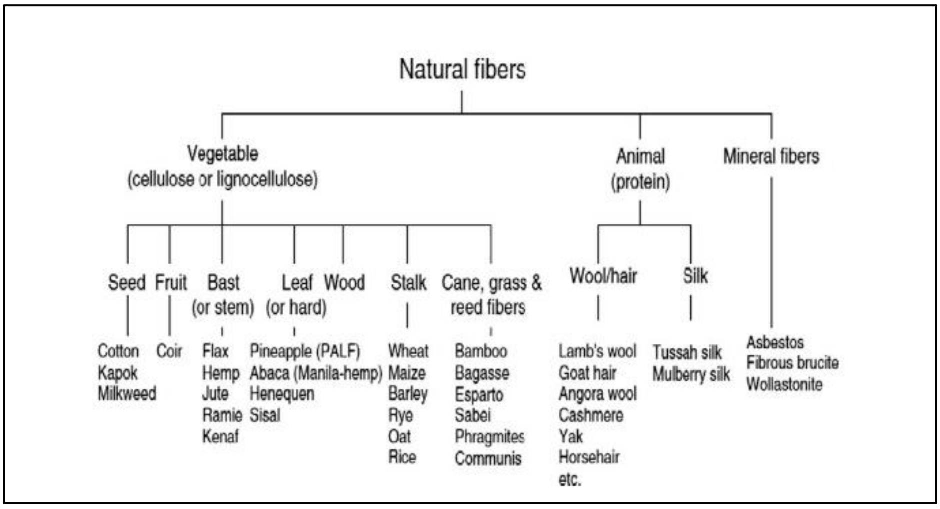

Figure 2.1 Classification of natural fibres (Akil *et al.*, 2011)

Kenaf, roselle, jute, sugar palm, oil palm empty fruit bunch, sisal, pineapple leaf, rice husk, kapok, wood, barley oats choir, and abaca are several types of natural fibres that have been used (Razali, Sapuan and Razali, 2018) while (Akil *et al.*, 2011) claimed that bast fibres such as hemp, jute, flax, kenaf and sisal are most common natural fibres that selected in industries. (Maslinda *et al.*, 2017) reported that the most widely used and studied fibre material are flax, jute, hemp, sisal, ramie and kenaf fibres.

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Natural fibres were introduced with intention of yielding lighter composites, coupled with lower costs compared to glass fibre (Mahjoub *et al.*, 2014). One advantage using natural fibres is their low density, which endows them with excellent specific mechanical properties, easier in handling and processing, recyclability and good thermal and acoustic insulation compared to glass fibre (Maslinda *et al.*, 2017). Other advantage that obtained from (Mahjoub *et al.*, 2014) is natural fibre have low density which is (1.2-1.6 g/cm^3) than glass fibre which is (2.4 g/cm^3) as to provide lighter composites. The main reason of consuming the natural fibres in industries because of natural fibres have a lot of advantages can surely save cost in order to get from the suppliers. Natural fibres are consist of 60%-80% cellulose, 5%-20% lignin and moisture up to 20% (Mohamed *et al.*, 2018) and (Oushabi *et al.*, 2017) said it contain pectin and rich in hydroxyl groups thus natural fibres also tend to be strong polar.

Large contain amounts of hydroxyl group are generally in natural (plant) fibre which makes them polar and hydrophilic in nature contrast with plastic that most of the are hydrophobic. The addition of hydrophilic natural plant into plastic hydrophobic will produce poor mechanical properties due to non-uniform fibre dispersion in matrix and an inferior fibre matrix interphase. The hydrophilic polar nature also results in high tendency of water absorption make which leads to fibre swelling and voids in the fibre matrix interphase. As a result, with high water absorption will affect the mechanical properties of the while structure and loss of dimensional stability. The hydrophilic problem is been solve with fibre surface treatment or matrix modifications (Akil *et al.*, 2011).

As a conclusion, natural fibres have reached the satisfactory ability in order as reinforced agent due to their biodegradable and renewable characteristics in thermoset and thermoplastic matrices (Mohamed *et al.*, 2018). The need of treatment to the natural fibres' surface is because to increase the strength of the fibre itself parallel with synthetic fibres as the natural fibres itself have different orientation makes the composites become much stronger and comparable with the synthetic fibres. The needs of surface treatment to improve the compatibility and interfacial bond strength as the method is the largest areas of recent research (Krishna and Kanny, 2016).

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2.2 Kenaf Plant

Kenaf is produced from the bast (Mahjoub *et al.*, 2014) and can be known as Hibiscus cannabinus L (Mohamed *et al.*, 2018) of Malvacea is a yearly plant that originally came from central Africa and can be found as a wild tropical and subtropical Asia (Nurul Fazita *et al.*, 2017). Kenaf plant, can take 150 days to grow (Mohamed *et al.*, 2018) or three months after sowing the seeds able to grow not in 3m high with the diameter range between 25-51mm. In late 19th century, the word kenaf is generated from Persian language (Mahjoub *et al.*, 2014) which indicate plant having short day and warm season (Saba, Paridah and Jawaid, 2015). Malaysian farm was supplier of kenaf fibre (Mahjoub *et al.*, 2016) and was noted as an important source of fibre composites and also to the other industrial applications (Akil *et al.*, 2011). Three layers bast, core and pitch that available in kenaf plant as one third of the plant represents as kenaf bast (Ververis *et al.*, 2012). In (Mohamed *et al.*, 2018) stated that in kenaf plant respectively contain 35%-40% of bast and 60%-65% of core.

Example of fibres that usually been taken and been widely used in engineering field (Saba, Paridah and Jawaid, 2015) and natural fibres reinforced polymer composite industry are kenaf, jute and hemp fibres (Maslinda *et al.*, 2017). Its favorable of reinforced polymer composites because of the good mechanical properties (Nurul Fazita *et al.*, 2017). For kenaf, normally kenaf bast fibre are largely used in reinforcing thermoplastics composites (Fiore, Bella and Valenza, 2015) as the properties is superior toughness and high aspect ratio in comparison to others with tensile strength 11.9GPa a and modulus 60Gpa respectively (Akil *et al.*, 2011).

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Figure 2.2 Kenaf plant (Akil *et al.*, 2011)

Highly extractable properties and good mechanical properties make the kenaf fibre are well known among natural fibre plants (Mohamed *et al.*, 2018). The processing bark of the kenaf plant in order to get the kenaf fibre exhibits low density, non-abrasive during processing, and biodegradable (Fiore, Bella and Valenza, 2015). Besides, among the natural fibres, kenaf is comparatively commercially available and economically cheap among others (Saba, Paridah and Jawaid, 2015). Kenaf fibres have interesting mechanical and physical properties (Mahjoub *et al.*, 2014).

Table 2.1 Mechanical properties of kenaf fibre

Source	Density (g/cm^3)	Tensile Strength (MPa)	Elastic Modulus (GPa)	Elongation at break (%)
(Sreenivasan *et al.*, 2013)	1.2-1.45	930	53	1.6
(Saba, Paridah and Jawaid, 2015)	1.45	930	53	1.6
(Akil *et al.*, 2011)	-	930	53	1.6
(Ververis *et al.*, 2012)	-	930	-	1.6
(Maslinda *et al.*, 2017)	-	930	53	1.6
(Nurul Fazita *et al.*, 2017)	-	295-930	25-53	1.50-6.90

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Table 2.2 Chemical content of kenaf fibre

Source	Lignin (wt%)	Hemicellulose (wt%)	Cellulose (wt%)	Ash (wt%)
(Fiore, Bella and Valenza, 2015)	20.1	-	44.4	4.6
(Akil *et al.*, 2011)	8-13	21.5	45.57	-
(Ververis *et al.*, 2012)	14.7	-	55	5.4
(Maslinda *et al.*, 2017)	9	20.3	72	-
(Mohamed *et al.*, 2018)	21.6	-	65.7	-
(Nurul Fazita *et al.*, 2017)	0.6-1.0	21.0	36.0	-

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Table 2.3 shows the chemical content in kenaf fibre with different of sources. In (Furtado, Silva and Alves, 2012) stated that generally in natural plant contain 60%-80% of cellulose, 5-20% lignin and up to 20% moisture. Aligned along the length of fibre contains the cellulose fibrils which can gives the maximum of tensile and flexural strengths. The function of hemicellulose is for biodegradation, moisture absorption and thermal degradation where lignin is thermally stable but responsible for UV degradation.

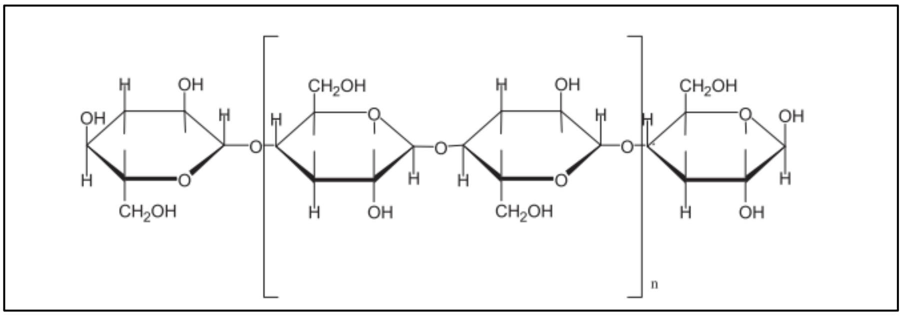

Figure 2.3 Chemical structure cellulose (Akil *et al.*, 2011)

Paper products, building materials, absorbent and animal feeds are proved that diverse novel applications are exist for kenaf fibres. This natural fibre act as substitution for fibreglass and other synthetic fibres, textile for example in automobile, dashboards, carpet padding, corrugated medium. Fibre lawn mats impregnated with grass seed and spray soil mulches for use along highway rights of way or construction sites to prevent soil erosion due to water and wind are example of kenaf fibre bast that are commercially used (Mohamed *et al.*, 2018). Natural fibre known as light weight for like kenaf fibre composites that used in automotive industries for fuel consumption and emission improvement (Mahjoub *et al.*, 2014) and had been proved in (Furtado, Silva and Alves, 2012) that stated kenaf fibre had been consumed in door inners panel. Kenaf fibre are naturally intertwined with each other (Mahjoub *et al.*, 2016). In order to ensure that the fibre is not entangle, the natural fibre is been heated after the surface treatment are done to ensure that there is no trap water as it can affect the result of the experiment.

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2.2.1 Kenaf Bast

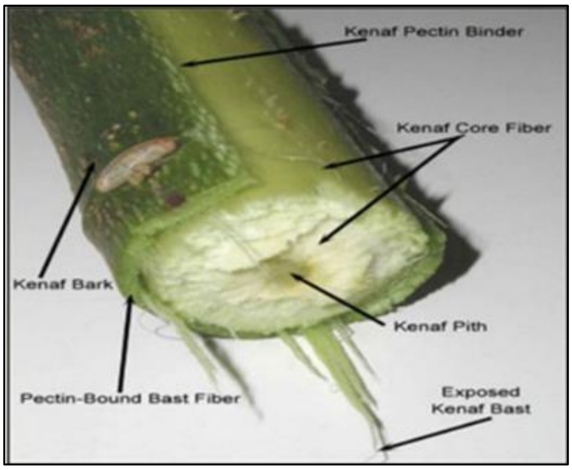

Figure 2.4 Kenaf stalk and its components fibre (Sreenivasan *et al.*, 2013)

Kenaf bast fibre reinforce polymer materials are popular among researchers nowadays as it is proven that it will give the best result in mechanical properties and physical properties especially for thermoset polymer. Kenaf bast fibre are actually extract from bark of the 35-40% kenaf stalk. Usually application that use kenaf bast fibre on paper or board making. The past result obtains also showed that kenaf fibre bast have good tensile strength, bending strength, good internal bonding, thickness swelling and water absorption properties(Sreenivasan *et al.*, 2013).

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2.3 Thermoplastic and Thermoset Properties

The natural fibre that easily available in Malaysia are kenaf, jute and hemp fibres and these type of fibres are also among the most widely used materials in the natural fibre reinforced polymer composites industry (Maslinda *et al.*, 2017). In order to replace the usage of metals in manufacturing polymers have taking over in many applications such as thermoset polymer have risen in the market for housing tools, automotive and electrical components as well as motor parts. Type of thermoplastics that commonly use are polypropylene (PP), poly-ethene (PE) and polyvinyl chloride (PVC) while thermosets are phenolic, epoxy, vinyl ester and polyester resins. Normally thermoplastic was been reinforced and thermoset reinforced with plant base fibres (Mohamed *et al.*, 2018).

There was a study that stated the tensile properties of natural fibre reinforced polymers (thermosets and thermoplastics) were affected directly by the interfacial adhesion between the fibres and matrix. Kenaf fibre reinforced thermoset and thermoplastic obtained similar results of mechanical properties but thermoset (Saba, Paridah and Jawaid, 2015)and thermoplastic have different properties itself (Kabir *et al.*, 2012).

Consider same properties between thermoset and thermoplastic composites at a room temperature. Thermoplastic usually have better fracture toughness and fatigue as many thermoplastics can tolerate higher strains prior to failure compared to thermoset (Muzzy and Kays, 1984)

Table 2.3 Comparison of thermoset and thermoplastic matrix (Kabir *et al.*, 2012)

	Advantages	Disadvantages
Thermoset	Low resin viscosity	Brittle
	Good fibre wetting	Non-recyclable via
	Excellent thermal stability once polymerised	Standard techniques
	Chemically resistant	Not post-formable
Thermoplastic	Recyclable	Poor melt flow
	Easy to repair by welding and solvent bonding	Need to be heated above the melting point for processing purpose
	Post formable	
	Tough	

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2.4 Alkaline Treatment of Kenaf Fibres

Silane, alkali, acylation, benzoylation, malleated coupling agents, permanganate, acrylonitrile and acetylation grafting, steric acid, peroxide, isocyanate, triazine, fatty acid derivate, sodium chloride and fungi are examples of various type of chemical treatment that available (Mohamed *et al.*, 2018)(Akil *et al.*, 2011) as the main purpose of the treatment is to increase fibre/matrix interfacial bonding and stress (Mohamed *et al.*, 2018). In order to enhance the matrix-fibre adhesion, chemical treatments are used by increasing the roughness through of the clean fibre surface from any impurities and by disrupting the moisture absorption process through of coat -OH groups in fibre (Krishna and Kanny, 2016) in other word, modifying of surface, cleaning the fibre surface, reduce the moisture absorption process and upsurge the surface unevenness can be done my endure the chemical treatment or known as pre-treatment (Saba, Paridah and Jawaid, 2015). Chemical modifications method or treatment of natural fibre surface including kenaf is basically carried out using reagents which contain functional groups that are capable of bonding with the hydroxyl group from the natural fibre itself (Akil *et al.*, 2011). In (Fiore, Bella and Valenza, 2015) stated that chemical method in modifying the natural fibre involve introducing a material compatible with both fibres and matrix.

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Natural fibre in way using the methods of chemical surface modifications are well documented in literature including alkaline treatment (Mahjoub *et al.*, 2014). One of the most higher research studied are ‘alkalization’ which consists on treating kenaf fibre using alkali solution in order to remove lignin, pectin, waxy substances and natural oil covering the external surface of the fibre cell wall improved the mechanical ability and physical ability of the natural fibre (Oushabi *et al.*, 2017)(Sreenivasan *et al.*, 2013) and also reduce the fibre diameter (Fiore, Bella and Valenza, 2015). Chemical treatment which use alkali solution also known as mercerisation (Sreenivasan *et al.*, 2013) increase the interfacial bonding strength between lignocellulosic fibres and thermoset resins (Fiore, Bella and Valenza, 2015). Alkaline treatment or mercerization is a well-known chemical treatment of surface modification of natural fibre reinforce polymer composites (Mahjoub *et al.*, 2014) and one of the most familiar and effective alkali solution that were applied in kenaf fibre that was been used are sodium hydroxide (NaOH) solution (Akil *et al.*, 2011)(Kabir *et al.*, 2012) and normally are the most popular usage among the kenaf reinforced thermosets and thermoplastics polymer composites. The addition of sodium hydroxide in alkaline treatment to the natural fibre promotes the ionization of the hydroxyl group to the alkoxide (Akil *et al.*, 2011).

$$\boxed{\text{Fiber}{-}\text{OH} + \text{NaOH} \rightarrow \text{Fiber}{-}\text{ONa}^{+} + \text{H}_2\text{O}}$$

Figure 2.5 Equation of natural fibre and alkali solution (Mahjoub *et al.*, 2014)

The natural fibres was immersed in alkali solution for a limited time (Mahjoub *et al.*, 2014) and as a results, the researcher discovered that alkaline treatment can improved the mechanical properties of kenaf fibres (Saba, Paridah and Jawaid, 2015). The fibre surfaces finishing becomes more uniform due to the eliminations of micro voids and thus the stress distributions capacity between the ultimate cells been improved (Kabir *et al.*, 2012). Do noted that by increase the alkali concentration can damage the natural fibres resulting the decreasing of mechanical (Mohamed *et al.*, 2018)(Kabir *et al.*, 2012) and physical properties. As a conclusion, alkaline treatment is the most effective treatment compares to others this is because alkali solution can stand for a long time of period and has a lasting effect on mechanical behaviour of the natural fibres especially on their strength and stiffness (Akil *et al.*, 2011).

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2.5 Kenaf Reinforced Polymer Composites

2.5.1 Kenaf Fibre Reinforced Thermoset Composites

A lot of researcher and journals that had been produced indicates of kenaf reinforced thermoset composites (Saba, Paridah and Jawaid, 2015). (Sreenivasan *et al.*, 2013) ‘Thermosets” or known as thermosetting materials are formed by an *in situ* chemical reaction. The majority of thermosetting are made that combining with natural fibres are polyester (UP), vinyl ester (VE) and epoxy. Polyester and epoxy are examples of thermosetting resins that will not produce any volatile by products during the curing cycle but noted that some of the thermosetting resins such as phenolics produce volatile substance as the reaction occurred. Curing cycle are state where thermosets will not reliquefy if heated or in other words will not change it shapes once it cured. Curing cycle is depend on type of thermoset resins that where been used in the experiment but thermoplastic can be softened and hardened via heating and cooling because it is the main properties of the thermoplastic which the polymer linkage is not permanent.

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Some researchers revealed that treated kenaf fibre reinforced thermoset for an example epoxy increase improved the flexural strength about 36% of the composites but fibres that untreated only shows 20% of enhancement (Saba, Paridah and Jawaid, 2015). The tensile test properties also show the increasing of the modulus elasticity and tensile strength. Some research that involve kenaf fibre reinforced thermoset (polyester resins) increase the polarity and surface adhesions (Sreenivasan *et al.*, 2013). The figure below shows the results of tensile modulus, flexural strength, tensile strength and flexural modulus of kenaf reinforced thermoset such as unsaturated polyester, epoxy and vinyl ester. The results obtain are depending with type of resins that are been used. Resins with low viscosity provide best wettability capacity and adhesion and the composites can be compression molding in more shapes compared to wood fibre-based material (Sreenivasan *et al.*, 2013).

Table 2.4 Mechanical properties of kenaf reinforced thermoset (Sreenivasan *et al.*, 2013)

Thermoset	Tensile Strength (MPa)	Tensile Modulus (GPa)	Flexural Strength (GPa)
Polyester	20-90	4-7	40-123
Vinyl Ester	20-90	4-5	60-80
Epoxy	50-65	3-5	70-100

In handling thermoset resins have a lot of method that can be divided into two categories where physical method such as hand lay-up most favorable technique, spray up, VARTM and etc. and manufacturing process such as pultrusion(injection) and compression molding. The unidirectional kenaf fibre reinforced thermoset composites can be fabricated though hand lay-up technique.

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2.5.2 Kenaf Fibre Reinforced Thermoplastic Composites

Thermoplastic have several advantages if compared to thermosets. Example advantages of thermoplastic are formed quickly into complex geometry due to low viscosity at high temperatures, can be easily reproduced and have extended life, the manufacturing cost is low, can be recycled. The thermoplastic resins also can improve the chemical resistance to increase the mechanical toughness. Thermoplastic also have high ductility and high glass transition of melting temperature. As thermoplastic have too many advantages make it if more preferable in plastic reinforce compared to thermoset composites. Thermoplastic resins are available in polypropylene (PP) and polyethene (PE) but also available in thermoplastic polyolefin composites which have many advantages such as poor adhesion between thermoplastic polymer matrix and high stiffness or strength reinforce fillers. Thermoplastic polyolefin normally used in automotive and aerospace production line.

In order to enhance the adhesion thermoplastic there were two methods that available physical and chemical modification. Physical modification like macroscopic modification approaches, fibre fibrillation, polymer rivet microscopic method and plasma treatment are examples of method that available to increase the adhesion between thermoplastic while for chemical treatment are such as grafting to form covalent bond, coating coupling agent such as silane and isocyanate, electrochemical treatment and oxidation process. Grafting method have higher interface strength (Ozsoy *et al.*, 2017).

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CHAPTER 3

METHODOLOGY

3.1 Overview of Methodology

The general methodology of this experiment has two parts which are preparation of the sample and characterization of the composites samples that will be done.

Kenaf where been refined and been treated with alkaline solution (NaOH) with different of loadings and concentrations in order to remove the impurities of the fibre surface.

Figure 3.1 shows the methodology of this experiment.

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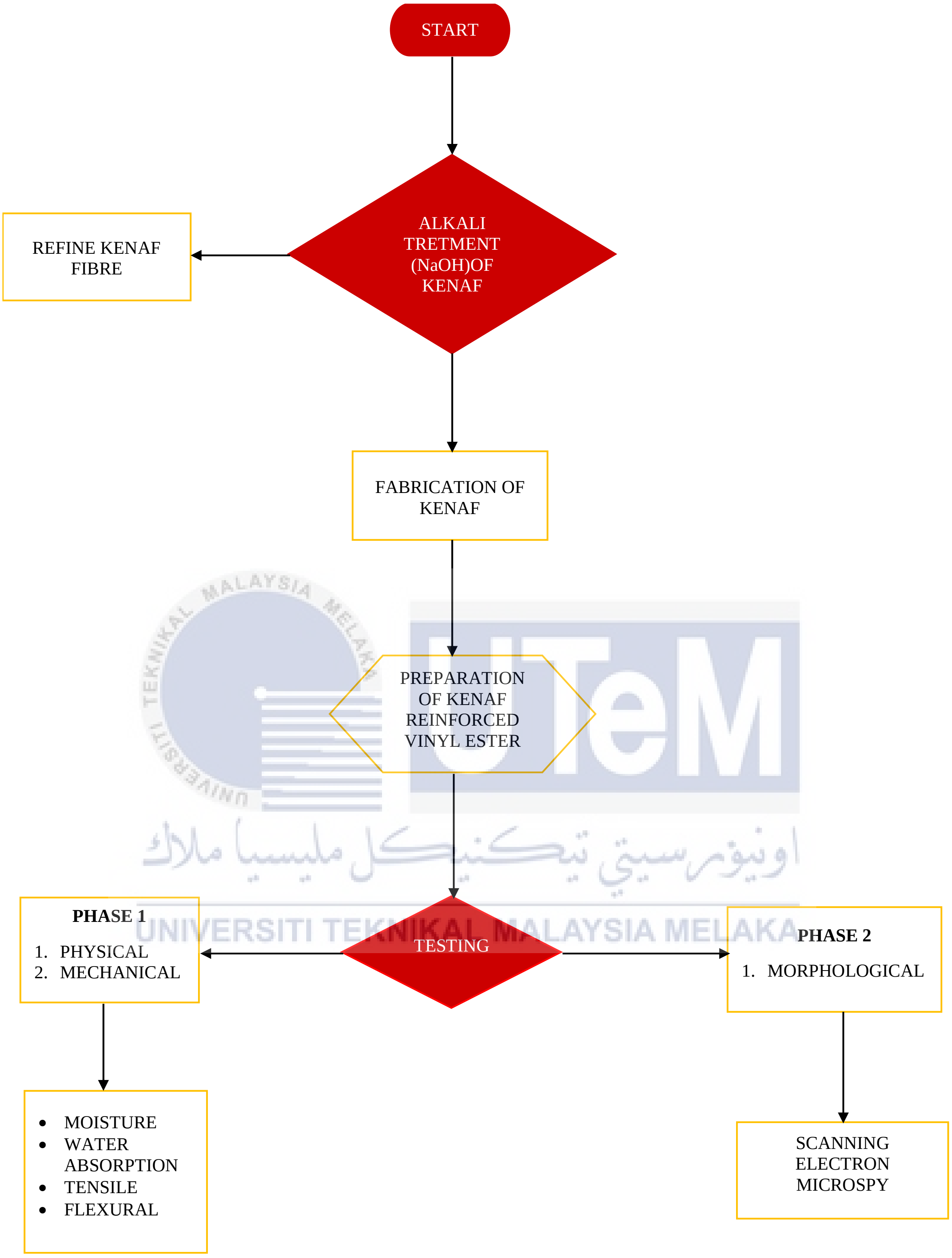

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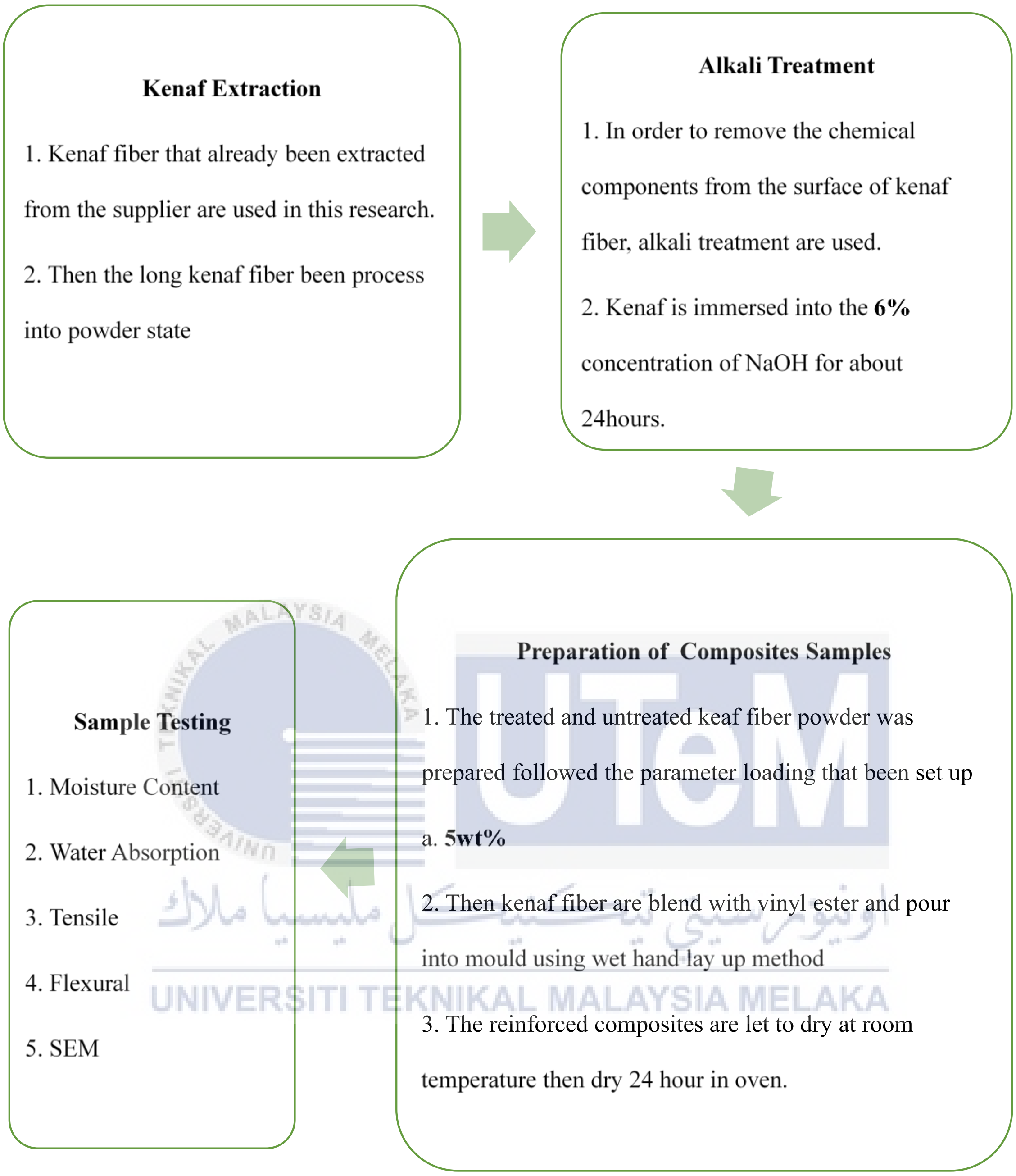
Figure 3.1 Methodology process



Kenaf Extraction

1. Kenaf fiber that already been extracted from the supplier are used in this research.
2. Then the long kenaf fiber been process into powder state

Alkali Treatment

1. In order to remove the chemical components from the surface of kenaf fiber, alkali treatment are used.
2. Kenaf is immersed into the **6%** concentration of NaOH for about 24hours.

Preparation of Composites Samples

1. The treated and untreated keaf fiber powder was prepared followed the parameter loading that been set up
   a. **5wt%**
2. Then kenaf fiber are blend with vinyl ester and pour into mould using wet hand lay up method
3. The reinforced composites are let to dry at room temperature then dry 24 hour in oven.

Sample Testing

1. Moisture Content
2. Water Absorption
3. Tensile
4. Flexural
5. SEM

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Figure 3.2 Preparation flowchart

Based on the flow chart of the research, the Gantt chart was been developed to ensure the work schedule much easier and to see the time duration that had been taken in each task that was done. Gantt chart also helpful to manage the time parallel with the task given.

Job Scope	Weeks														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Literature review	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥	🟥		
Preparation of Kenaf													🟥	🟥	🟥
Final Report Final Year Project								🟥						🟥	

Figure 3.3 Gantt chart of FYP I

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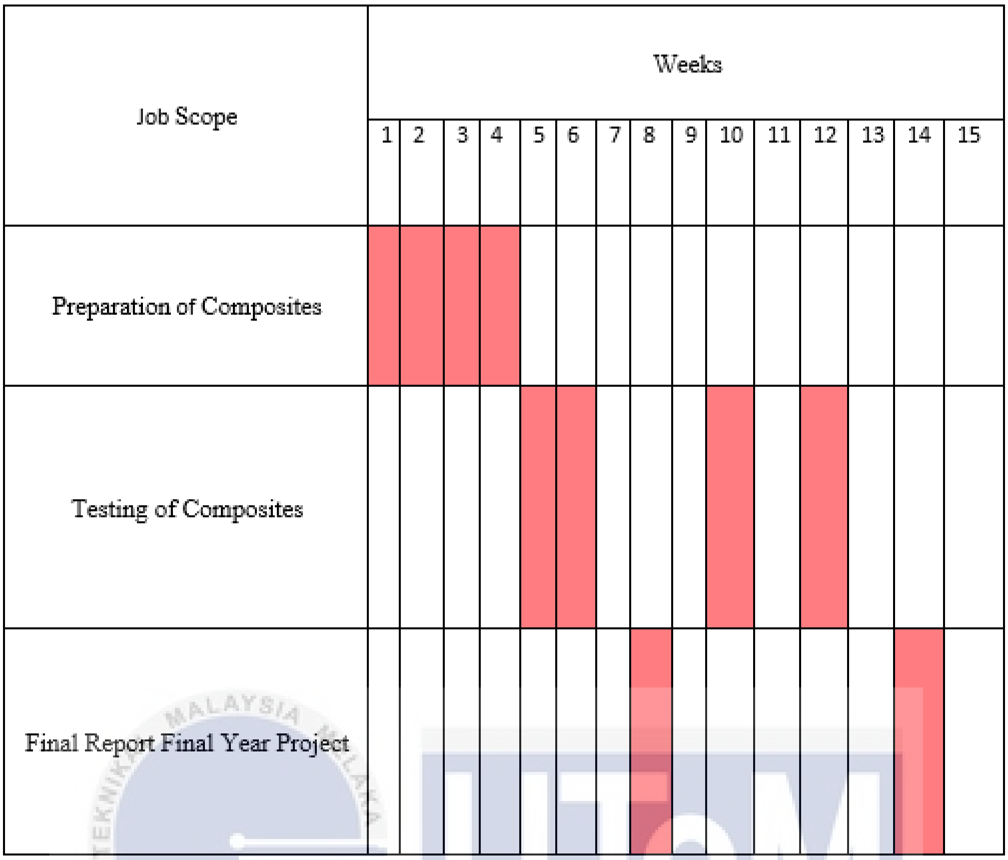

Job Scope	Weeks														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Preparation of Composites	🟥	🟥	🟥	🟥											
Testing of Composites					🟥	🟥				🟥		🟥			
Final Report Final Year Project								🟥						🟥	

Figure 3.4 Gantt chart of FYP II

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3.2 Material

3.2.1 Sodium Hydroxide (NAOH) Pellets

In this experiment, sodium hydroxide pellets were used to prepare alkali solution for alkaline treatment. Sodium hydroxide (NaOH) pellets are easily dissolve in water and the dissolution of this pellets were that used are endure the exothermic reaction. The sodium hydroxide (NaOH) solution pellets are colorless and odorless

3.2.2 Kenaf Fibre

The raw kenaf bast fibre was been inserted into crusher machine for processing. The kenaf fibre will become very fine (powder state) but sticking with each other.

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Figure 3.5 Crusher machine (TW-SC-400F)

(3-1)

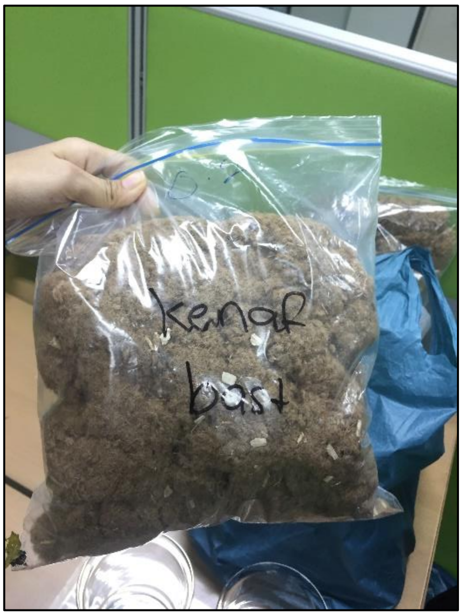

Figure 3.6 Kenaf bast (Powder state)

Figure 3.6 showing kenaf fibre after been processed into powder 0.2μm in powder state.

3.2.3 Vinyl Ester

The thermosetting resin that used in this experiment was vinyl ester (VE). In this matrix preparation, vinyl ester was poured into the dried treated and untreated kenaf fibre and handle using wet hand lay-up technique.

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3.3 Experimental Procedure

3.3.1 Preparation of Fibre and Matrix

The fibre and matrix was prepared using the law of mixture formula. The weight of elements is been calculated using equation **3-1**.

$$\text{Weight Percentage of Element, } w_e \times \text{Weight of composites} = \text{Weight of elements} \quad \textbf{(3-1)}$$

Table 3.1 shows the parameter of the fibre and matrix that had been measured and used in this experiment.

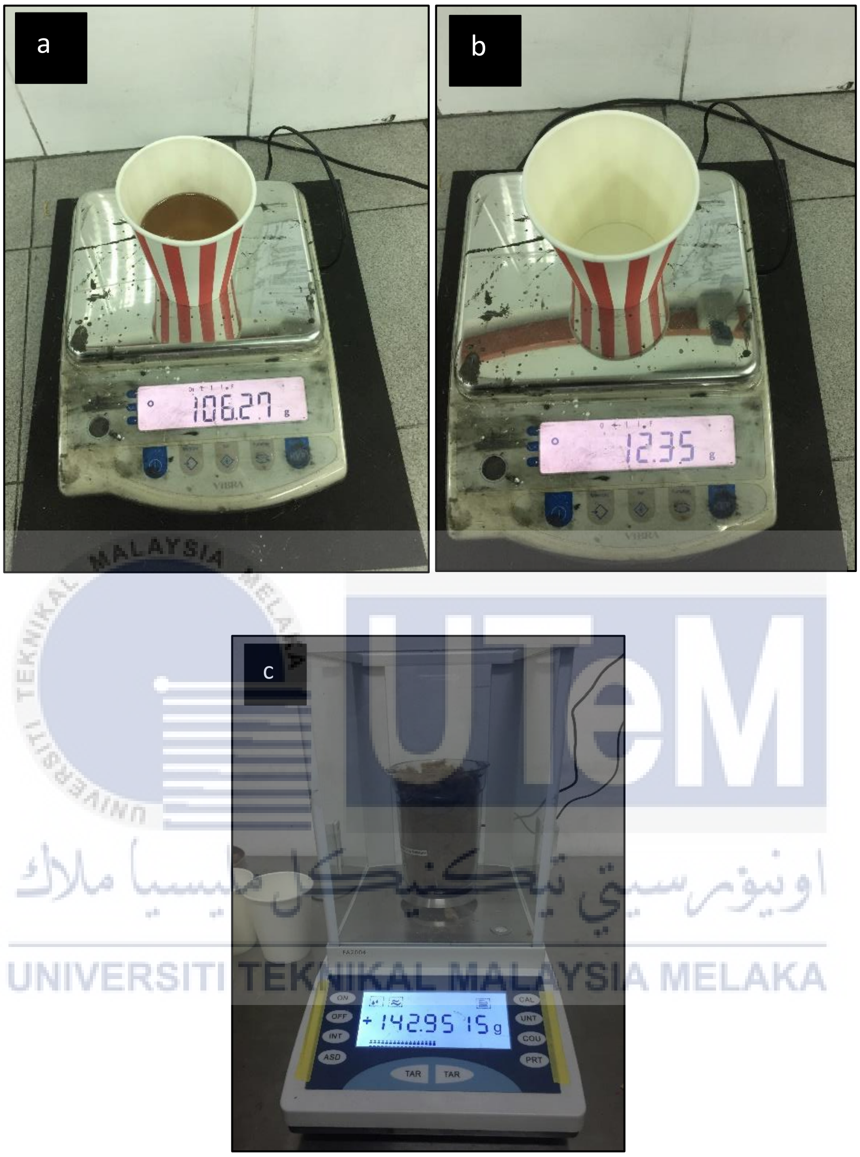

Figure 3.7 (a) Weight of vinyl ester **(b)** Weight of MekP **(c)** Weight of kenaf fibre

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Table 3.1 Parameter of composites

Elements	Weight of Composites (g)	Weight of Fibre (g) 5 wt%	Weight of Matrix (g) 93 wt%	Weight of MekP (g) 2 wt%
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 Surface Treatment of Kenaf Fibre

The kenaf was treated using alkaline treatment which been selected from chemical treatment. The alkaline treatment that using NaOH solutions are going to help in order to remove the impurities of the kenaf fibre surface and improve the properties. The kenaf is immersed into the 6% NaOH solution with for 24 hours. After 24 hours, the kenaf was clean using distilled water and the fibres are let dry in room temperature for 24 hours.

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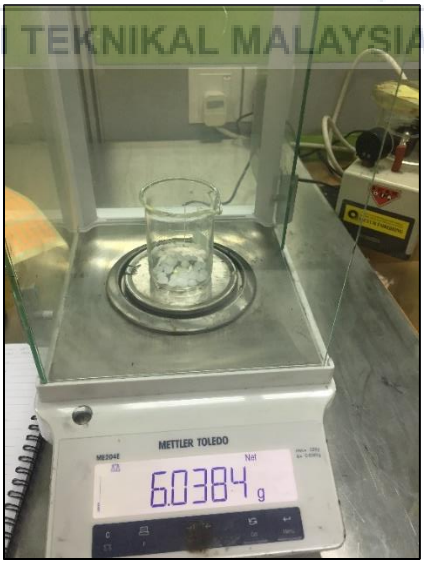

Figure 3.8 6g of NaOH pellets

Figure 3.9 Immersion of kenaf

Figure 3.10 Oven

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3.3.3 Fabrication of The Composites

Figure 3.11 Drying process of treated kenaf fibre

The treated kenaf fibre need to clean after been dried under room temperature for about 24 hours. This is because the kenaf powder might been sticking with each other after the alkaline treatment.

Figure 3.12 Drying process of treated kenaf fibre in oven

The treated and untreated kenaf fibre was put into the oven with temperature 80˚c for 24 hours to remove all the excess water and reduce attraction between fibre.

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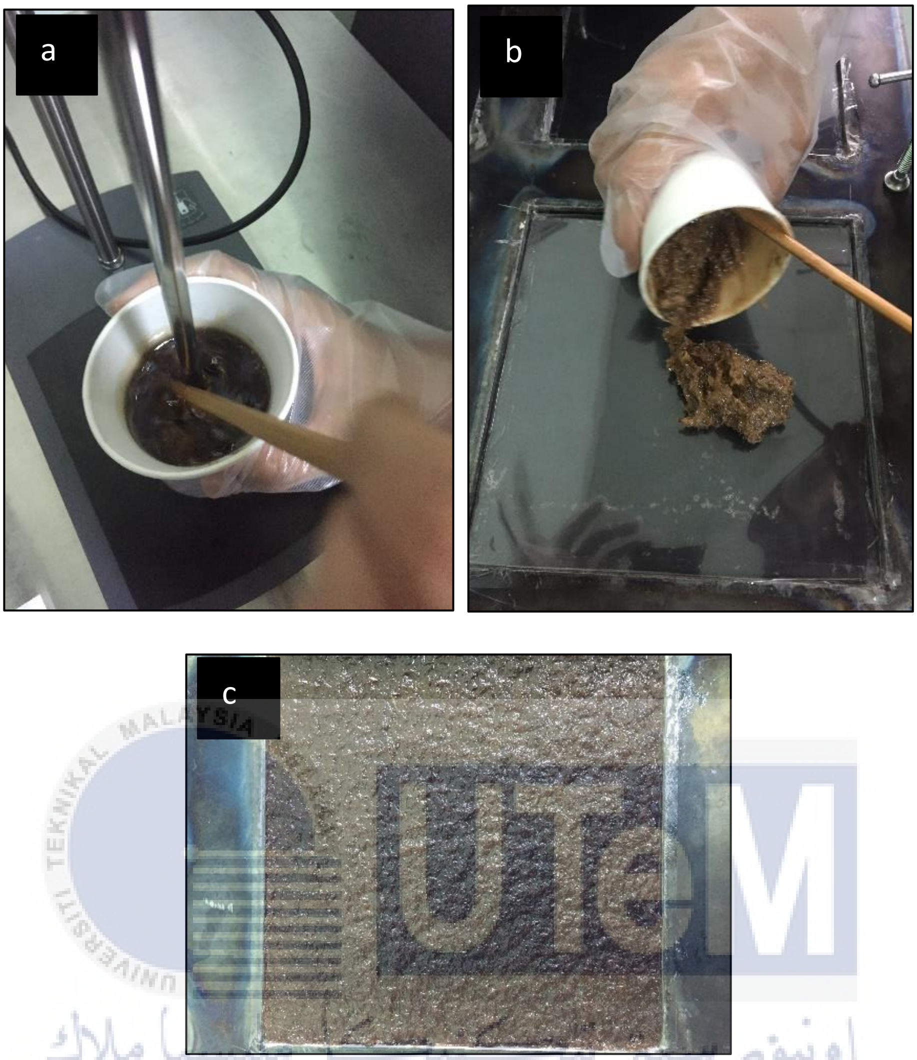

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Figure 3.13 (a) Blending process kenaf fibre with resins **(b)** Pouring blending composites into mold **(c)** Composites in mold

After the cleaning process, the treated and untreated kenaf fibre has been mixed and stir using mechanical stirrer in the beaker and been poured into the rectangular mold case. By using mylar sheet to cover the bottom and the top of the reinforce composites in pouring vinyl ester resins in order to obtain smooth surface while doing the hand lay-up process. The bubble that produce during the process has been conducted using a stick of chopstick.

After that, the kenaf fibre reinforced composites that had been cured were heated in oven up until 110˚c for 24 hours. The curing process is obeying the curing cycle of vinyl ester resins.

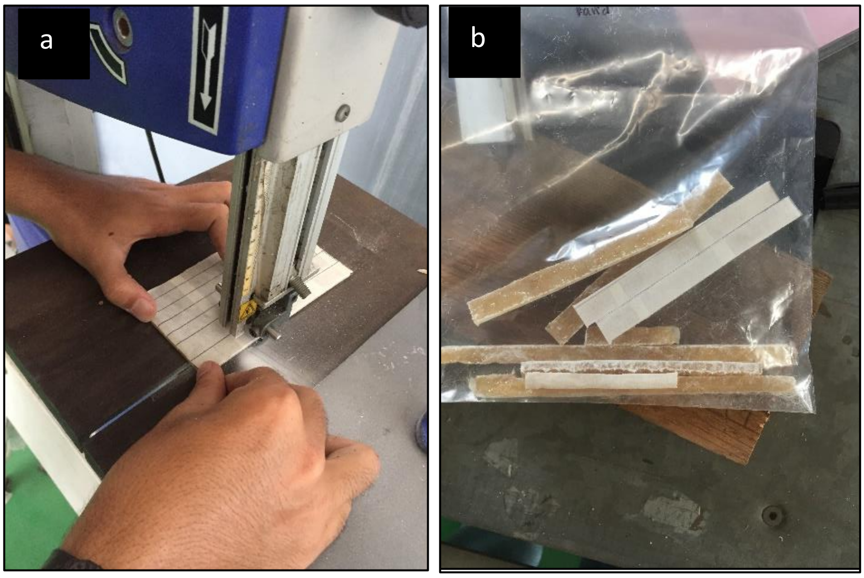

Figure 3.14 (a) Cutting process **(b)** Samples

The sample was then been cut following the correct dimension of ASTM mechanical testing and for physical testing.

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3.4 Material Characterization

3.4.1 Mechanical Testing

3.4.1.1 Tensile Test

In order to evaluate the mechanical properties of the biodegradable composites, tensile test is applied. Some of the properties that can be obtained after endure the tensile test such as Young's modulus, tensile stress, maximum elongation, tensile strain and yield stress. For this research, the testing was carried out by following to ASTM D3039. The tensile properties of kenaf reinforced vinyl ester were determined using the Universal Testing Machine model Instron 887.

Figure 3.15 Universal Testing Machine model Instron 887

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The tensile strength of the single fibre can be calculated using equation **3-2**.

$$\sigma = \frac{F}{A} \tag{3-2}$$

Where: -

σ = tensile strength of the fibre (Pa)

F = maximum force at break (N)

A = area of the cross section (m^2)

3.4.1.2 Flexural Test

Using a three-point bending set up by following the ASTM D790, the flexural test was conducted. About five sample for treated and untreated kenaf fibre reinforced vinyl ester was taken and tested using Universal Testing Machine model Instron 5585.

Figure 3.16 Universal Testing Machine model Instron 5585

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

$$\sigma = \frac{3PL}{2bd^2}$$

(3-3)

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Where: -

σ = flexural strength of the fibre (Pa)

P = maximum force at break (N)

L = support span (mm)

b = width of beam tested (mm)

d = depth of beam tested (mm)

3.4.2 Characterization of Physical Properties

3.4.2.1 Density

The density of the kenaf fibre reinforced vinyl ester was determined by using electronic densimeter.

Figure 3.17 Electronic densimeter

Five sample of treated and untreated fibre loading were taken to take the value of density. The weight of all samples was being weighed (m), the volume (V) of the samples were recorded and the density is been calculated using equation **3-4**.

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

(3-4)

Where: -

ρ = density of the composites

m = mass of the composites

V = volume of water

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3.4.2.2 Moisture Content

5 sample of treated and untreated kenaf fibre reinforced vinyl ester was prepared for moisture value. All sample were heated up at 110˚c in the oven.

The weight of the sample before put into hot oven and after being heated was taken to determine the moisture content. The weight of sample was taken before put into the oven M_i, and the weight of the sample after being heated, M_f were measured as these two values will be used to calculate the moisture content of the product. The equation of calculation is as **3-5**.

$$Moisture\ Content\ \% = \frac{Mi-Mf}{Mi} \times 100 \tag{3-5}$$

3.4.2.3 Water Absorption

Before immerse the sample in water, the initial weight,W_i of the sample is recorded. The sample then been immersed in water at room temperature (23±1˚c) for half an hour. Then after the immersion, the final weight, W_f of the samples is recorded again. The water absorption of the sample is calculated using equation **3-6**.

$$Water\ content\ \% = \frac{Wi-Wf}{Wi} \times 100 \tag{3-6}$$

3.5 Scanning Electron Microscope (SEM)

For this research, morphological studies were done in detail on the fractured surface of the tensile test sample by using Scanning Electron Microscope (SEM). The sample of treated and untreated kenaf reinforced vinyl ester were taken that 5 wt% of fibre loading. The samples were coated with platinum to get a better result of resolution as it offers a good electrical conductivity.

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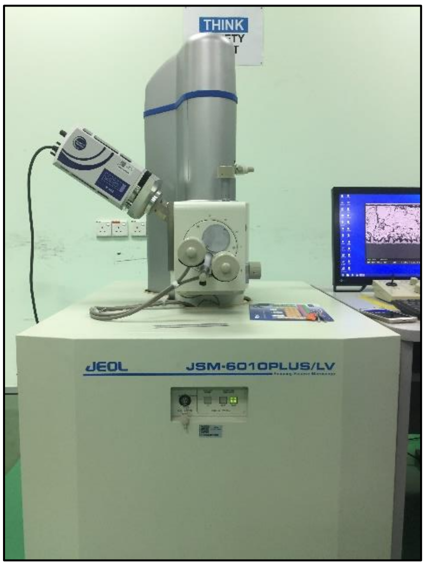

Figure 3.18 Scanning Electron Microscope JSM-6010PLUS/LV

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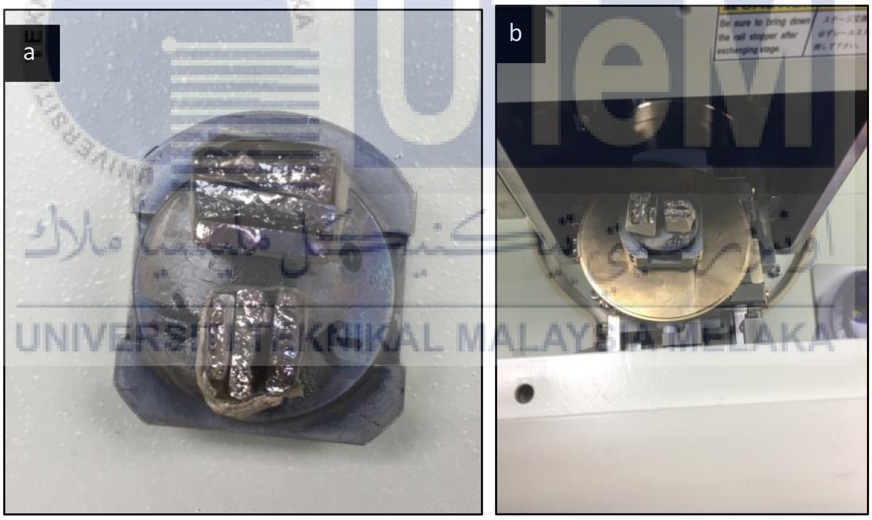

Figure 3.19 (a) Sample coating with platinum **(b)** sample in SEM machine

CHAPTER 4

RESULTS AND DISCUSSIONS

4.0 Introduction

The alkaline treatment have been done as the surface treatment is way to enhance the interfacial bonding effectiveness between fibre and matrix(Kabir *et al.*, 2012). This treatment was to ensure any impurities that presence on the fibre surface can be remove and enhance roughness of the surface to achieve good interlocking bonding between fibre and matrix, can withstand high load pressure and remove some of chemical content such as part of lignin, hemicelluloses and other impurities(Oushabi *et al.*, 2017) of natural fibre. As result, good mechanical and physical properties can be obtained.

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4.1 Effect of Chemical Treatment on The Mechanical Properties

In this section, mechanical testing is being done to analyze the difference of strength between neat polymer, treated and untreated kenaf fibre that blend with vinyl ester. Specific load is applied using two type of testing; tensile test and flexural test.

4.1.1 Tensile Test

From (Ververis *et al.*, 2012) stated that tensile test can be represented as one type of stress works in one direction (1-D). From this testing, it can indicate whether the sample have good in interfacial bonding or vice versa. Tensile test was subjected to determine their strength, elastic modulus and strain to failure (Maslinda *et al.*, 2017).

Figure 4.1 Neat polymer tensile test

Tensile test was carried out according to ASTM D3039 (Tensile Properties of Polymer Matrix Composite Material)(ASTM D3039/D3039M-00, 2002) standard using Universal Testing Machine model Instron 8872, equipped with load cell of 5kN, with cross-head 1mm/min. For each composite, five samples from each parameter with dimensions 150 ×15× 3 mm were tested.

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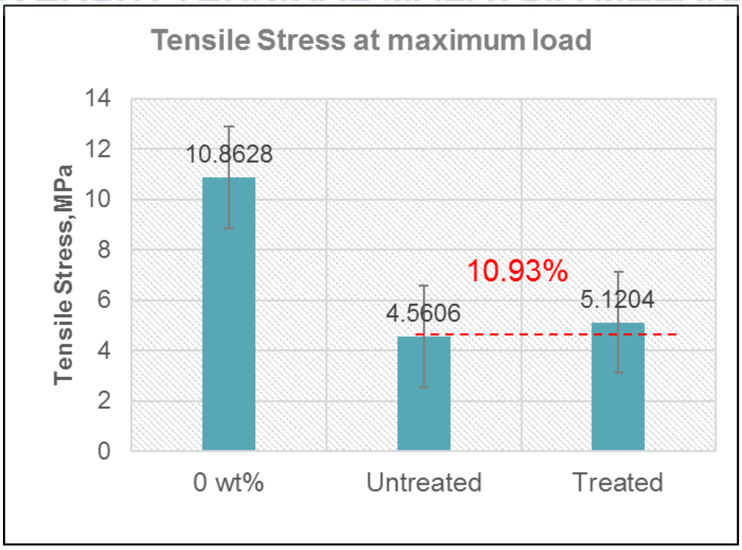
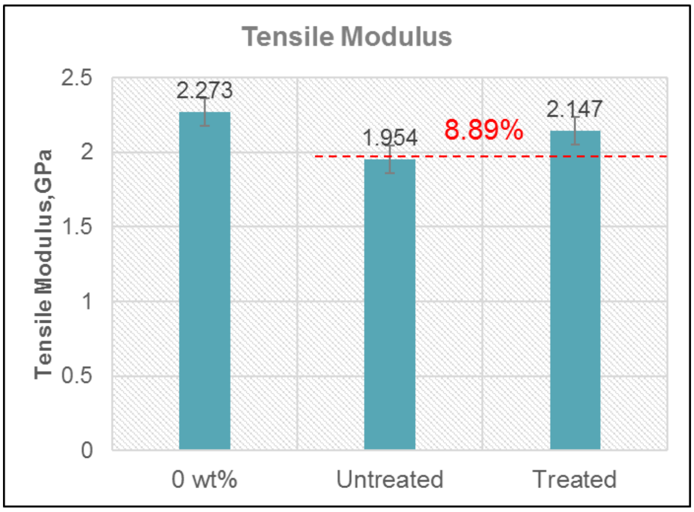


(a)



(b)

Figure 4.2 (a) Tensile strength of kenaf fibre reinforced vinyl ester **(b)** Tensile modulus of kenaf fibre reinforced vinyl ester

The graph bar showed that treated kenaf reinforced polymer indicates the highest in strength compared to untreated. As the we can conclude that mechanical properties of treated that use surface treatment (Inder *et al.*, 2017) are enhanced compared to untreated composites. In this case, fibre orientation is not involved because it is 3D case or known as isotropic which means it does not have specific fibre orientation.

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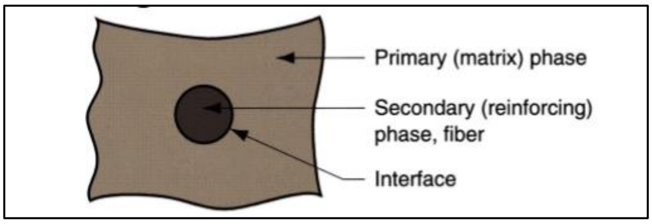

Figure 4.3 Interfacial bonding of interface

Inclination in tensile stress and modulus means the treated composites have good interfacial bonding between fibre and polymer and good stress dispersion of load towards the composites compared to neat polymer. The good interfacial bonding and lower void content of the samples can make the stress that applied during the tensile test transferred effectively (Razali, Sapuan and Razali, 2018). The results of treated composites really prove that the mechanical interlocking fibre/matrix are increase due to alkaline treatment compared to the untreated one that have lower data. The indication of higher tensile strength is because the removal of any impurities or voids in natural fibre(Oushabi *et al.*, 2017). Clean surface of treated fibre produce a better adhesion between two phases (fibre and matrix) with an enhancement of mechanical properties(Fiore, Bella and Valenza, 2015).

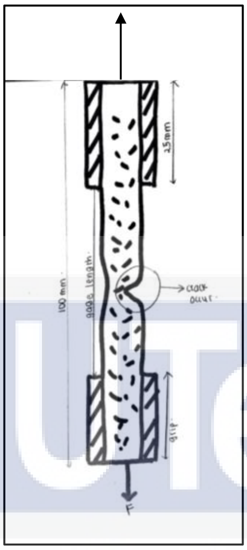

Figure 4.4 Fibre as stopping crack

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The other function of natural fibre as reinforce agent are act as stopping crack propagation(Razali, Sapuan and Razali, 2018) while stress is applied during testing. Treated composites increase around 10.93% compared to treated that currently have 1.2504 MPa tensile strength for treated and increase 8.89% of tensile modulus that have 2.147 GPa for treated. From the experiment observation, crack is mostly propagated at the end-tab not at the gage length.

Table 4.1 Theoretical result tensile strength and tensile modulus of kenaf fibre reinforced vinyl ester treated with NaOH solution (Sreenivasan *et al.*, 2013)

Composites	Tensile Strength,MPa	Tensile Modulus,Gpa
Kenaf reinforced Vinyl Ester (Theoretical)	40-50	4-5
	88.62%	52.29%
Kenaf reinforced Vinyl Ester (Experimental)	5.1204	2.147

The treated from experimental indicate lower data compared to theoretical data. The difference between theoretical and experimental data of tensile strength is 88.62% while for tensile modulus is 52.29%. This high different values occurred may due to several factors such as speed of testing that had been set, void and impurities of treatment not remove perfectly while cleaning, kenaf fibre condition, during blending process and volume percentage of reinforcement is slightly different(Razali, Sapuan and Razali, 2018).

As conclusion,(Razali, Sapuan and Razali, 2018) stated that good tensile test result depends on more effective and stress that distributed uniformly between the fibres and polymer and lower tensile stress indicate weak of ability of fibre in order to transfer the load from one point to another(Ververis *et al.*, 2012). The crack propagation deform only at one point as the stress are not contributed to other part.

4.1.2 Flexural Test

Flexural testing was done in order to determine the strength and the ability of the material to resist the deformation under loading before reach the break point(Razali, Sapuan and Razali, 2018). This technique to evaluates and obtain modulus elasticity in bending, flexural stress and flexural strain as the material is been set up as supported beam under two supports and a load is applied at a point(Sood and Dwivedi, 2018).

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Figure 4.5 Neat polymer flexural test

Flexural testing is to test whether the composites can withstand bending load and deformation before it breaks the tight structure (Maslinda *et al.*, 2017). The alkaline treatment really effects the results data of flexural strength and flexural modulus (Fiore, Bella and Valenza, 2015).

Three-point bending tests was carries out according to ASTM D790 (Flexural Properties of Unreinforced and Reinforced Plastics and Electrical Insulating Materials)(ASTM D790-03, 2017) using Universal Testing Machine model Instron 5585 equipped with a 5kN load cell and with cross-head of 1mm/min. For each composite, 5 sample was taken with dimensions 100 × 10 × 3mm. The span length was equal to 50mm.

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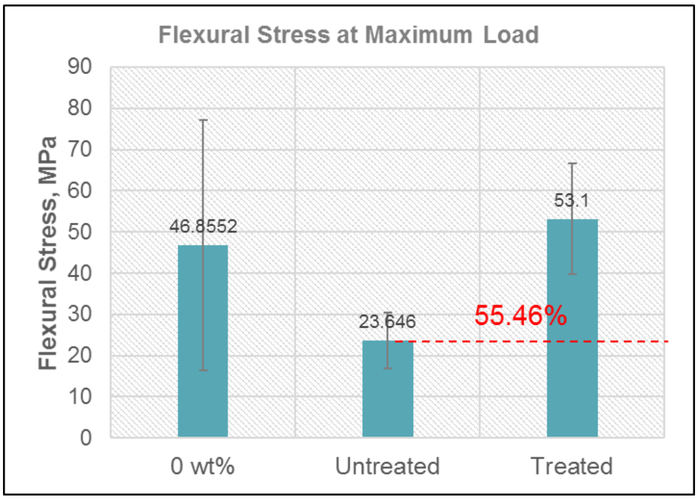
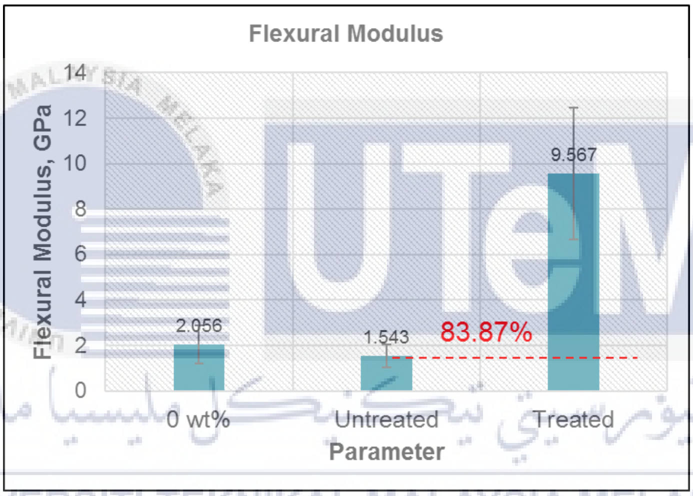


(a)



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(b)

Figure 4.6 (a) Flexural strength of kenaf fibre reinforced vinyl ester **(b)** Flexural modulus of kenaf fibre reinforced vinyl ester

It was revealed that treated fibre have highest flexural strength and flexural modulus compared to untreated. The alkaline treatment revealed can improved the flexural strength of the composites. The flexural modulus of treated increase 83.87% compared to untreated which are 9.567 GPa and flexural strength of treated increase at 55.46% which are 53.1MPa. The untreated composites have a low data may be because the interaction linkage between fibre and matrix, poor dispersion of fibre towards matrix that leads to the weak load transfer and there were voids during fabrication process(Razali, Sapuan and Razali, 2018). The higher the load bending capacity of composites can withstand, the better the mechanical interlocking between the kenaf and vinyl ester(Maslinda *et al.*, 2017). 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.

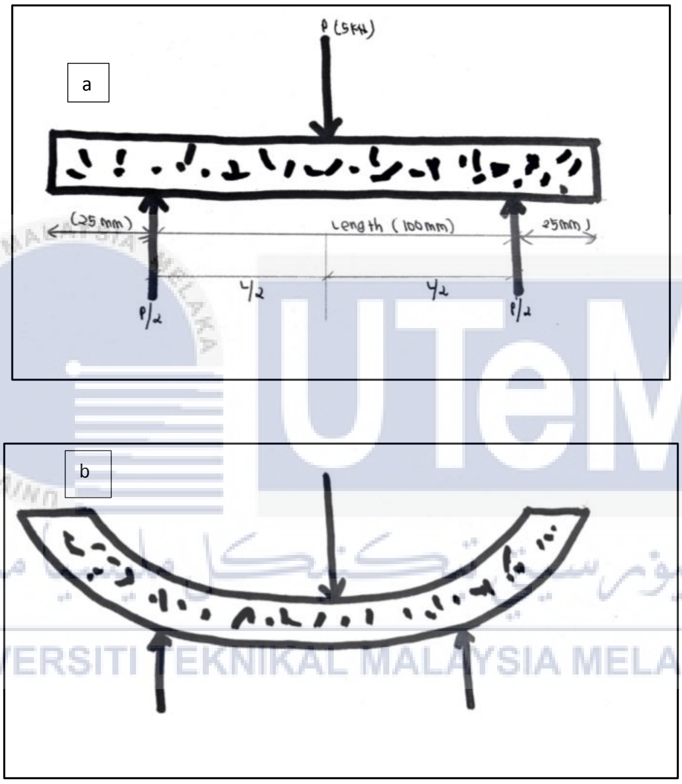

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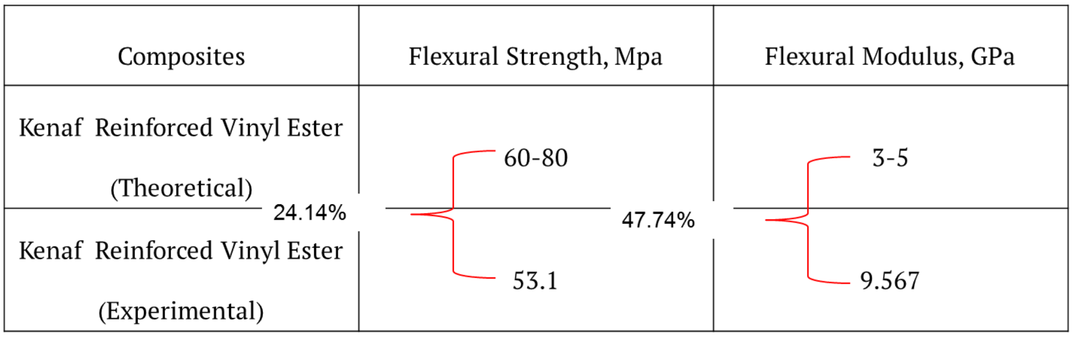
Figure 4.7 (a) Mid-span measurement **(b)** Three-point bending

The drawing defines that the fibre in the matrix was actually increase the modulus of the composites because the presence of rigid particles (fibre) in the matrix. The addition of kenaf fibre improve the stiffness of vinyl ester polymer matrix(Razali, Sapuan and Razali, 2018). The modulus become larger for treated composites because of the good mechanical interlocking between fibre and matrix.

Table 4.2 Theoretical flexural strength and flexural modulus of kenaf fibre reinforced vinyl ester treated with NaOH solution (Sreenivasan *et al.*, 2013)



Composites	Flexural Strength, Mpa	Flexural Modulus, GPa
Kenaf Reinforced Vinyl Ester (Theoretical)	60-80	3-5
Kenaf Reinforced Vinyl Ester (Experimental)	53.1	9.567

The comparison of experimental results and theoretical results conclude the same value in addition the flexural modulus in experimental get higher that theoretical really showed that processing of composites is in good terms. The difference between theoretical and experimental data of flexural strength is 24.14% while for flexural modulus is increase around 47.74% The correct method of testing also reflects the good experimental data such as correct cross-head usage, load applied, void content remove perfectly and sample are in good condition.

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4.2 Effect of Chemical Treatment to The Physical Properties

In order to check the enhancement of physical properties between treated and untreated composites, water absorption and moisture content testing had been endured. The treated composites which surface of kenaf fibre has been modified using alkali solution. As natural fibre have hydrophilic characteristic that react with hydroxyl group, so the fibre is blended with polymer matrix which is hydrophobic (resist water).

4.2.1 Water Absorption and Moisture Content

During alkaline treatment process, many voids have been remove including the chemical content of natural fibre such as hemicellulose, lignin, cellulose and any other impurities. This process lead to enhancement of bonding between fibre and matrix, fibre wetting characteristic and mechanical interlocking (Sood and Dwivedi, 2018).

The most critical issue that natural fibre needs to overcome is hydrophilic nature. The hydrophilic characteristic that available in natural fibre can cause absorption of water in fibre, the growth of fungi on natural fibre surface, the swelling of natural fibre and rot due to fungi attacks. 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. The swelling that occur due to the absorption of water will lead to poor mechanical properties of the composites. Many surface modifications are available such as alkaline treatment. The alkaline treatment indicated better wetting between fibre and matrix compared to untreated(Sreenivasan *et al.*, 2013) as we can see with the water absorption and moisture content experimental data below.

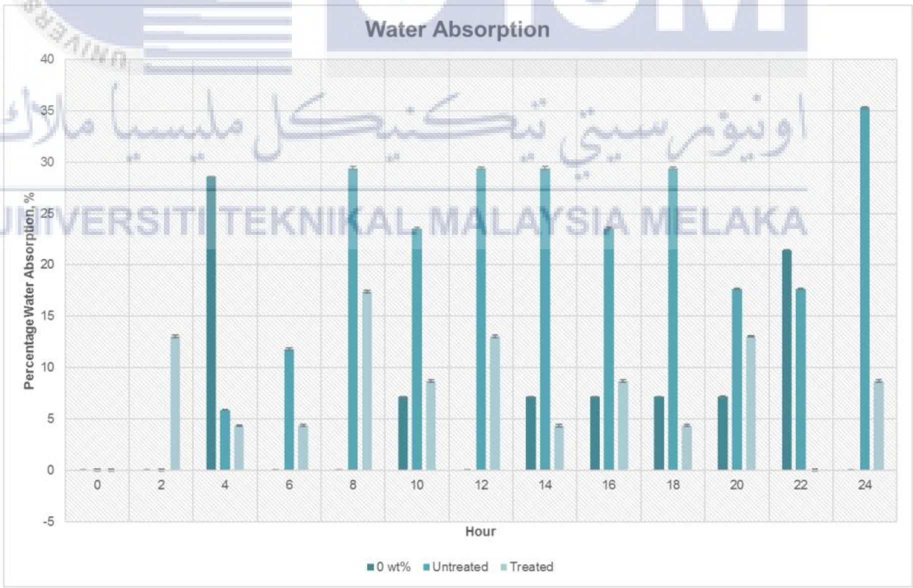

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Figure 4.8 Water absorption of composites and neat polymer at 24 hours

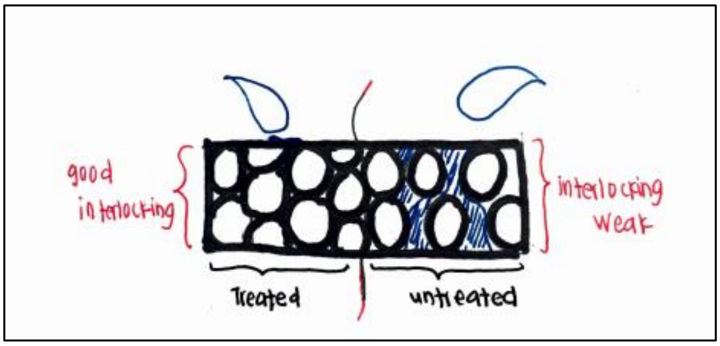

Figure 4.9 Difference penetration of water between treated and untreated

The water absorption and moisture content of the natural fibre reinforced polymer composites is being investigated. From **figure 4.8** most of neat polymer does not have any value. This is because polymer plastic characteristic which they resist water better compared to natural fibre. For untreated kenaf fibre have bad wetting characteristic compared to treated and neat polymer. The untreated fibre cannot resist water because of the contain of cellulose in natural fibre have higher chemical content which it can absorb more water than treated. The high content of cellulose allows more water to penetrate into the fibre-matrix interphase lead to crack in microstructure and swelling. If there were small cracks such as porosity, as more water that penetrated will enter the small cracking and make the small crack become larger. Neat polymer can resist water because polymer is hydrophobic characteristic which they cannot react with hydroxyl groups contrast with natural fibre (Maslinda *et al.*, 2017).

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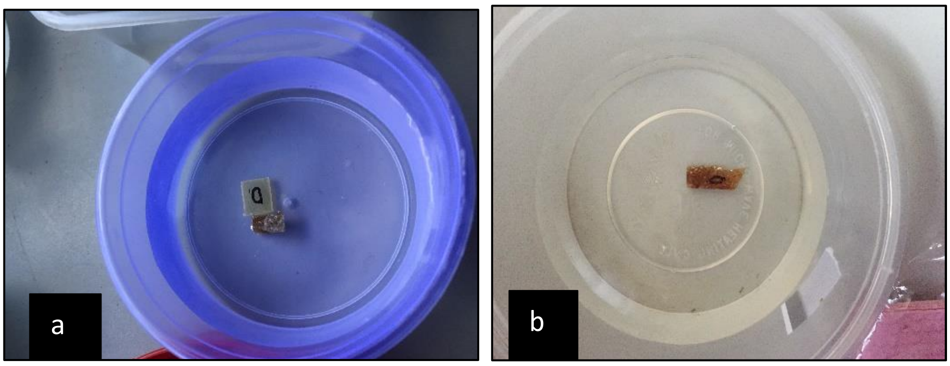

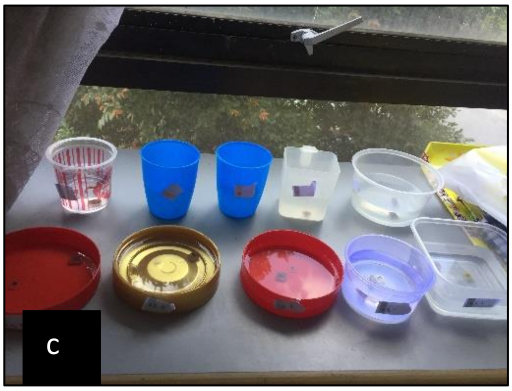

Figure 4.10 (a) Water absorption of neat polymer and treated composites **(b)** Water absorption of untreated composites **(c)** Water absorption for composites

During the water absorption test, neat polymer, untreated composites and treated composites are immersed in water for 24 hours and the reading was taken every 2 hours(ASTM D570-98, 1985). The final mass indicates the penetration of water that enter the composites and swell. The swelling that occur will change the dimension of the composites and micro cracks will starts visible. The longer the time of immersion, the larger amount of water will penetrate the interphase of the composites through the micro cracks and the detachment between fibre and matrix will occur. As can conclude in water absorption of composites issue, it is very important especially for outdoor application(Maslinda *et al.*, 2017). (Akil *et al.*, 2011) stated that water absorption may occur due to fibre dispersion in matrix, permeability of the fibres, void content temperature and etc.

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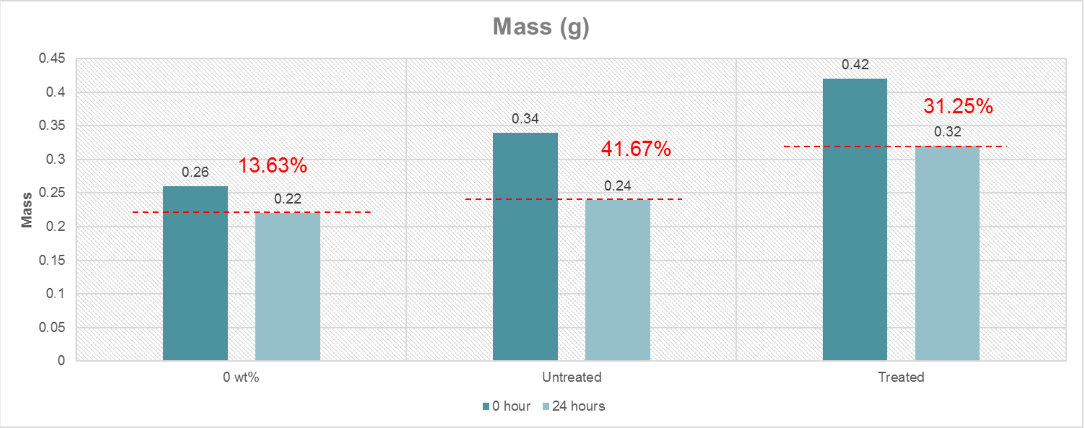
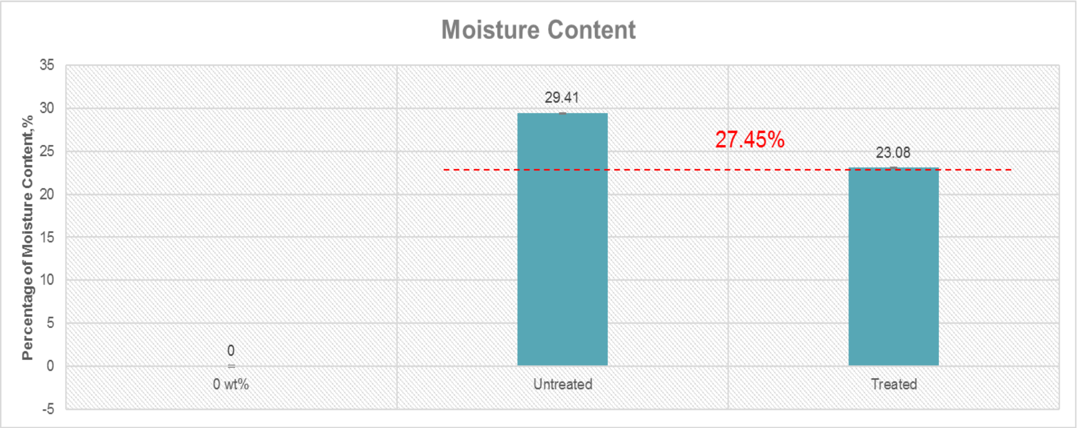


(a)



(b)

Figure 4.11 (a) Mass initial and after moisture content test

(b) Percentage of moisture content of composites and neat polymer after 24 hours

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Figure 4.12 Heating process for moisture content

Moisture content is the ability of any liquid phase such as salt water, water or jet water that may diffuse into the surface at a small quantity and up to certain temperature can dispersed into gas (ASTM D5229/D5229M-92, 2004).

As for moisture content, the composites and neat polymer were heated in oven at 110˚c. When the composites been expose to humidity environment and contact with fibre, the hydrogen bond breaks and hydroxyl group will form new hydrogen bonds with water molecule. As neat polymer is no value obtained because of vinyl ester resins have hydrophobic characteristic which plastic will never absorb water line natural fibre does. The percentage of mass that treated absorb is only 31.25% compare with the value of moisture that absorb at the untreated sample is 41.67%. This prove that untreated absorb more moisture molecules compare with the treated one. Thus, the percentage of moisture content that exist in treated sample decrease around 27.45% from the sample that are not treated with alkali solution. This data proven that air or liquid molecule cannot penetrate the treated sample as the mechanical interlocking between fibre and matrix are in a good term.

As a conclusion, the less liquid molecules that allow to penetrate into the samples, the more stable the sample can be produce. This prevention can prevent the sample swelling and fail for a short period of time. The alkaline treatment can enhance the bonding well and increase the time for liquid molecules to penetrate into the sample and make the sample become more stronger to resist it.

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4.2.2 Density

Density is conduct to check the weight of the composites in way to produce high performance product with light weight.

Table 4.3 Parameter of composites (2)

Elements	Weight of Composites (g)	Weight of Fibre (g) 5 wt%	Weight of Matrix (g) 93 wt%	Weight of MekP (g) 2 wt%
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

Table above show the different weight of composites with the same optimum weight percentage which is 5 wt%. From the table we can see that neat polymer have 100g of composites, untreated is 120g of mixture composites between matrix and fibre and lastly treated have 150g of mixture composites between matrix and fibre.

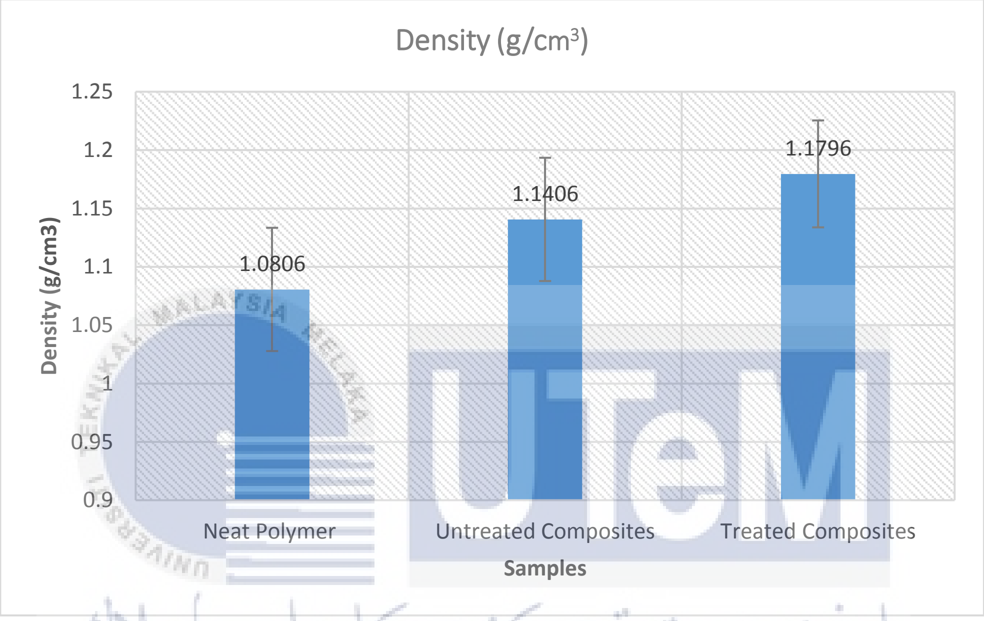

Figure 4.13 Density of sample composites

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From the data graph above reflect that increase the weight of fibre will increase the weight of composites parallel with the amount of density that are calculated. The optimum of fibre weight percent has to be correct in order to ensure the all the fibre can react with the mixture of matrix that had been blend.

4.3 Morphological of Failure Samples

4.3.1 Scanning Electron Microscope (SEM)

Using Scanning Electron Microscope (SEM) method, the review of detail of kenaf fibre reinforced vinyl ester surface had been done. This test has been done to check the adhesion bonding between treated and untreated of composites that endure tensile test.

Figure 4.14 (a) indicated the smooth neat polymer surface defects. From the observations, it has been proven that vinyl ester resins have ductile manner compared with untreated and treated have brittle manner.

Both **figure 4.14 (c) and figure 4.15 (c)** of untreated composites really shows that fibre pull out are lesser than treated one. This happen due to the bonding of fibre and matrix are not strong enough. As a result, when load was applied during the tensile test, the fibre cannot stand to the high load and off from the grip of the matrix. Compare with the treated fibre in **figure 4.14 (b) and figure 4.15 (b)**, the adhesion is strong between the matrix and fibre make the quantity of fibre pull out that left on the surface are greater than untreated. The good interaction between the fibre and matrix produce the good experimental data of tensile and flexural test (Razali, 2016).

The bubble that appears on the top of composites surface at both of treated and untreated composites happened due to the processing method and curing process. The sample composites are not been vacuum to release the air trap and bubble at the surfaces make the air produce and trap between the fibre and matrix.

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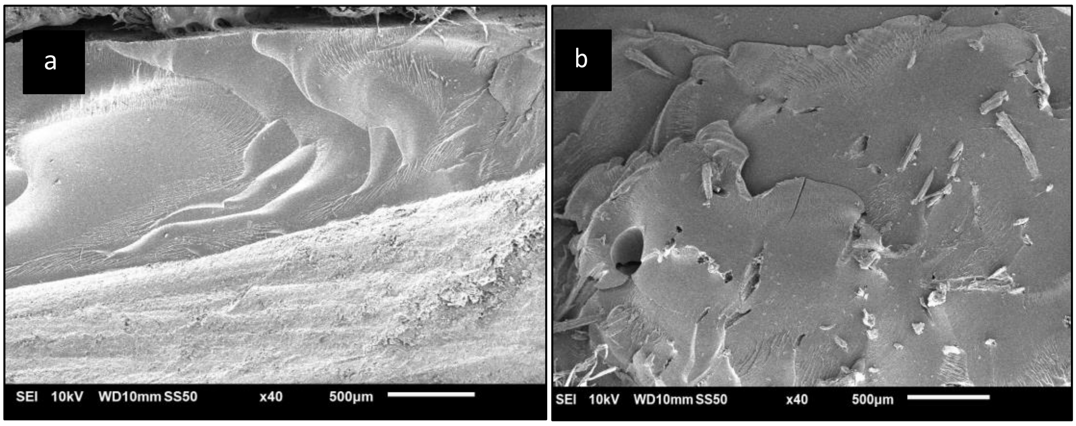


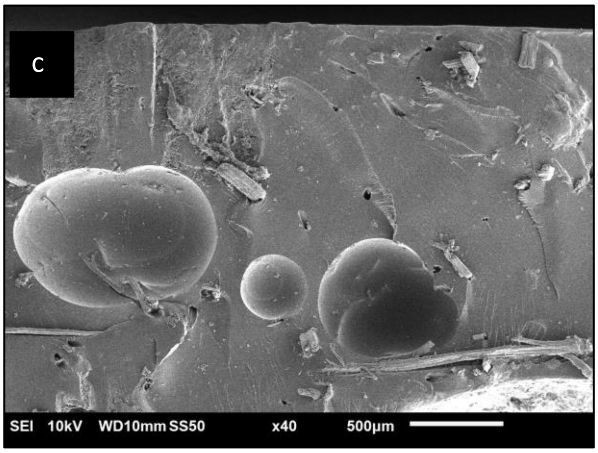
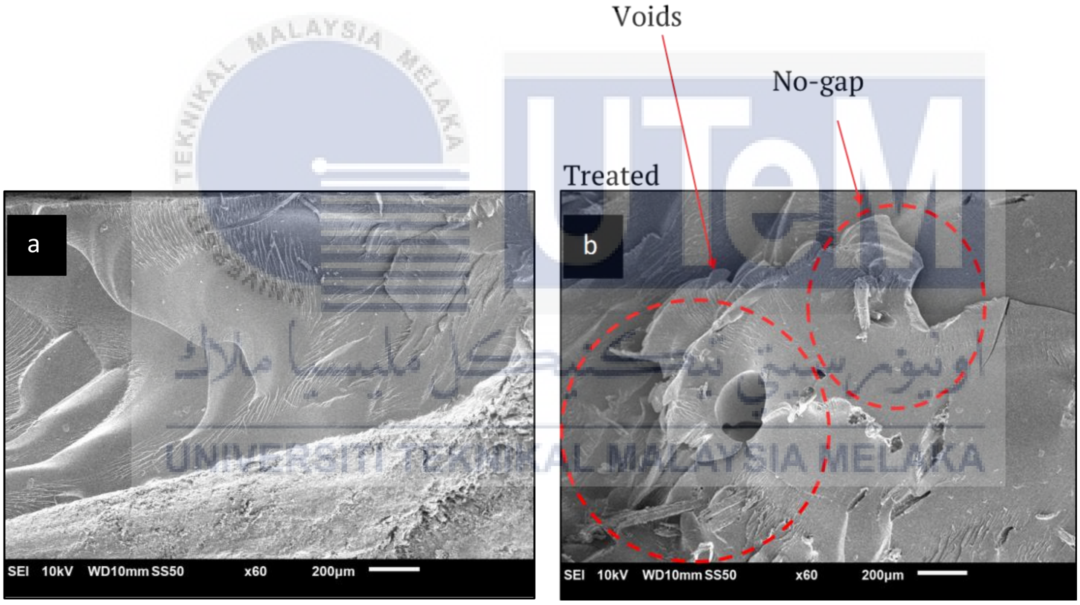


Figure 4.14 (a) Neat polymer 40x **(b)** Treated composites 40x **(c)** Untreated composites 40x



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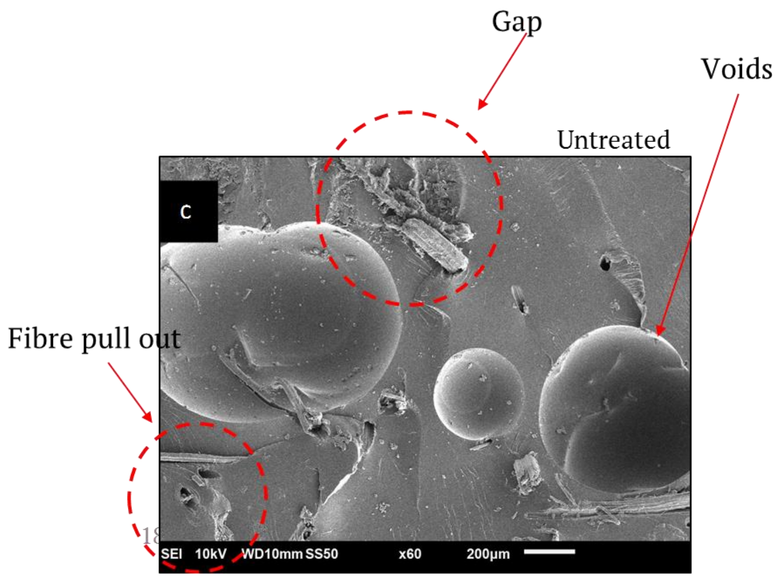

Figure 4.15 (a) Neat polymer 60x **(b)** Treated composites 60x **(c)** Untreated composites 60x

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CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

For many of years, researchers trying to discover the correct way to produce the high-performance material by using natural fibre as reinforced. Many methods that had been conducted to process natural fibre by combining with polymer matrix. For this find-out research, it is more focusing on the effect of using chemical treatment towards kenaf fibre reinforced on the mechanical, physical and morphological properties.

In this experiment, the optimum weight percentage (5 wt%) of the particle kenaf fibre had been measured to blend with the specific weigth of vinyl ester and three different sample was produce which is neat polymer (0 wt% of fibre) and treated and untreated with 5 wt% kenaf fibre. As a comparison between these two treated and untreated chemical treatments, it obviously shown from the data that treated kenaf fibre have much better properties compared to untreated one.

The effect of alkaline treatment with 6% concentration of NaOH is clearly enhanced the interlocking bonding between the matrix and the fibre, the chemical content of the natural fibre itself especially lignin, hemicellulose and cellulose and remove the impurities at the natural fibre that may affect the performance of the composites. The surface treatment also strengthens the mechanical properties such as tensile strength and flexural strength.

For future study, the improvement on handling the reinforced and matrix process should be enhanced to prevent the failure towards the sample. The study about optimum weight with the optimum concentration of Sodium Hydroxide (NaOH) also should be emphasize in order the natural fibre surface can be cleaned thoroughly to reduce the number or voids and impurities. The optimum time of immersion time of kenaf fibre also have to be learned to ensure that the natural fibre chemical content can be reduce perfectly so the bonding between the matrix and reinforcement are in a good term. As conclusion, the further study should be done before conducting experiment so the high-performance composites can be produced without facing any side problems.

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APPENDIX A

ASTM D3039/D3039M-00

Designation: D 3039/D 3039M – 00$^{\epsilon 1}$

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.

ϵ^1 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

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[1] This test method is under the jurisidiction 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.

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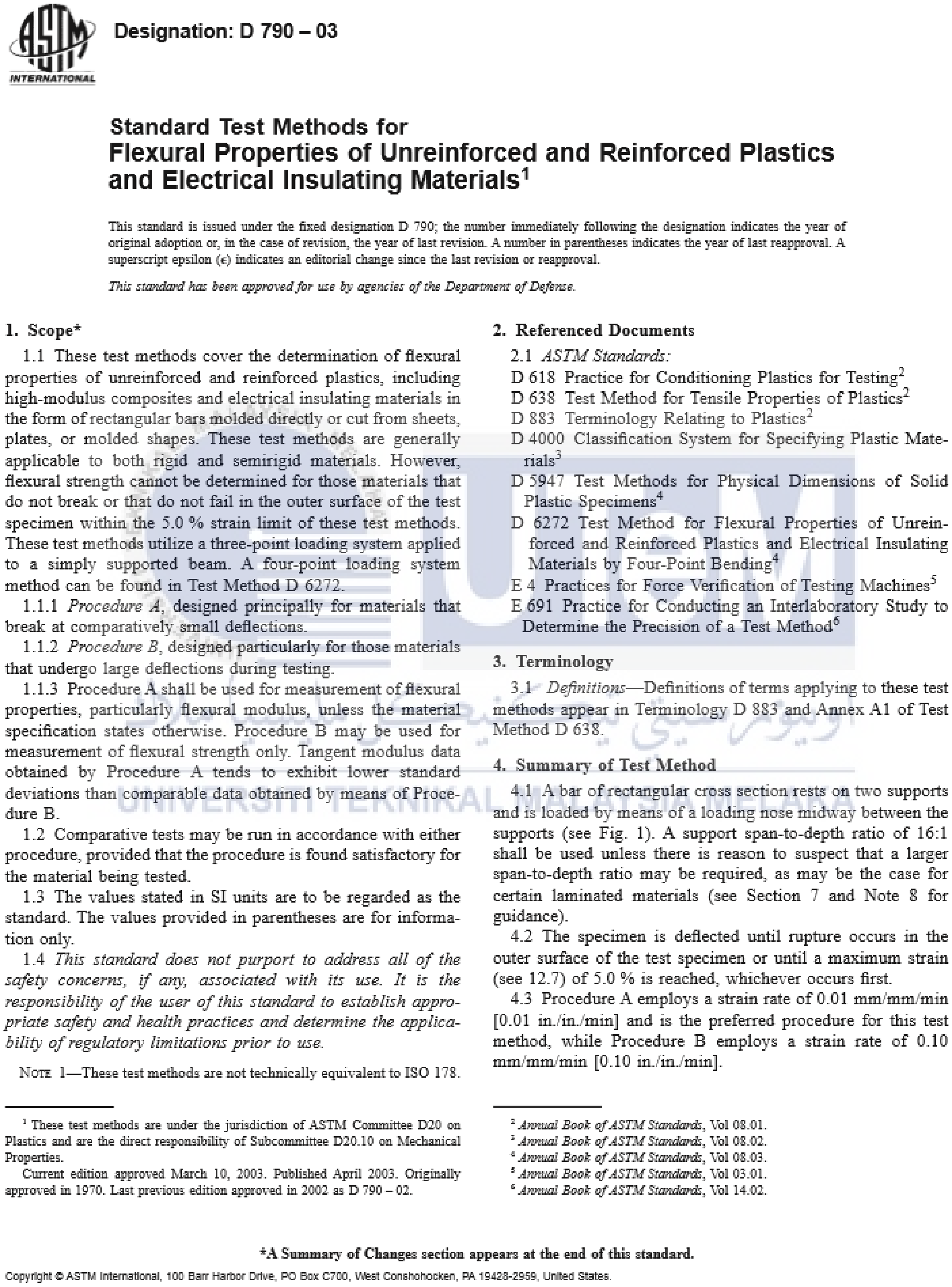
APPENDIX B

ASTM D790-03



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].

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[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.**

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APPENDIX C

ASTM D570-98

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 (⅛ 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.

Current edition approved July 10, 1998. Published January 1999. Originally published as D 570 – 40 T. Last previous edition D 570 – 95.

[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, 1985, 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.

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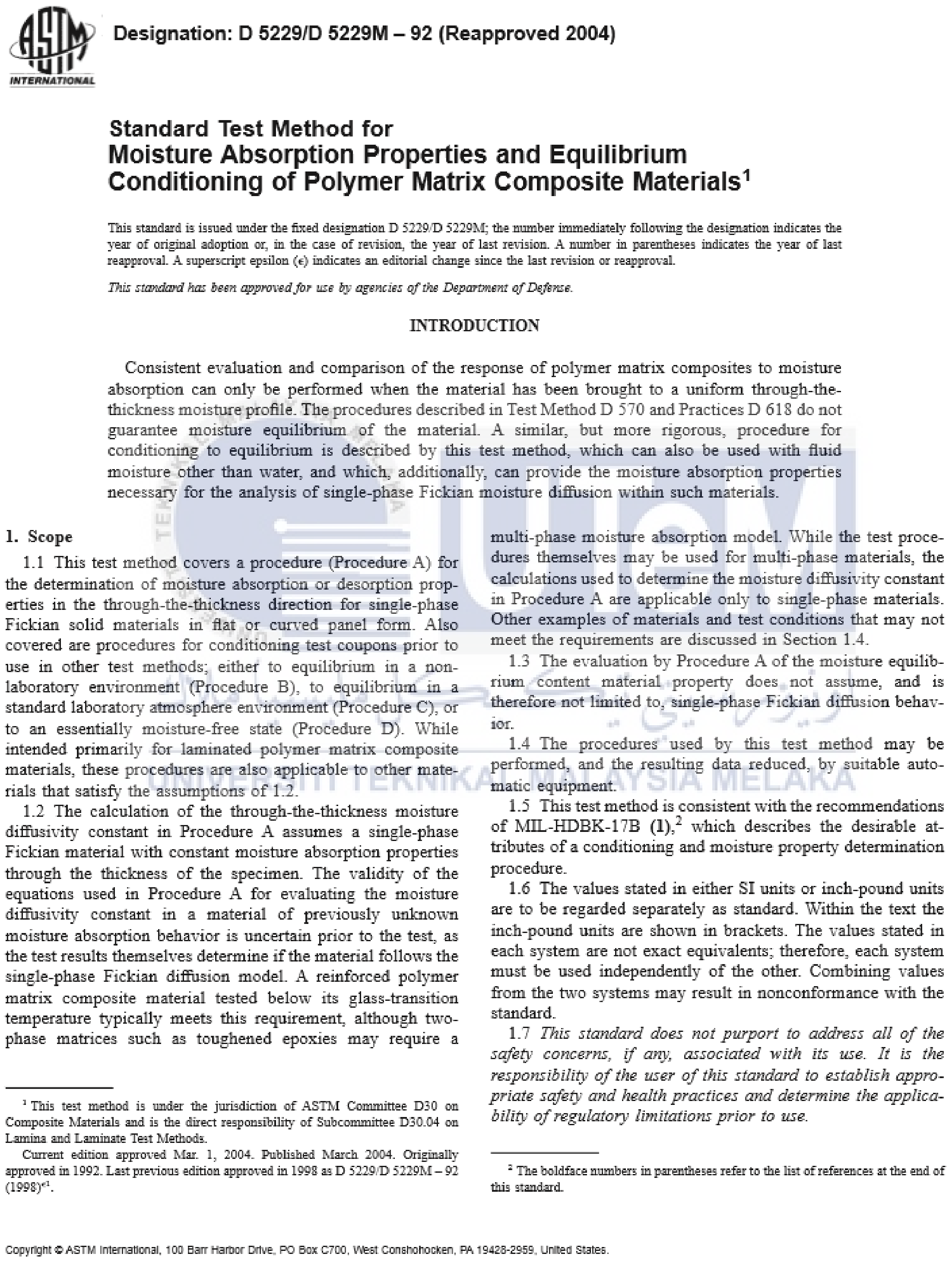
APPENDIX D

ASTM D5229/D5229M-92



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.

Current edition approved Mar. 1, 2004. Published March 2004. Originally approved in 1992. Last previous edition approved in 1998 as D 5229/D 5229M – 92 (1998)$^{\epsilon1}$.

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

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