



UNIVERSITI
MALAYSIA
KELANTAN

Effect of Chemical Treatment on Mechanical and Water Absorption Properties of Kenaf Fibre Mat-Wood Sawdust Reinforced Unsaturated Polyester Hybrid Composite

by

NurMunirah Binti Ismail

A thesis submitted in fulfillment of the requirements for the degree of Bachelor of Applied Science (Material Technology) with Honours

**Faculty of Earth Science
UNIVERSITI MALAYSIA KELANTAN**

DECLARATION

I hereby certify that the work embodied in this report is the result of the original research and has not been submitted for bachelor degree to any other University or Institution.

- OPEN ACCESS** I agree that my report is to be made immediately available as hard copy on on-line open access (full text).
- CONFIDENTIAL** (Contain confidential information under the Official Secret Act 1972)*
- RESTRICTED** (Contains restricted information as specific by the organization where research was done)*

I acknowledge that University Malaysia Kelantan reserve the right as follows.

1. The report is the property of University Malaysia Kelantan.
2. The library of University Malaysia Kelantan has the right to make copies the purpose of research only
3. The library has the right to make copies of the report for the academic exchange

Certified by:

Signature

Date:

Signature of supervisor

Name:

Date:

Note: * If the report is CONFIDENTIAL OR RESTRICTED, please attach the latter from the organization stating the period and reasons for confidentiality and restriction.

ACKNOWLEDGEMENT

Bismillahirrahmanirrahim. Praise to Allah, the Lord of the Universe for His bounties bestowed upon us.

First of all, I would like to take this opportunity to present my greatest appreciation toward those who have been helping me in completing this final year project. A thousand of appreciations are dedicated to my advisor, Dr Mohamad Bashree bin Abu Bakar for his high willingness to spend his time and guiding me from the beginning of my project. I will always remember all of his guidance, advice, opinion and also support.

I also want to present a lot of thanks to my family for the support and prayers for me and always stay by my side by giving good advice for me. Importantly, the high appreciation to all my friends, classmates, lab asistant and also a master student, a million thanks for your views and opinions regarding to my project. Lastly, I would like to thanks for all whose involve to help me directly or indirectly in this project.

Thank you.

UNIVERSITI
MALAYSIA
KELANTAN

TABLE OF CONTENT

	Pages
ACKNOWLEDGEMENT	i
TABLE OF CONTENT	ii
LIST OF TABLES	v
LIST OF FIGURE	vi
LIST OF SYMBOLS	vii
LIST OF ABBREVIATIONS	viii
ABSTRAK	ix
ABSTRACT	x
CHAPTER 1 : INTRODUCTION	1
1.1 Background of Study	3
1.2 Problem Statements	4
1.3 Objectives	4
1.4 Expected Outcome	5
CHAPTER 2 : LITERATURE REVIEW	6
2.1 Reinforcement of Composite	6
2.1.1 Natural Fibre	6

2.1.2 Kenaf Plant	7
2.1.3 Timber Plant	7
2.1.4 Hybridization Fibres Reinforcement	8
2.2 Matrix in Composite	9
2.2.1 The Classification of Matrix Composite	9
2.2.2 Thermoset Matrix	9
2.2.3 Unsaturated Polyester	10
2.3 Chemical Treatment	12
2.3.1 Introduction to Chemical Treatment	14
2.3.2 Effect Chemical Treatment on Mechanical Properties	17
2.3.3 Effect Chemical Treatment on Water Absorption Properties	21
CHAPTER 3 : MATERIALS AND METHODS	22
3.1 Materials	23
3.2 Equipments	24
3.3 Methodologies	25
3.3.1 Chemical Treatment	25
3.3.2 Alkaline Treatment	26

3.3.3 Peroxide Treatment	27
3.3.4 Composite Fabrication and Preparation	28
3.3.5 Flexural Test	31
3.3.6 Water Absorption Studies	32
CHAPTER 4 : RESULT AND DISCUSSION	34
4.1 Fourier Transform Infrared Spectroscopy Analysis	34
4.2 Flexural Test	38
4.3 Water Absorption Studies	41
4.3.1 Water Uptake	41
4.3.2 Thickness Swelling	44
CHAPTER 5 : CONCLUSION AND RECOMMENDATIONS	46
5.1 Conclusion	46
5.2 Recommendations	48
REFERENCES	49

LIST OF TABLE

		Page
Table 2.1	Composition of some Natural Fibre	5
Table 3.1	Formulation of Composite	22
Table 3.2	Alkaline reatment Formulation	26
Table 3.3	Hydrogen Peroxide treatment Formulation	27
Table 3.4(a)	Fixed Composite Composition	29
Table 3.4(b)	Composite Fabrication	29
Table 4.1	Maximum Water Uptake of UPE Composites	43
Table 4.2	Maximum Thickness Swelling of UPE Composites	45

LIST OF FIGURE

	Page
Kenaf Flower	7
Kenaf Plant	7
Lumber	8
Kenaf Fibre Mat	22
Wood Sawdust	22
Metal Roller	23
FTIR	23
Compression molding	24
Type of Chemical Treatment	25
Chemical Treatment Process	28
Composite Fabrication	30
Flexural Testing	31
Research Flow Chart	33
FTIR spectra	35
Flexural strength of UPE composites	40
Flexural modulus of UPE composites.	41
Water Absorption Process	42
Water uptake of UPE composites	43
Thickness Swelling of UPE Composite	45

LIST OF SYMBOL

%	Percentage
°C	Degree Celcius
cm	Centimeter
g/cm^3	Gram per centimeter cube
g	Gram
m	Meter
mm	Millimeter
mm/min	Millimeter per minutes
kg/ms^{-1}	Density
vol %	Volume Percentage
wt %	Weight percentage
h	Hour
cm^{-1}	Reciprocal centimeter
μm	Micrometer

UNIVERSITI
MALAYSIA
KELANTAN

LIST OF ABBREVIATIONS

KFM	Kenaf Fibre Mat
UPE	Unsaturated Polyester
MPa	MegaPascal
GPa	GigaPascal
NF	Natural Fibre
WS	Wood Sawdust
NaOH	Sodium Hydroxide
MEKP	Methyl Ethyl Ketone Peroxide
CN	Cobalt Naphthalene
H ₂ O ₂	Hydrogen Peroxide
FTIR	Fourier Transform Infrared Spectroscopy
KFM/WS	Kenaf Fibre Mat hybrid Wood Sawdust
100% UPE	Nute UPE
UPE/UK-S	Untreated KFM/WS Composite
UPE/1NK-S	1% treated NaOH KFM/WS Composite
UPE/3NK-S	3% treated NaOH KFM/WS Composite
UPE/2PK-S	2% treated H ₂ O ₂ KFM/WS Composite
UPE/4PK-S	4% treated H ₂ O ₂ KFM/WS Composite
W _i	Initial Weight
W _f	Final Weight
T _i	Initial Thickness
T _f	Final Thickness

**KESAN RAWATAN KIMIA TERHADAP SIFAT MEKANIK DAN
PENYERAPAN AIR BAGI GENTIAN KENAF TERANYAM-
SERBUK KAYU DIPERKUKUH KOMPOSIT HIBRID POLIESTER**

TAK TEPU

ABSTRAK

Dalam kajian ini, komposit hibrid gentian kenaf teranyam-serbuk kayu diperkukuh polyester tak tepu (UPE) telah dihasilkan dengan menggunakan gentian kenaf teranyam yang tidak dirawat dan yang telah dirawat. Komposit yang terdiri daripada dua jenis gentian semulajadi telah dihasilkan melalui proses hibrid. Komposit hibrid antara KFM dan serbuk kayu (WS) telah terhasil dengan menggunakan teknik melayang atas dan diteruskan dengan proses acuan mampatan. KFM telah dibasuh, dikeringkan dan disisir dengan elok menggunakan berus nilon. KFM telah dirawat dengan dua jenis rawatan kimia yang berbeza iaitu rawatan alkali dan rawatan peroksida. KFM yang tidak dirawat turut diguna untuk menyediakan komposit yang bertindak sebagai komposit kawalan. Selepas menjalani rawatan kimia, gentian kenaf teranyam sama ada yang telah dirawat atau tidak dirawat, kedua-duanya telah menjalani ujian Fourier Transform Infrared (FTIR) dan seterusnya digunakan untuk menghasilkan komposit hibrid bersama serbuk kayu dengan komposisi bahan yang telah ditetapkan. Setiap komposit hibrid yang berjaya dihasilkan akan diteruskan dengan ujian kelenturan dan ujian penyerapan air. Keputusan yang terhasil telah menyatakan ada peningkatan dalam kekuatan kelenturan dan pengurangan dalam proses penyerapan air disebabkan oleh rawatan kimia terhadap gentian. Hal ini disebabkan oleh penyingkiran lignin, selulosa separa, pectin and lain-lain bendasing. Ini menunjukkan, peningkatan dalam gentian yang mengandungi banyak selulosa dalam sesuatu aplikasi ckomposit boleh ditingkatkan melalui rawatan kimia gentian. Daripada proses penyerapan air, ketebalan komposit membengkak diambil kira dan keputusan mendapati, semakin banyak air diserap, semakin tebal komposit membengkak.

**EFFECT OF CHEMICAL TREATMENT ON MECHANICAL AND
WATER ABSORPTION PROPERTIES OF KENAF FIBRE MAT-
WOOD SAWDUST REINFORCED UNSATURATED POLYESTER
HYBRID COMPOSITE**

ABSTRACT

In this study the treated and untreated kenaf fiber mat (KFM) reinforced unsaturated polyester composites were prepared and hybridization of two types of natural fibres reinforced unsaturated polyester (UPE) composites was fabricated. Hybrid composites of KFM and wood sawdust (WS) were created by lay-up technique and continue with compression moulding. The KFM was retted enzymatically, washed, dried and combed with a nylon brush. The KFM was treated with two type of chemical treatment to composite fabrication. There were alkaline treatment and peroxide treatment. Untreated KFM was also used to prepare the composites to serve as control. After chemical treatment, the treated and untreated KFM undergo Fourier Transform Infrared Spectroscopy (FTIR) process and continues to composite fabrication with the fixed material composition. Finish composites were subjected to mechanical test which was flexural test and water absorption test. The results obtained indicated an increase in all the parameters tested with the fiber treatment. This is because during the chemical treatment, the lignin, hemi-celluloses, pectin and other impurities had removed. This shows that, the enhancement of the natural cellulosic fibers to different composite applications can be achieved via fibre chemical treatments. From water absorption process, the thickness swelling of each composite measured and the result had was shown that the higher water absorption the bigger the thickness swelling.

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Natural fibres reinforced polymer composites have used in many applications, because of their various desirable properties which include high specific strength and high specific stiffness and environmentally friendly. In recent years, natural fibres reinforced unsaturated polyester are used in many applications such as automotive, sporting goods, marine, electrical, industrial, construction, and household appliances (Wallenberger et al. 2004). A number of investigations have been conducted on several types of natural fibres such as kenaf, hemp, flax, wood, bamboo, and jute to study the effects of these fibres on the mechanical properties of composite materials (Satyanarayana et al., 1990; Mansur and Aziz, 1983).

Hybrid composites contain more than one types of reinforcement materials in a single polymer matrix. In principle, several different fibre types may be incorporated into a hybrid, but it is more likely that a combination of only two types of fibres would be most beneficial (Chamis and Lark 2014). They have been developed as a logical sequel to conventional composites containing one fibre. Hybrid composites have unique features that can be used to meet various design requirements in a more economical way than conventional composites. This is because expensive synthetic fibres like glass, Kevlar, graphite, boron can be partially replaced by less expensive natural fibres such as kenaf and jute. Some of the specific advantages of hybrid composites over conventional composites include balanced strength and stiffness, balanced bending and membrane

mechanical properties, balanced thermal distortion stability, reduced weight and/or cost, improved fatigue resistance, reduced notch sensitivity, improved fracture toughness and/or crack arresting properties, and improved impact resistance (Chamis and Lark 2014).

However, natural fibre reinforced unsaturated polyester composite also have some disadvantages such as the incompatibility between the hydrophilic fibres and hydrophobic thermoplastic, as well as thermoset matrices requiring appropriate treatments to enhance the adhesion between the fibre and the matrix (Gassan and Cutowski, 2000; Dhakal et al., 2007). Therefore, this study is necessary in order to evaluate the interfacial adhesion strength between untreated kenaf fibre and chemically treated kenaf fibre reinforced composite.

In this study the treated and untreated kenaf fibre mat/bamboo sawdust reinforced unsaturated polyester composites will be prepared by using hand lay-up technique followed by compressing moulding. There is only kenaf fibre mat will be chemically treated by alkaline and saline treatments.

1.2 Problem Statement

The composites reinforced with synthetic fibres are very popular because they demonstrated good mechanical performance. Besides, the composite made from synthetic fibres have the superior strong and great stiffness characteristics but not environmentally friendly. The use of synthetic fibre can be hazardous, not environmental friendly and can affect the living things for example cancers and other diseases. All of these problems could be solved by replacing the use of synthetic fibre with natural fibres. Moreover, natural fibres have prefer been used as reinforcing components in composites due to their certain advantages such as high specific strength and stiffness, cheap, abundant renewable resources and more environmental friendly materials.

However, the natural fibres have a lot of drawbacks in several aspects therefore they are not able to fulfill the need of demand in its applications. Caused by hydrophilic behavior, natural fibres show lack of compatibility with the hydrophobic polymers. Then, the loads are not full enough transferred between fibre and matrix when the composites used as structural materials. So, the fibre-matrix interfacial adhesion is the main problem in natural fibre reinforced polymer. This problem is handled by conducting chemical treatment on fibres to improve the interfacial adhesion but the composite reinforced with single layer of natural fibre is not strong enough. The hybrid composite will be introduced in order to enhance certain properties that can improve the performance of properties better than the single one.

1.3 Objectives

There are two objectives for this research:

- To fabricate the kenaf fibre mat (KFM)/wood sawdust (WS) reinforced unsaturated polyester composite (UPE).
- To determine the effect of chemical treatment on mechanical and water absorption properties of KFM/WS reinforced UPE composites.

1.4 Expected Outcome

From this research, the use of hybrid reinforcement is expected to meet various design requirements in a more economical way in terms of strength, cost as compared to conventional composites. Moreover, the chemical treatments on the composite formulation are expected to improve fibre-matrix interfacial adhesion. As a result, the mechanical and water absorption properties of the hybrid composite could be enhanced as well, and produce the high performance of structural product for certain application.

CHAPTER 2

LITERATURE REVIEW

2.1 Reinforcement of Composite

2.1.1 Natural Fibre

Natural fibres have advantages such as cheap and environmentally friendly material comparing with glass synthetic fibres. These advantages make natural fibre have been used as reinforcing components in thermoplastic composites. Researchers concluded that polymer composites on the reinforcement role of many types of natural fibres like jute, kenaf, bamboo, flax, husk, sisal, pineapple leaf, banana, coir, wood, and so on (Bledzki et al., 1999). The applications of natural fibre composites are very familiar in automotive, aerospace parts, sporting tools and architecture field. The great performances of natural fibre are light-weight, strong enough and resistance to corrosion (Furtado et al., 2014).

Table 2.1: Composition of some natural fibre

Natural Fibre	Cellulose(%)	Lignin(%)	Hemicellulose(%)
Kenaf	44-57	15-19	930
Bamboo	26-43	21-31	140-230
Palm Oil	65	19	200-250
Sugar cane	55	15	290
Jute	47-71.	12-26	13.6-21
Hemp	57-77	3.7-13	14-22.4
Abaca	56-63	7-9	15-17
Sisal	47-78	7-11	10-24

2.1.2 Kenaf Plants

The scientific name of kenaf was *Hibiscus cannabinus*, was a plant in the Malvaceae family. *Hibiscus cannabinus* was in the genus *Hibiscus* and was probably native to southern Asia, though its exact natural origin is unknown. The name also applies to the fibre obtained from this plant. Kenaf was one of the allied fibres of jute and shows similar characteristics.

KFM was a good replacement for glass fibres as reinforcement, because the natural fibre had competitive strength-to-weight ratio contributions. The natural fibre provides good adhesion and therefore superior properties, low cost, with renewability and biodegradability being some of the other merits over synthetic fibres and low density. Low density of kenaf makes it possible for it to make automobiles more fuel efficient

The fibres in kenaf are found in the bast and core. Crude fibre separated from the bast was multi-cellular, including of several individual cells stuck together. The individual fibre cells are about 2–6 mm long and slender. The cell wall was thick (6.3 μm). The core was about 60% of the plant and has thick ($\approx 38 \mu\text{m}$) but short (0.5 mm) and thin-walled (3 μm) fibre cells.

Kenaf has a single, straight, and unbranched stem consisting of two parts, namely an outer fibrous bark and an inner woody core. The attractive features of kenaf grow quickly, rising to heights of 4-5m in a 4-5 month growing season and 25-35 mm in diameter. with high biomass output, broad growth area, strong adaptability to environment, and low cost in cultivated condition. Furthermore, the KFM composites have excellent strength and renewability. Kenaf has a bast fibre which contains 75%

cellulose and 15% lignin, and it offers the advantages of being biodegradable and environmentally safe.



Figure 2.1: Kenaf Flower(Rabar et al. 2014)



Figure 2.2: Kenaf Plant(Mohd Hadi et al. 2013)

2.1.3 Timber Plants

Timber plants were considered as a giant and plants that have hard woody stem. They can be characterized depending on their variation in size, growth habit, sun tolerance, soil moisture needs and heat/cold temperature tolerance. One of the benefits of using wood fibers was that they were abundant natural resources in Asia and Middle & South America. Wood fibres were often known as natural fibre because of its high strength with respect to its weight derives from fibres longitudinally aligned in its body (Okubo *et. al.*, 2004). The tensile strength of wood was relatively high and they can reach 370 MPa (Thwe *et. al.*, 2003). Hence, this makes wood as an attractive alternative to steel in tensile loading application. They also have the density of between 0.6 – 1.1 g/cm³.

Other than that, wood fibre composites can supplement and will, eventually replace petroleum based composite materials in several applications, thus offering new agricultural, environmental, manufacturing and consumer benefit (Krishnaprasad *et. al.*, 2009; Han *et. al.*, 2008 and Chung *et. al.*, 2002). Overall, the potential of these composites to have positive impact on material for the mechanical properties were great.



Figure 2.3: Lumber

2.1.4 Hybrid Fibre Reinforcement

Hybrid composites defined as the mix of two or more reinforcing materials in one matrix to get the better performances than the single one (Fu et al., 2002). Common hybrid composites reinforced with natural fibres are combining with synthetic fibres such as glass because they can produce good mechanical performance (Jawaid et al, 2011). Researcher investigated that the hybrid composite can be done by combining the two of natural fibre. However, hybrid composites that involved of two types of natural fibres as reinforcing materials still face the problems. Combining sisal and oil palm

fibres, hemp and KFM and kenaf and pineapple leaf fibre are some examples of studies of hybrid composites reinforced by two types of natural fibres (Aji et al., 2011).

Hybrid composites contain more than one type of fiber in a single matrix material. In principle, several different fiber types may be incorporated into a hybrid, but it was more likely that a combination of only two types of fibers would be most beneficial (Chamis et al., 2012). They have been developed as a logical sequel to conventional composites containing one fiber. Hybrid composites have unique features that can be used to meet various design requirements in a more economical way than conventional composites. This was because expensive fibers like graphite and boron can be partially replaced by less expensive fibers such as glass and Kevlar (Chao et al., 1980). Some of the specific advantages of hybrid composites over conventional composites include balanced strength and stiffness, balanced bending and membrane mechanical properties, balanced thermal distortion stability, reduced weight and/or cost, improved fatigue resistance, reduced notch sensitivity, improved fracture toughness and/or crack arresting properties, and improved impact resistance (Chamis et al., 2012).

2.2 Matrix in Composite

2.2.1 The Classification of Matrix in Composite

There are three type of polymeric matrix in composite which are thermoset resin, thermoplastic resin or biodegradable resin.

2.2.2 Thermoset Matrix

Thermosets have low viscosity that can provide great fibre wetting. Unsaturated polyester, vinyl esters and epoxy are commonly used as thermoset matrices. Catalyst was used to start the chemical reaction of these matrices by transferred from a liquid to solid phase. Thermosets undergo irreversible chemical cross-linking reactions upon heating and curing. Cured thermosets are insoluble materials. Besides, cured matrices have well mechanical properties, which easily processed and resisted to environmental degradation. High glass transition and high decomposition temperature, high ultimate strength and modulus were mechanical properties of thermoset matrix (Hayes et al., 2010). However, cured matrices are brittle which results in poor resistance to crack propagation.

A lot of research work about the kenaf reinforced thermoset composites. Researchers revealed that treated KFM reinforced in epoxy improved the flexural strength of the composite while untreated fibres perform the lower enhancement (Yousif et al., 2012). The KFM reinforced polyester composites are fabricated by hand-layout technique showed higher disintegration rates and load carrying capacity by composites that having higher fibre content without presenting any significant progress at very high number of cycles (Abdullah et al., 2012).

2.2.3 Unsaturated Polyester

Unsaturated polyester resins (UPE) are the condensation products of unsaturated acids or anhydrides and diols with/without diacids. UPE are compounded with varied fillers, reinforcements and cured by free radical initiators to yield thermoset articles having a wide range of chemical and mechanical properties depending upon the choice

of diacids, diols, cross- linking agents, initiators and other additives (Boenig et al., 1964). This versatility of the final thermoset product properties associated with comparatively low cost has renewed the interest in these resins as an important matrix material for many applications.

General purpose unsaturated polyester resins were prepared by using maleic anhydride, phthalic anhydride and propylene glycol with the molar ratio of phthalic anhydride: maleic anhydride ranging from 1:2 to 2:1 (Schmidt et al., 1970). Chemical resistant thermosetting unsaturated polyester resins for laminates having low content of volatile organic components were prepared from isophthalic acid, maleic anhydride and propylene glycol with 35% styrene as reactive diluents (Howard et al., 2001). Unsaturated polyester composites are known for their poor resistance to aromatic solvent, due to the styrene as a reactive diluent.

Unsaturated polyester resins are the most commonly used thermoset resins in the world. Unsaturated polyester resins are produced by chemical reaction of saturated and unsaturated di-carboxylic acids with alcohols. Unsaturated polyester resins form highly durable structures and coatings when they are cross-linked with a vinyl reactive monomer, most commonly styrene. The properties of the cross-linked unsaturated polyester resins depend on the types of acids and glycols used and their relative proportions. Their versatility in use allows unsaturated polyester resins to be used in a myriad of composite applications. Composite parts can be made at temperatures as low as 15°C to as high as 150°C depending on the processing requirement of the application. Unsaturated polyester resins also have excellent service temperatures.

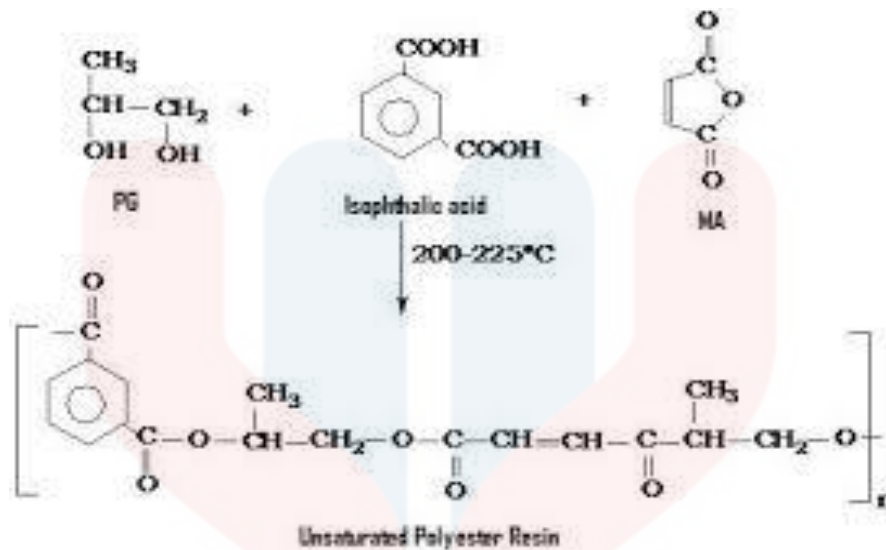


Figure 2.4: Production and curing of UPE resin (Dholakiya,2012)

2.3 Chemical Treatment

2.3.1 Introduction of Chemical Treatment

Natural fibre reinforced thermoset matrices have poor mechanical properties due to low interface bonding between the fibres and matrix. To improve the interfacial bonding, the reinforcing fibre can be modified by using different chemical treatments. Chemical treatments allow cellulose surface to react with the matrix (Dash, et al. 2000). Strong fibre-matrix interface bonding can be achieved in greater mechanical properties of the composites. Several investigations have been conducted to enhance fibre adhesion properties through chemical treatments. There is several type of chemical treatment as it alkaline treatment, acetyl treatment, peroxide treatment, isocyanate treatment and others.

Surface modification of fiber can be done by using chemical treatments such as alkali treatment, potassium permanganate treatment, benzoyl chloride treatment and acrylic acid treatment in order to optimize the effective interfacial bonding between

natural fibers and thermoset matrix so to improved properties can be obtained. From the chemical treatment, chemical reaction involved and the changes occurred during the chemical treatments of natural fibers (Dhanalakshmi et al., 2015). Chemical treatments improved chemical interlocking at the interface by exposing more reactive groups on the natural fiber surface. Further, based on the study of areca fibre, chemical treatment of areca fiber surface became more hydrophobic and there was enhancement in surface characteristics such as adhesion and porosity of areca fibers. Also, there was improvement in interfacial adhesion between the treated areca fiber surface and matrix.

The natural fibers consist of several components, such as hemicellulose, cellulose and lignin (E.Cheng et al, 2004). Some fibers consist of oil and wax components. Natural fibers are amenable to chemical modification due to the presence of hydroxyl groups. Thus, the adhesion interface between natural fibers and polymer matrices has often been a vital issue in several natural composite materials. The properties between natural fiber and polymer matrix cause poor adhesion interface when both materials were combined. There were because of the natural fibers was hydrophilic and the polymer matrix was hydrophobic. Therefore, the surface of the fiber has to be treated in order to improve adhesion interface. Results from studies using acetylation treatment, saline treatment, and bleaching treatment, showed that the treatment on the surface of the fiber can improve the mechanical properties of composites.

The study of complex nature of fiber was important to optimize the modification processes that increased the role of fiber as part of the component in composites. According to researcher, the polymers and fibers have different chemical properties that produce effective adhesion for an excellent transfer of stress and bond distribution throughout the network. Based on the adhesion performance, several studies includes

chemical bonding, water absorption and acid or base interaction act as a driving force for bonding network.. In this study, the effect of alkaline treatment using sodium hydroxide (NaOH) on the fibers properties were investigated using Fourier transform infrared spectroscopy analysis.

2.3.2 Effect of Chemical Treatment on Mechanical Properties

The mechanisms that enhanced the interfacial adhesion between kenaf-UPE composites by employing alkaline treated kenaf fibre in kenaf-UPE composites. Thus, chemically surface treatments considerably improved the modulus of elasticity, modulus of rupture, tensile strength (bonding) of the formulated kenaf-UPE composites. The pronounced effects of chemical treatment of the fibre on the composite properties are explored fabricated by hand lay-up method (Ren et al., 2012).

From the chemical treatment, the improvement of interfacial adhesion between the fibres and matrix directly affects the tensile properties of natural fibre reinforced polymers. Moreover, increasing the fibre content (hybrid or mix fibre) also leads to increase in Young's modulus and tensile properties up to certain maximum value of the natural fibre polymer composites. The treated kenaf fibre for both the woven hybrid and random mix composite improved the mechanical properties because the permeability of the treated kenaf fibres reinforced polymer composite (Thiruchitrabalam et al., 2009). The chemical treatment towards the increment of untreated and treated composites tensile strength, elastic modulus also shows considerable improvement in the matrix-filler adhesion. From the review, the weaker transference of load between fibre and matrix occurred due to the poor interfacial adhesion causing lowering in the mechanical properties (Yakubu et al., 2014).

Besides, silane treatment generally coats the fibre surface to create a chemical bridge and increasing the bonding strength between the natural fibre and the matrix (Sgriccia et al., 2008). Chemical treatment also reduces the hydrophilic nature of a natural fibre and improves the lifespan by reducing development of fungi (Leman et al., 2008). Further than that, the investigation of the effects of chemical treatment using stearic acid on mechanical bonding strength between flax fibre and polypropylene concluded that acid treatment modified flax fibre surface but not the fibre bulk (Pothen et al., 2003).

Tribological performance and mechanical properties of treated and untreated kenaf fibre reinforced composites concluded that the latter shown excellent alternative natural fibre composite (Nishino et al., 2003). Mechanical strength of the kenaf fibre composites relies on the content of fibre in the composites which usually up to 70% of volume (Kim et al., 1992). As the general knowledge from the researcher, mechanical failures of certain composites are mostly due to fibre pullout, fibre debonding, and fibre breakage (Agrawal et al., 2000). Limited literature was available on the effects of fibre treatment and concentration of chemical treatment on the mechanical properties and interfacial adhesion strength of kenaf fibre. Therefore, to evaluate the interfacial adhesion strength between untreated kenaf fibre and chemically treated kenaf fibre impregnated in thermoset (epoxy, polyester) was a very important to investigate.

According to researchers, in the case of hybrid fibres unsaturated polyester hybrid composite fabricated through sheet molding compound process display higher tensile, flexural and impact strength obtained from treated kenaf fibre. The hybrid fibre on treatment with 6% sodium hydroxide using mercerization method for three hour yields better mechanical strength to the composite (Atiqah et al., 2014). Researchers

concluded that kenaf fibre alone (30% volume fraction) or higher percentage (22.5% volume fraction) cannot withstand higher impact load leading to brittleness and less toughness hybrid composite (Ghaani et al., 2012).

A lot of research work also been done with the kenaf reinforced polymer composites. Researchers revealed that treated kenaf fibres reinforced in epoxy improved the flexural strength by about 36% of the composite while, without treated fibres shows only 20% enhancement (Yousif et al., 2012). Owing to strong adhesion at the interface of the fibres through chemical treatment and the permeability of the composites shows to inhibited the detachments, debonding or pull out of fibres (Yousif et al., 2012).

Several studies and research work established that the chemically treated kenaf fibre significantly has improved mechanical properties with respect to untreated kenaf fibre as to support above statement. Researcher also suggested an improvement in impact strength, flexural, tensile and also on the stiffness of the kenaf fibres reinforced composites by using several types of polymers (Anuar et al., 2011).

Recent research showed that significant improvements of these properties can be obtained via fibre treatment. Treated natural fibre composites had being used extensively in many structural applications such as natural fibre reinforced car roof. Moreover, there will be a dramatic increase on the consumption of natural fibre composites in various applications due to overwhelming interest of using natural fibres in the aforesaid mainly in aerospace industry, marine sector, automotive industry, sporting segments, and construction industries (Nirmal et al., 2012). The alkaline treatment and hydrogen peroxide treatment had been done by researcher to prove the enhancement of the mechanical properties of composites.

The research works on hybrid composite with banana also bring some important reports by treating the fibres (banana/kenaf) with 10% Sodium Lauryl Sulfate (SLS) and 10% of sodium hydroxide (NaOH) for 30 min. The SLS treatment has enhanced the flexural, impact and tensile strength for both the woven hybrid and random mix composite. Thus, SLS treatment can provide better mechanical properties as it improved the adhesion between fibre and matrix which in turn improved the mechanical strength than the alkali treated. Moreover SLS treatment also scrubbed fibre surface and decreased the lignin level in fibres by SLS in a better way compared to alkali (Thiruchitrambalam et al., 2009).

The flexural strength and impact strength of natural fiber reinforced polymer composites depends on the nature of the fiber, polymer and fiber–matrix interfacial bonding (Joseph et al., 2003). The hydrophilic behavior of untreated areca fibers and the hydrophobic nature of polymer matrix resulted in clash between the areca fibers and the natural rubber matrix. Hence, the untreated areca fiber reinforced polymer composites indicated low flexural strength values compared to treated areca fiber. Alkali treatment of areca fibers removed hemicelluloses, lignin, pectin, waxy tissue, oil covering materials and reduced areca fiber diameter and effective fiber surface area available for good adhesion with the natural rubber matrix is increased (Dipa et al., 2001, Kumar et al., 2013)

2.3.3 Effect of Chemical Treatment on Water Absorption Properties

Last but not least, unlikable effects to the dimensional stability and mechanical properties of composites also caused by humidity aging or water absorption process (Ghani et al., 2012). Moreover, review also been done depicting the kenaf fibre

mechanical properties which get depreciated by the water penetration into the kenaf reinforced polymer composite either hybrid or non-hybrid even under dissimilar environmental situations including distilled water, rain water and sea water (acidic solutions) at room temperature (Ghani et al., 2012).

In other review work, hybrid composite has been effectively explored by researchers to study the pronounced effect of water absorption on mechanical properties. The fracture toughness shows decrement due to the water absorption displayed that it was been influenced by the fibre orientation, fibre content, exposed surface area, hydrophilicity of the individual component, void content and fibre permeability (Salleh et al., 2012). Furthermore, humidity aging also originated the unwanted effects to the dimensional, stability and fracture toughness properties of composites.

KFM and its composites are hydrophilic materials. Water content in KFM composites significantly affects their physical and mechanical properties that influenced dimensional stability and durability of composite. The water not only affects the vacancy of polymer matrices physically or chemically but also attacks the hydrophilic natural fibre as well as the fibre matrix interface. Unsaturated polyester composites contained styrene as a reactive diluent make it become poor resistance to aromatic solvent. The experiment of water uptake and assuming one dimension diffusion based on Frick's law, several researchers have discussed characteristics of moisture absorption.

Fickian and non – Fickian are the two type of moisture diffusion in polymeric composites (Bonniau et al., 1981). Therefore, natural fibre may be considered as a viable reinforcement in composite materials. The flexural test of kenaf unsaturated polyester

composites have been showed the result that the optimum fibre content was 10wt% (Ishak et al., 2010).

From water absorption and thickness swelling behavior of recycle natural fibre plastic composites investigation, the researcher revealed that the fibre loading depends on a wide array of factors including interface quality, chemical composition, fibre length and distribution and also density. From the result, the recycle process enhances water resistance and dimensional stability in the studied formulations had been confirmed. (Tajvidi et al., 2010). In hybrid composites, the researcher were found that the water absorption of all formulation approach the Fickian diffusion as the mechanism of water transports (Ghasem et al., 2009). The aim of this research was to study the suitability of these composites in outdoor applications. The fracture toughness shows decrement due to the water absorption displayed that it was been influenced by the fibre orientation, fibre content, exposed surface area, hydrophilicity of the individual component, void content and fibre permeability (Salleh et al., 2012). Treatment on natural fibre by NaOH was widely used for both thermoset and thermoplastic composites. This treatment changes the orientation of highly packed crystalline cellulose order and forms amorphous region by swelling the fibre cell wall. This provides more access to penetration of chemicals. Alkali sensitive hydrogen bonds existing among the fibres break down and new reactive hydrogen bonds form between the cellulose molecular chains. Due to this, hydrophilic hydroxyl groups are partially removed and moisture resistance property was improved. It also takes out certain portion of hemicelluloses, lignin, pectin, wax and oil covering materials (Xue, Lope, & Satyanarayan, 2007; Leonard et al., 2007, Dipa, Sarkar, Rana, & Bose, 2001). As a result, the fibre surface becomes cleaner. In other words, fibre surface become more uniform due to the

elimination of micro voids and thus stress transfer capacity between the ultimate cells improves. In addition to this it reduces fibre diameter and thereby increase aspect ratio, which results in better fibre matrix interfacial adhesion (Joseph, 2001). Composite properties such as mechanical and thermal behavior can be improved significantly by this treatment. If the alkali concentration is higher than the optimum condition, excess delignification of fibre take place, which results in weakness or damage to the fibre (Wang et al., 2007; Xue et al., 2007).

CHAPTER 3

MATERIALS AND METHODES

3.1 Materials

There are several materials that will be used in this experiment:

- i. Kenaf Fibre Mat (KFM)
- ii. Wood Sawdust (WS)
- iii. Unsaturated Polyester (UPE)
- iv. Methyl Ethyl Ketone Peroxide (MEKP)
- v. Cobalt Naphthalene (CN)
- vi. Sodium Hydroxide (NaOH)
- vii. Hydrogen Peroxide (H₂O₂)

The reinforcements in this hybrid composite involved of KFM and WS. University Malaysia Kelantan was supplied KFM used in this work and the WS gotten from wood factory. The KFM came in pieces and woods are ready in form of sawdusts. The UPE was prepared as thermoset matrix. To achieve the great reaction in the experiment, MEKP was provided and act as catalyst following by CN as promoter or accelerator that was used in the curing of UPE resins with MEKP catalyst. In chemical treatment of KFM, the chemicals used were NaOH pallets and distilled water for alkaline treatment. Besides, the second chemical treatment was hydrogen peroxide treatment. In this process, the acetone solution, distilled water and H₂O₂ solution needed to run this chemical treatment.

The material compositions in each composite mixed regarding the fixed composition:

Table 3.1: Composition of composite

Matrix (60%)	57% of Unsaturated Polyester + 2% of MEKP + 1% of CN
Fibre (40%)	20% of KFM + 20% of WS



Figure 3.1: Kenaf Fibre Material



Figure 3.2: Wood Sawdust

3.2 Equipment

Hand Lay-Up Roller

Resins impregnated by hand into fibres which were in the form of woven, knitted, stitched or bonded fabrics. This was usually accomplished by rollers or brushes, with an increasing use of nip-roller type impregnators for forcing resin into the fabrics by means of rotating rollers and a bath of resin. Laminates were left to cure under standard atmospheric conditions.



Figure 3.3: Metal Roller

Fourier Transform Infrared Spectroscopy

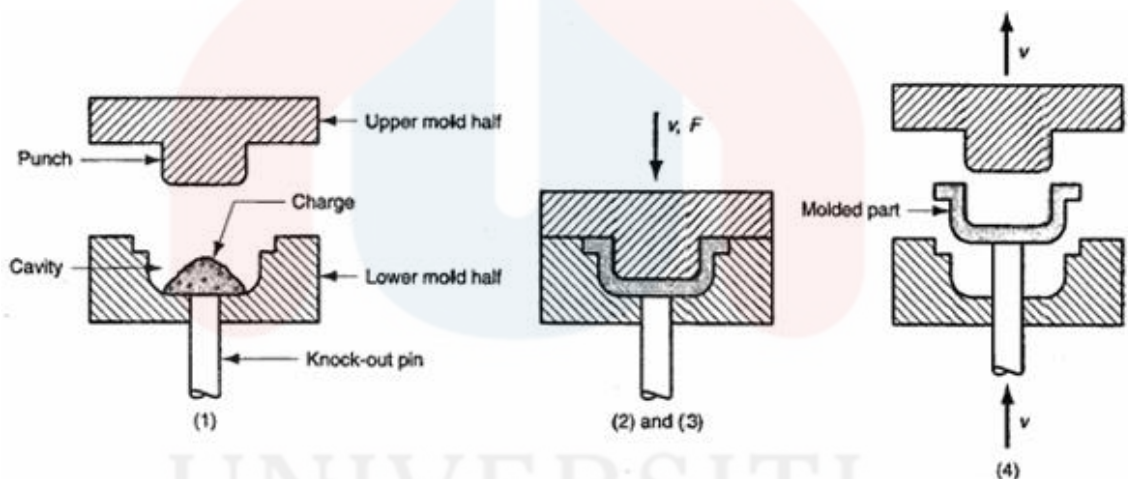
Functional groups of natural fibers were obtained using the Fourier transform infrared spectroscopy (FTIR) test. Infrared spectrum was obtained through infrared solution software which presented information in transmittance mode. The results obtained were in the range of 400 cm^{-1} to 4000 cm^{-1} for both untreated and treated natural fibers samples. The test was prepared according to ASTM E168-06 standards. Based on the results obtained, the functional groups and behavior of the natural fibers were classified and discussed.



Figure 3.4: Fourier Transform Infrared Spectroscopy (FTIR)

Compression Molding

Specifically designed to ease the replacement of metal components with polymer (and other composite), the compression molding process was a method of molding in which a preheated polymer was placed into an open, heated mold cavity. Base on Figure 3.5, polymer was put into the mold and then closed. The pressure was applied to force the material to fill up the cavity. Excess material was transfer away by the overflow channel. The heat and pressure were maintained until the plastic material cured. The final part after the mold was removed.



Compression molding for thermoplastics: (1) charge is loaded, (2) and (3) charge is compressed and cured, and (4) opening the mold halves and removing the part from the cavity.

Figure 3.5: Compression molding

3.3 METHODOLOGY

3.3.1 Chemical Treatment

For the treated KFM and WS, the chemical treatment that will use are alkaline treatment by using Sodium hydroxide (NAOH) solution and the second treatment is hydrogen peroxide treatment (H_2O_2). The composite of treated fibre will be compare to the untreated fibre.

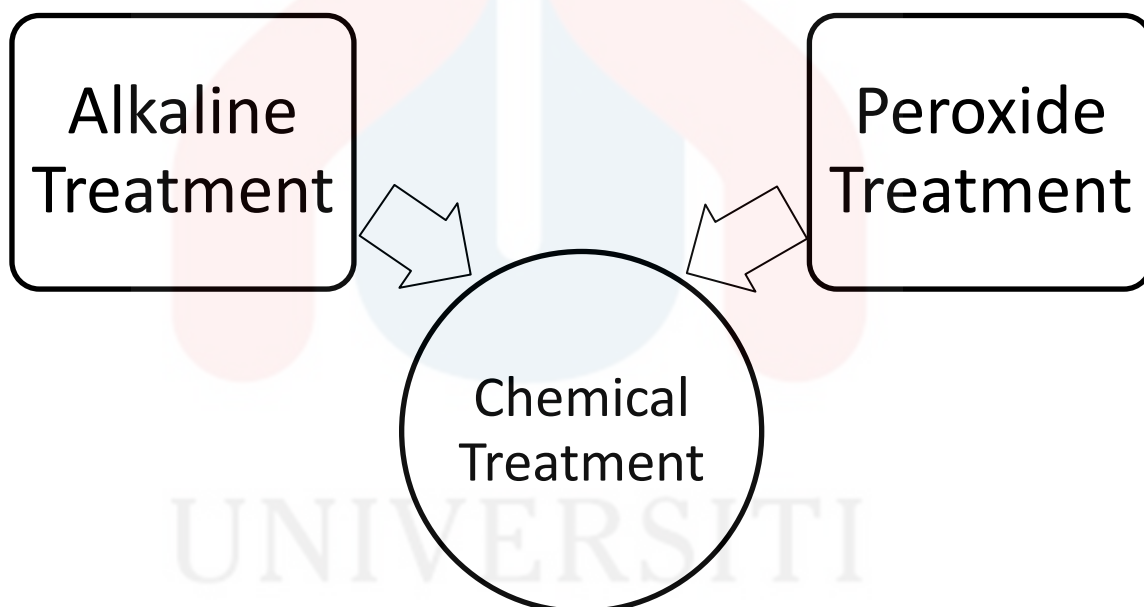


Figure 3.6: Type of Chemical Treatment

3.3.2 Alkaline Treatment

The treatment on natural fibres by sodium hydroxide (NaOH) was being widely used to modify fibre structure. Natural fibre absorbs moisture due to the presence of hydroxyl groups in the amorphous region of cellulose, hemicellulose and lignin constituents. The alkaline treatment process was shown in equation 3.1.



The fibres will treat in two different concentration of NAOH solution which was mixed in distilled water (600 ml). The 1% and 3% aqueous solution of NaOH (by weight) was used in this chemical treatment. Every fibres were soaked in the solution for 4 hours. Subsequently, the fibres washed 6 times with distilled water and dried in oven at 80°C for 24 hours. The treatment regarded by the formulation below:

1% NaOH = 1ml of NaOH

100ml of water = 1ml/3 ml of NaOH

Table 3.2: Alkaline treatment Formulation

Concentration (%)	Composition
1%	594 ml distilled water + 6 ml NaOH
3%	582 ml distilled water + 18 ml NaOH

3.3.3 Peroxide Treatment

Interface properties of fibres and matrix were improved by peroxide treatment. The peroxide induced grafting of polyethylene adheres onto the fibre surface and the peroxide initiated free radicals react with the hydroxyl group of the fibre and matrix. As a result, the great fibre matrix adhesion occurs. This treatment also reduced moisture absorption tendency by the fibre and improved thermal stability (Wang et al., 2007; Kalaprasad et al., 2004). The mechanism of peroxide treatment involved alkali pre-treatment on the fibres and then coated with hydrogen peroxide (2% and 4% concentration) in the acetone solution (400 ml) for 30 minutes. The complete decomposition of peroxide can be achieved by heating the solution at higher temperature (Xue et al., 2007, Kalaprasad et al., 2004). Joseph, Thomas, and Pavithran (1996) investigated optimum concentration of hydrogen peroxide treatment on KFM composites and reported improved flexural strength. The treatment regarding formulation below:

1% H_2O_2 = 1ml of H_2O_2

100 ml of water = 2ml /4 ml of H_2O_2

Table 3.3: Hydrogen Peroxide treatment Formulation

Concentration (%)	Composition
2%	396 ml distilled water + 8 ml NaOH
4%	384 ml distilled water + 16 ml NaOH



Figure 3.7: Chemical Treatment Process

3.3.4 Composite Fabrication and Preparation

Kenaf fibre mat hybrid with wood sawdust (KFM/SW) reinforcement unsaturated polyester composites was prepared by utilizing hand lay-up processes. During composite processing, the 340 mm x 100 mm x 10 mm size of mould was used. The mould was utilized to maintain a consistent thickness throughout the samples, with smooth surface finishes achieved in the final composites.

Initially, the steel mould was waxed several times with a mould releasing agent. KFM/WS mixed with UPE matrix by 40:60 ratios. The ratio defined as 40% of fibre and 60% of matrix. The 40% of fibre divided to 20% of KFM and the other 20% is WS that calculated by weight. Both of fibre fixed at the same weight. 1% cobalt was added to act as accelerator and 2% of the Methyl Ethyl Ketone Peroxide (MEKP) as hardener in this composite preparation. The 60% of unsaturated polyester matrix was spread quickly into

the mould and mixed with the KFM/WS by hand lay-up technique followed by compression moulding.

Once the fibres fully impregnated with the matrix, the composite was cured under mould pressure at room temperature for 24 hours. The laminate released from the mould and kept in an oven at 80°C for 4 hours to post-cure. Seven composite samples were prepared. The prepared samples were shown in Table 3.4(a) and Table 3.4(b).

Table 3.4(a): Fixed Composite composition

Matrix (60%)	57% of Unsaturated Polyester + 2% of MEKP + 1% of CN
Fibre (40%)	20% of KFM + 20% of WS

(a)

Table 3.4(b): Composite Fabrication

Composite	Chemical Treatment	
	NaOH(%)	H₂O₂(%)
100% UPE	0	0
UPE/UK-S	0	0
UPE/1NK-S	1	
UPE/3NK-S	3	
UPE/2PK-S		2
UPE/4PK-S		4

(b)

Calculation of composite material theoretical composition:

$$\text{Density} = \frac{\text{mass}}{\text{volume}}$$

$$\begin{aligned} \text{Mass composite} &= 600 \text{ kg/ms}^{-1} (\text{assume density}) \times (0.34\text{m} \times 0.1\text{m} \times 0.005\text{cm}) \\ &= 0.102 \text{ kg/102 g of composite} \end{aligned}$$

$$\begin{aligned} 40\% \text{ of fibre} &= \frac{40}{100} \times 102 = 40.8 \text{ g} \\ &= 20\% \text{ of KFM (20.4g)} \\ &= 20\% \text{ of WS (20.4g)} \end{aligned}$$

$$\begin{aligned} 60\% \text{ of matrix} &= 102 - 40.8 = 61.2\text{g} \\ &= 2\% \text{ of MEKP (2g)} \\ &= 1\% \text{ of CN (1g)} \\ &= 57\% \text{ of UPE (58.2g)} \end{aligned}$$



Figure 3.8: Composite Samples

3.3.5 Flexural Test

Three point bending test was used in this study. Flexural testing of the polyester matrix (following ISO 178) were conducted using a 10 kN MTS testing machine at a loading rate of 3 mm/min. The specimens were simply supported and tested under a point loading with the span set at approximately 16 times (80mm) the thickness of the specimen. Seven specimens were tested for each sample. The load was applied at mid-span of the specimens, perpendicular to the fibre. The testing showed in Figure 3.6.



Figure 3.9: Flexural Testing

3.3.6 Water absorption Studies

Water Uptake

Water absorption and thickness swelling of samples were conducted according to ASTM D-1037. All the cut samples of every composite dried in oven at 60 °C for 24 hours to make sure all the moisture in composite completely released and got the actual and constant weight of composite sample. Water absorption and thickness swelling tests were conducted by immersing the specimens in distilled water for different time durations. First day going through immersion process, the specimens took out from immersion for every hour to get the current weight and current thickness diameter. The sample surfaces dried using a clean dry cloth. The specimens reweighed 1 min of removing them from the water. The water uptake of specimens was calculated by the weight different. The thickness swelling of specimens were measured to get different size expand by using external viner caliper.

Water absorption (%)

W_i = Initial weight

W_f = Final weight

$$\frac{W_f - W_i}{W_i} \times 100\% \text{-----Equation 3.2}$$

Thickness Swelling (%)

T_i = Initial Thickness

T_f = Final Thickness

$$\frac{T_f - T_i}{T_i} \times 100\% \text{-----Equation 3.3}$$

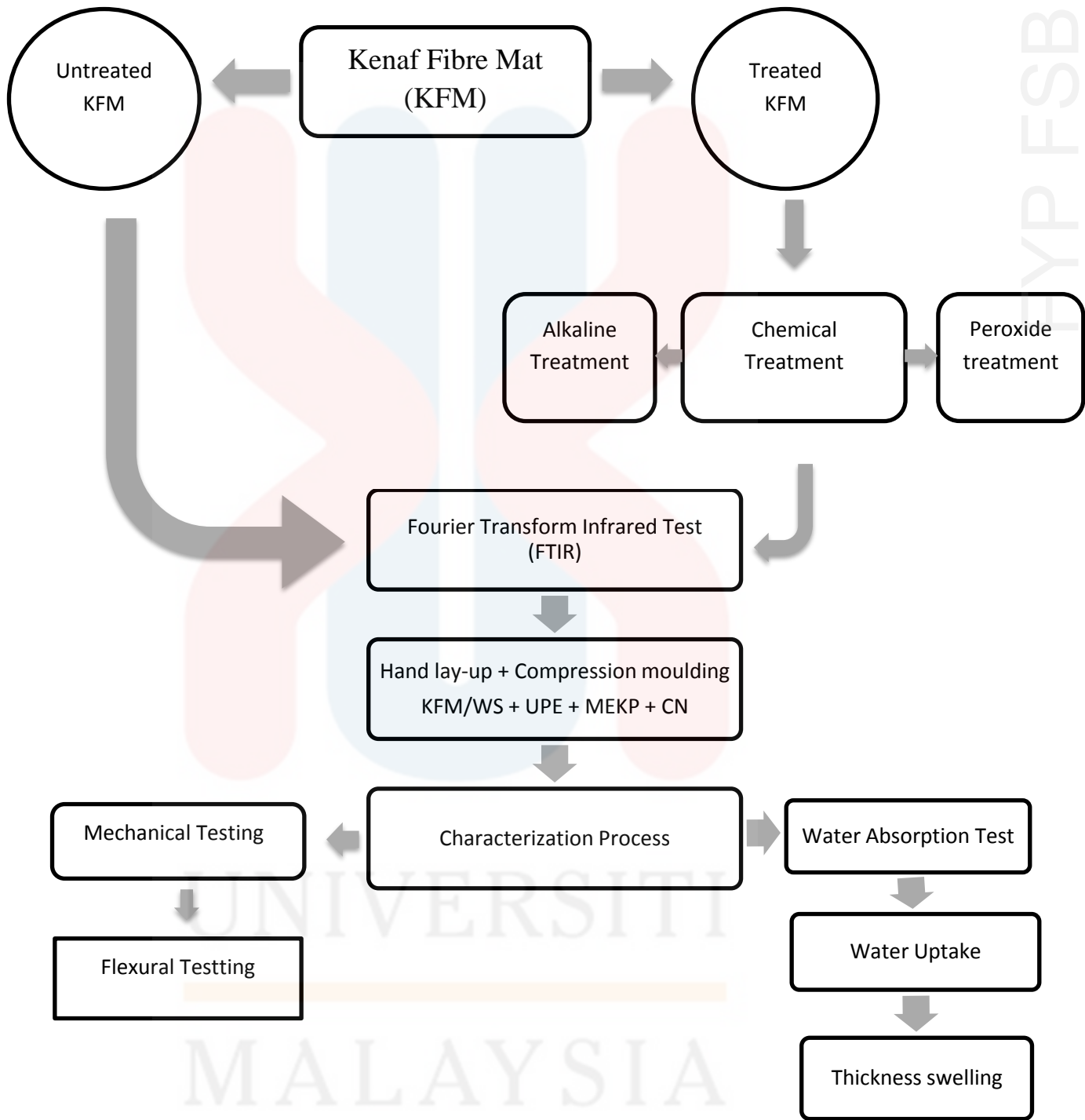


Figure 3.10: Research Flow Chart

CHAPTER 4

RESULT AND DISCUSSION

4.1 Fourier Transform Infrared Spectroscopy Analysis

The Fourier transforms infrared spectroscopy (FTIR) was conducted to study the characteristics of the kenaf fibre mat (KFM), before and after chemical treatment with alkaline (NaOH) and hydrogen peroxide (H_2O_2). The FTIR spectra of the untreated, NaOH treated and H_2O_2 treated KFM are shown in Figure 4.1. The broad absorption band at 3337 cm^{-1} - 3321 cm^{-1} which appeared in all spectra were attributed to the H-bonded and O-H stretching structure that mostly contained major functional groups of phenols, alcohols and waters. The peaks at 2915 cm^{-1} and 2904 cm^{-1} predominantly arise from C-H stretching (Jonoobi et al., 2010).

The O-H stretching and H-bonded broad absorption band in the regions were decreased after alkaline treatment for KFM. The decreased of the broad absorption band attributed to O-H stretching and free hydroxyl bond structure for functional group of alcohol, waters and phenols. The decrease in the functional group of phenolic and aliphatic hydroxyl in the fibre due to reaction with sodium hydroxide promotes free hydroxyl bond structure. According to (Sreekala et al. 1997), the alkaline treatment caused the globular pultrusions present in the untreated fibre disappeared that leading to the formation of a larger number of voids. These voids might promote mechanical interlocking between fibre and matrix. The loss of cuticle by the rupture due to alkaline sensitive bonds leads to the rough surface.

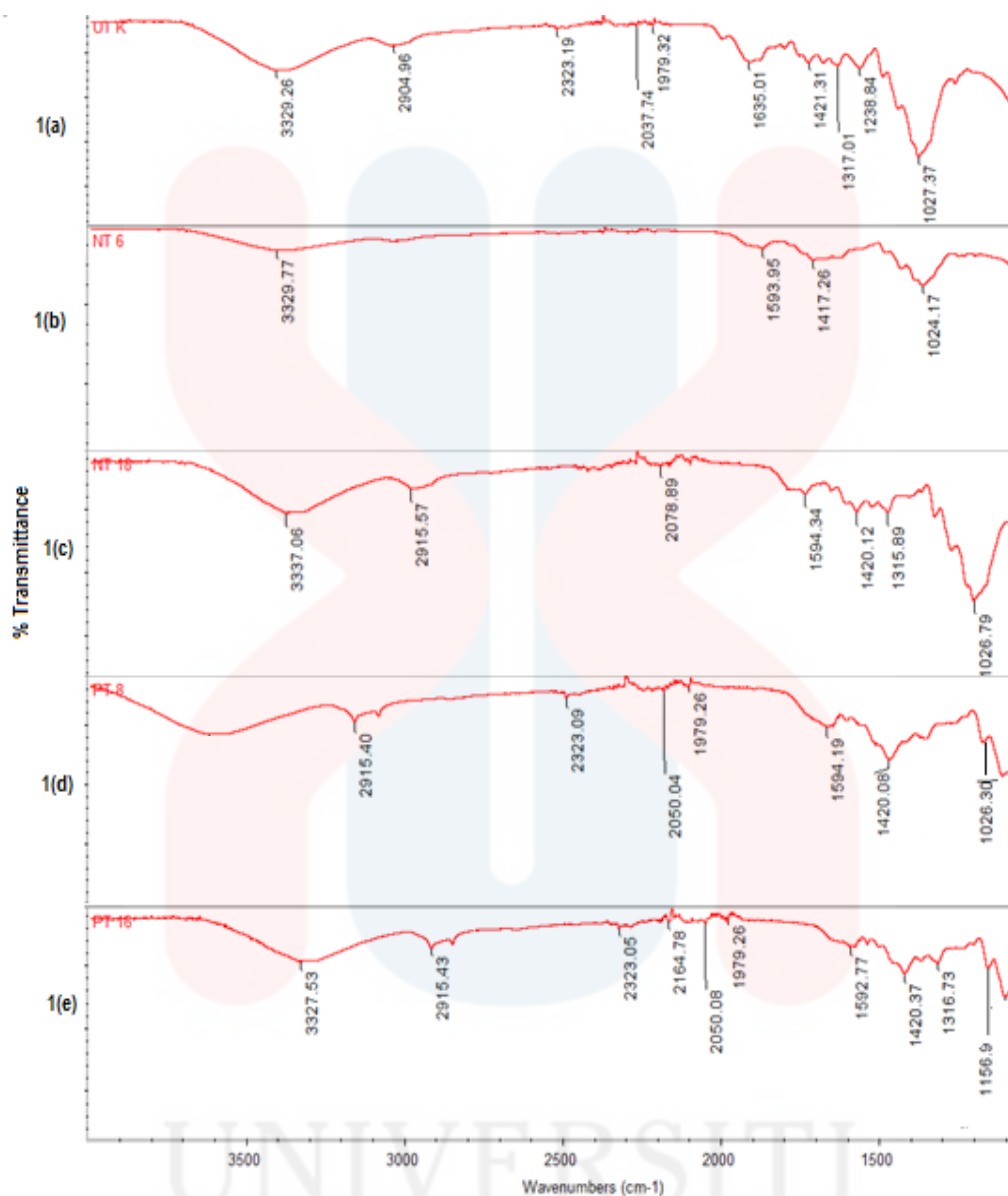


Figure 4.1: FTIR spectra of (a) untreated KFM (UK) (b) 1% NaOH treated KFM (1NK) (c) 3% NaOH KFM (d) 2% hydrogen peroxide treated KFM and (e) 4% hydrogen peroxide treated KFM.

The small peak at 2904.96 cm⁻¹ for untreated kenaf fibre and 2915.96 cm⁻¹ for treated kenaf fibre were attributed to the C-H stretching and O-H stretching bond structure for functional group of alkanes (cellulose and lignin) and carboxylic acids (Dwivedi et al., 2012). The small peak in the region of the C-H stretching bond structure includes the functional group of methyl (CH₃), methylene (CH₂) and aliphatic saturated

(CH) (cellulose and lignin) (Saw et al., 2013). The small peak at 2323.19 cm^{-1} for the untreated kenaf fibre were not change in 1 % sodium hydroxide but disappeared in 3% NaOH for the treated kenaf fibre. This showed that not all concentration tend to have the same behavior and reaction due to alkaline treatment.

The reading of 1635.01 cm^{-1} for the untreated kenaf fibre and 1593.95 cm^{-1} as well as 1594.34 cm^{-1} for the both treated KFM 1% and 3% are characterized as the C=C stretching bond structure of the functional group of alkenes. The decreased in the absorption in the peak were due to removal of lignin. The peak of 1421.31 cm^{-1} and 1236.37 cm^{-1} for the untreated kenaf fibre and 1417.26 cm^{-1} (NaOH 1%) and 1420.12 cm^{-1} (NaOH 3%) for the treated kenaf fibre are characterized as the C-H bending bond structure of the functional group of alkanes. The decreased in the absorption in the peak were due to removal of cellulose, hemicellulose and lignin.

Lastly, the peak 1027.37 cm^{-1} for the untreated kenaf fibre, 1024.17 cm^{-1} (NaOH 1%) and 1026.79 cm^{-1} (NaOH 3%) for the treated kenaf fibre are characterized as the C-O stretching bond structure of the functional group of alcohols. It can be seen that in the untreated kenaf fibre, the peak brought deeper for the treated kenaf fibre for both of sodium hydroxide concentration. The different concentrations of the chemical treatment tend to remove cellulose, hemicellulose and lignin and change in bending or stretching structure. A small peak at 846.61 cm^{-1} for the untreated kenaf fibre is characterized as C-H bond structure of the functional group for aromatic. The peak was totally disappeared that showed alkaline treatment removed the aromatic smell and the lignin structure of kenaf fibre.

Next, FTIR analysis also was conducted to study the characteristics of the kenaf fibres, before and after treatment with hydrogen peroxide (H_2O_2). As depicted in Figure 4.1(d), the broad peak at 3000 cm^{-1} to 3500 cm^{-1} of untreated KFM which appears in all the figure spectra is attributed to the O–H frequency, whereas the peaks at 2904.96 cm^{-1} and 2915.40 cm^{-1} for H_2O_2 2% and 2915.43 cm^{-1} for H_2O_2 4% cm^{-1} predominantly arise from C–H stretching (Jonoobi et al., 2010). Another peak at 2323.19 cm^{-1} to 1979.32 cm^{-1} of untreated KFM corresponds to ester carbonyl vibrations from the acetyl, feruloyl and p-coumaryl groups in lignin.

After chemical treatment, there was a decrease in the intensity of these peaks 2323.09 cm^{-1} - 1979.26 cm^{-1} for 2% of H_2O_2 and 2323.06 cm^{-1} - 1979.26 cm^{-1} for 4% of H_2O_2 indicating that most of the lignin had removed (Sun et al., 2002). A stretching peak detected at 1635.01 cm^{-1} for unbleached fibre is attributed to the carbonyl group of the acetyl ester in hemicellulose and the carbonyl aldehyde in lignin (Hamdan et al., 2010). An absence of this peak after the chemical treatment was due to the removal of lignin and hemicellulose. For treated kenaf fibre, the disappearance of the vibration peak at 1238.84 cm^{-1} that corresponds to C–O vibration was also attributed to the removal of lignin (Fávaro et al., 2010).

4.2 Flexural Properties

Several authors reported that modification of fibres improved the mechanical properties of composites. Pre-impregnation of cellulosic fibres in alkaline solution (Herrera-Franc P et al., 1997), silane treated fibres, benzoylation, polystyrene maleic anhydride coating and acetylation of fibre (Nair et al., 2001) and cyanoethylation of fibres (Saha et al., 1999) have been reported to improve fibre matrix adhesion and hence the mechanical properties of natural fibre composites.

Flexural strength was measured under a three-point bending approach using a universal testing machine according to ISO 178. The dimensions of the samples were 34mm x 10mm x 5mm. The distance between the spans was 80mm, and the strain rate was 3 mm/min. The seven specimens were tested for each case, the average was reported as a results. The flexural strength of 100% UPE composite showed the best flexural strength properties (618.74MPa). However, the effect of chemical treatment on the flexural strength properties of KFM/WS reinforced unsaturated polyester was shown in Figure 4.2. Results show that mechanical properties were strongly changed with chemical treatment. In this case, the flexural strength of UPE/4PK-S composite showed the best flexural strength properties (522.02MPa) which was nearly 6% more than UPE/3KF-S of composite (486.35Mpa). Compared to this last composite, the flexural strength in both composites UPE/1NK-S and UPE/2PK-S increased significantly by 30% and 12%, respectively. The result concluded that, the treated fibre was stronger than untreated fibre and the higher the concentration of chemical treatment, the higher the strength of composite. This was due to bonding of the fibre with the polyester matrix

there by improving the fibre-matrix interaction compare to UPE/UK-S (391.46MPa) which was the lowest strength.

From the Figure 4.2, it was observed that the flexural strength of the UPE/1NK-S was considerably 1.05% lower than UPE/UK-S composite. The composite from treated fibre should be stronger than untreated but the remarkable reduction in the mechanical properties found in the composite UPE/1NK-S maybe mainly the result of the longer treatment time of fibre (Mishra et al. 2012). Researcher reported that 5% NaOH treated sisal fibre-reinforced polyester composite had better flexural strength than 10% NaOH treated composites. This was because at higher alkali concentration, excess delignification of natural fibre occurs resulting in a weaker or damaged fibre. The flexural strength of the composite decreased drastically after certain optimum NaOH concentration (Bledzki et al .2012). Base on the result, UPE/3NK-S and UPE/4PK-S had higher flexural strength than UPE/1NK-S and UPE/2PK-S. In this case, at over low alkali concentration, less delignification of natural fibre occurs resulting in not strong enough to remove hydroxyl group in fibre. Overall, the hydrogen peroxide treatment (H_2O_2) produced greater flexural strength of composite compared to sodium hydroxide treatment (NaOH).

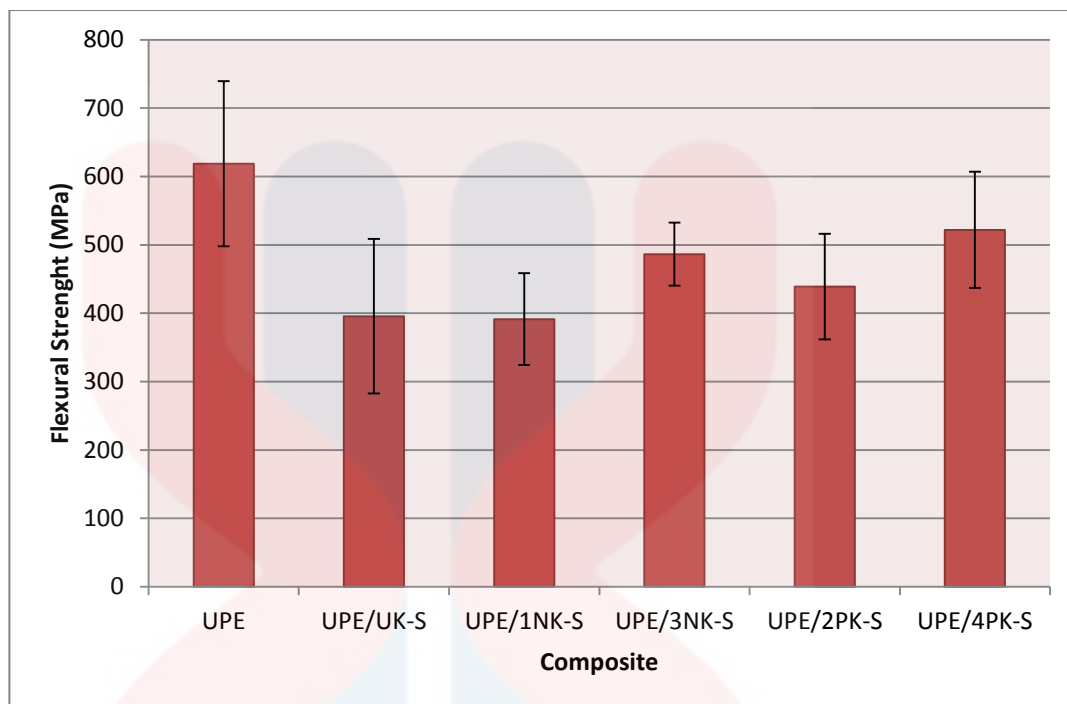


Figure 4.2: Flexural strength of UPE composites.

Three-point bend tests showed that, the chemical treatments of KFM/WS had also a significant effect on flexural modulus. From Figure 4.3, it was clearly seen that, the UPE/4PK-S composite showed the highest flexural modulus which increased by 19% compared to UPE/UK-S composite. This remarkable increase in flexural modulus may be an indication of better adhesion between and the unsaturated polyester and treated KFM/WS. From the Figure 4.3, it was observed that the flexural modulus of the composite UPE/2PK-S was considerably same those for the untreated UPE/UK-S composite. It was compared to UPE/UK-S composite, the improvement in flexural modulus irrelative to both materials.

UPE/1NK-S and UPE/3NK-S were about 45% and 55% respectively. It was observed that the flexural modulus of the treated UPE/KFM-WS increases with an

increase NaOH concentrations as same as H₂O₂ treatment that the flexural modulus increases with an increase of H₂O₂ concentrations.

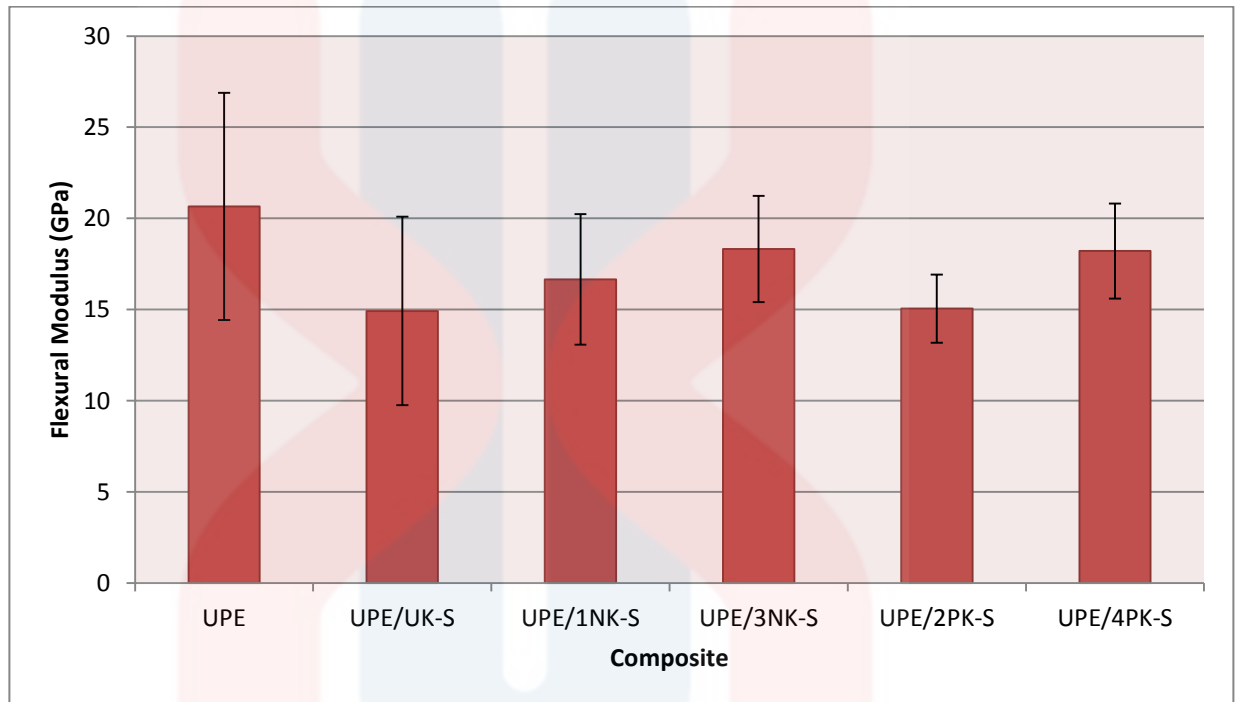


Figure 4.3: Flexural modulus of UPE composites.

4.3 Water Absorption Studies

4.3.1 Water Uptake

The water absorption of UPE/KFM-WS composites at the different hours were studied and shown in Figure 4.4. UPE/1NK-S composites with the treated kenaf fibre were shown lesser value than untreated KFM. Kenaf fibre material was a hydrophilic polar which tends to absorb water and resulting the composite to higher water content compared with the 100% plastic matrices (Kuo et al., 2009). In this study, alkali treatment showed a significant positive impact on the water absorption behavior of the

composites as shown in Figure 4.4. The UPE/UK-S was the highest tendency absorbed water. The first attempt of alkali treatment on the UPE/1NK-S with 1% NaOH showed a slight decrease in water absorption behavior cause by removal of OH group.



Figure 4.4: Water Absorption Process

However, an apparent decrement of water absorption was observed when the alkali concentration increased up to 3% (UPE/3NK-S composite). Dissolving the hemicellulose and volatile extractives had contributed in reducing the numerous hydroxyl groups that were available on the fibre surfaces. This was then resulting in less interaction of the fibre and water molecules as more rigid and crystalline packed of cellulose was left. On the other hand, accumulated of the hydrogen peroxide (H_2O_2) concentrations towards 2% (UPE/2PK-S composite) resulted in slightly increment of water absorption behavior on the composite at the end of the time. But somehow, the increment had no significant difference with the 4 % (UPE/4PK-S composite). In general, 4% concentration of H_2O_2 seems to be the optimal concentration in gaining a good water absorption behavior for the UPE/KFM-WS composite. Low of water absorption level showed the best composite behavior.

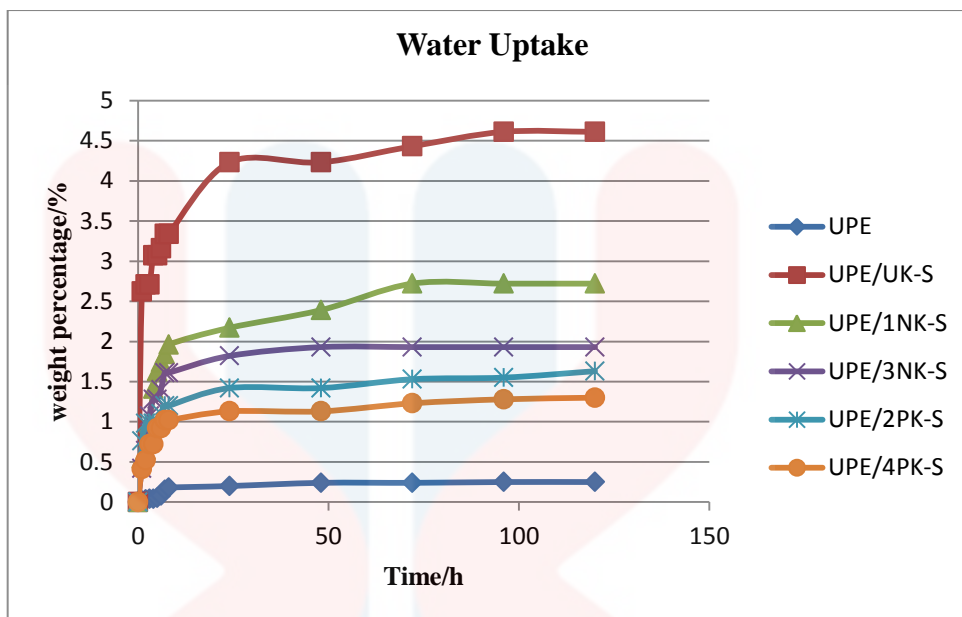


Figure 4.5: Water uptake of UPE composites.

Table 4.1: Maximum water uptake of UPE composites

Composite	Maximum water uptake (%)
UPE	0.25
UPE/UK-S	4.61
UPE/1NK-S	2.72
UPE/3NK-S	1.93
UPE/2PK-S	1.63
UPE/4PK-S	1.30

4.3.2 Thickness Swelling

Moisture absorption also resulted in the swelling in the lignocellulose of fibre composite. This was proven in this research in which as the changes in the specimen thickness were measured right after water immersion. The percentage of thickness swelling was used to measure the moisture concentration in the composite. The effects of water absorption on the thickness of UPE/KFM-WS composites sample were shown in Figure 4.5. Base on the Figure 4.5, UPE had lowest value compared to other composite. This was due to the polyester formed a rigid and 3D structure in which they linked by double hydrogen bonding which resulting in less void produced in the composite.

The kenaf fibre changed dimension with changing moisture content due to the cell wall of polymers contain hydroxyl and other oxygen-containing groups that attracts moisture the process of hydrogen bonding. During the immersion process, the cell wall of fibre expended until saturated with water. Saturation stage had shown the water act as free water in the void structure and does not contribute to any cell wall expansion. UPE/3NK-S had shown lesser thickness swelling than the UPE/UK-S composites due to the lesser water absorption. According the reference, the alkaline and peroxide chemical treatment had minimized the water absorption. When the water absorption less, the thickness swelling of treated KFM became less compared with untreated one.

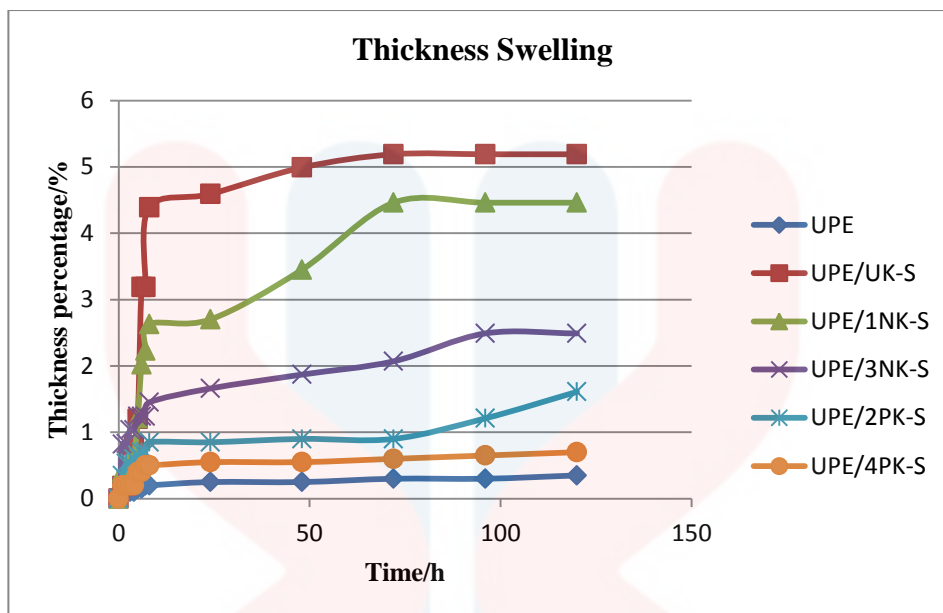


Figure 4.6: Thickness Swelling of UPE Composite

Table 4.2: Maximum Thickness Swelling of UPE composites

Composite	Maximum Thickness Swelling (%)
UPE	0.35
UPE/UK-S	5.19
UPE/1NK-S	4.46
UPE/3NK-S	2.49
UPE/2PK-S	1.61
UPE/4PK-S	0.70

CHAPTER 5

CONCLUSION AND RECOMANDATIONS

5.1 Conclusion

In the conclusion, modification in the structure and the removal of cellulose, hemicellulose, lignin, waxes and other impurities during the chemical treatment affected the improvement on the adhesion interface of fibers. It produced unsmooth, void and tortuous structure of the fibers that cause different natural frequencies that produce different strength and moisture absorption. The rough surfaces caused better adhesion between fiber and polymers.

From the FTIR result, it can be conclude that chemical treatment can caused reduction of materials inside the fiber structure by the removal of unstable materials with rupture bonds. Furthermore, the chemical treatment obtained crystalline structures of fibers. The chemical treatment also changed the color of natural fiber. The color changes of natural fiber were because of the natural dye inside the natural fiber materials was removed. Besides that, due to change in the structure and surface roughness of the fiber, the strength of treated fibre were different than the untreated fiber. The removal of certain molecules such as hydroxyl and free radical group were also caused the changes in the strength and water absorption of composite.

The modification in the fiber caused swelling in the fibers. Moreover, the fiber may either be more stiffness or less stiffness depend on the behavior of chemical treatment. Other than that, the changes in the molecular bond structure of O- or H- bond

either by stretching or bending, the fiber became more brittle or ductile as it reacted with chemical treatment.

The effect of fibre chemical treatment on the flexural strength of composites was concluding that the flexural strength increases with increasing of chemical concentration of the treatment. It was shown in UPE/3NK-S and UPE/4PK-S flexural test result. The result concluded that the higher flexural properties at higher concentration of chemical treatment were due to the increase of interfacial adhesion between the fibre and matrix. Base on the overall result, hydrogen peroxide treatment demonstrated greater flexural strength and modulus than alkaline treatment. Generally, there were increases flexural strength of composites with treated fibre compared untreated.

The effect of fiber chemical treatments on the water absorption of composites was investigated by increase the immersion time. It was concluded that the water absorption process was sharp at the beginning and levelled off for some length of time where it approached to equilibrium. Generally, the rate of water absorption was greatly affected by the hydrophilic behavior and void content in the fibre. The high hydrophilic behavior was due to the high polar hydroxide groups in the fibre. That's why the chemical treatment needed to break the hydrogen bonding in fibre to reduce hydrophilic behavior. The low water absorption created the great of composite strength.

5.2 Recommendations

In the future, this research should be done further by using variation type of treatment such as physical treatment, plasma treatment and corona treatment by using other natural fibre like jute, sugarcane, coir and others. Besides, the research can carry out by the hybrid of reinforced composite trough natural fibre (NF) with other polymer matrix composite such as thermoplastic polymer composite and biodegradable polymer composite. From this hybrid reinforced composites, it can be used to compare the physical, mechanical and chemical properties of composite matrix.

For further research, the preparation of composites involving hot compression which can be spread the matrix more evenly so that the result later on might improve and the quality of the composite will increase. Lastly, more research work can be conduct on the woven fabric. The fabrics produced are impregnated with a thermoplastic or thermosetting resin by hand lay-up technique or compression moulding to enhance better out-of-plane strength and stiffness than laminate and unidirectional composites. When a strand is punctured or bent woven fabric in a weave exposed to bearing load, the surrounding fibres withstand the stress without subsequent failure of the composite. This is unlikely to occur in unidirectional strands where the fibres can only withstand tensile, compressive, and flexural loads parallel to the loading axis.

REFERENCES

- Abdul Khalil, HPS., Yusra, AFI., Bhat, AH., & Jawaid, M. (2010). Cell wall ultrastructure, anatomy, lignin distribution, and chemical composition of Malaysian cultivated kenaf fibre. *Indices Crops Production*, 31, 113–21.
- Atiqah, A., Maleque, MA., Jawaid, M., & Iqbal, M. (2014). Development of kenaf-glass reinforced unsaturated polyester hybrid composite for structural applications. *Composite Part B Engineering*, 56, 68–73.
- Agrawal, R., Saxena, N. S., Sharma, K. B., Thomas, S., & Sreekala, M. S. (2000). “Activation energy and crystallization kinetics of untreated and treated oil palm fibre reinforced phenol formaldehyde composites,” *Materials Science and Engineering A*, vol. 277, 77–82
- Anuar, H., & Zuraida, A. (2011). Improvement in mechanical properties of reinforced thermoplastic elastomer composite with kenaf bast fibre. *Composite Part B Engineering*, 42, 462–5.
- Batouli, SM., Zhu, Y., Nar, M., & D’Souza, NA. (2014). Environmental performance of kenaf fibre reinforced polyurethane: *A Life Cycle Assessment Approach*. *J Clean Production*, 66, 164–73.
- Bonniau, P., & Bunsell, A. R. (1981). “Comparative study of water absorption theories applied to glass epoxy composites”. *Composite Materials*, Vol. 15, 272-293,
- Bledzki, A.K., & Gassan, J. (1999). Composites reinforced with cellulose based fibres. *Progress Polymer Sciences*, 24, 221-274.
- Cordes, R. D. & Daniel, I. M., (1995). “Determination of interfacial properties from observations of progressive fibre debonding and pullout,” *Composites Engineering*, vol. 5, 633–648
- Chung, K.F. & Yu W.K. (2002). Mechanical properties of structural bamboo for bamboo scaffoldings. *Engineering structures*, 16, 711 – 729
- Cheng E, Sun X, Karr G.S, (2004). Adhesive properties of modified soybean flour in wheat straw particleboard, *Composites A: Applied Science and Manufacturing*, 35 (3): 297-302.
- Dhanalakshmi S, Ramadevi P and Basavaraju B. (2015). Effect of chemical treatments on tensile strength of areca fiber reinforced natural rubber composites. *IOSR Journal of Applied Chemistry (IOSR-JAC)*. 8(5): 43-52
- Dipa R, Sarkar B.K, Rana A.K, et al., (2001). Effect of alkali treated jute fibers on composite properties. *Bull Mater Sci*, 24(2):129-135.

- Edeerozey, AMM., Akil, HM., Azhar, AB., & Ariffin, MIZ. (2007). Chemical modification of kenaf fibres. *Mater Letter*, 61, 2023–5.
- Fu, S.Y., Xu, G. & Mai, Y.W., (2002). On the elastic modulus of hybrid particle/shortfibre/polymer composites. *Composite*, 33, 291-299.
- Ghani, MAA., Salleh, Z., Hyie, KM., Berhan, MN., Taib, YMD., & Bakri, MAI. (2012). Mechanical properties of kenaf/fibre glass polyester hybrid composite. *Procedia Engineering*, 41, 1654–9.
- Ghasemi, I. & Kord, B., (2009). “Long-term water absorption behaviour of polypropylene/wood flour/organoclay hybrid nanocomposite”. *Iranian Polymer Journal*, Vol. 18-9, 683-691
- George J, Sreekala M.S, Thomas S, (2004). A review on interface modification and characterization of natural fiber reinforced plastic composites. *Polymer Engineering & Science*, 41 (9): 1471-1485.
- Han G., Lei Y., Wu Q., Kojima & Suzuki S. (2008). Bamboo – Fiber filled high density polyethylene composite. Effect of coupling treatment and nanoclay. *Journal of Polymer Environment*, 10, 1007/s10924-008-0094-7
- Herrera-Franc P, Aguilar-Vega M. (1997) Effect of fibre treatment on the mechanical properties of LDPE–henequen cellulosic fibre composites. *Journal Appl Polym Sci* ;10:197–207.
- Ishak, M. R., Leman, Z., Sapuan, S. M., Edeerozey, A. M. M., & Othman I. S., (2010). “Mechanical Properties of Kenaf Bast and Core Fibre Reinforced Unsaturated Polyester Composites”. *Materials Science and Engineering*, Vol. 11, 012-006
- Jacob, M., Thomas, S., & Varughese, K.T. (2007). Biodegradability and aging studies of hybrid biofibre reinforced natural rubber biocomposites. *Journal Biomaterial and Bioenergy*, 1, 118–126.
- Joseph P.V, Mathew G, Joseph K, et al., (2003). Dynamic mechanical properties of short sisal fiber reinforced polypropylene composites. *Composites Part A*, 34:275-290.
- Jonoobi, M.; Harun, J.; Tahir, P.; Zaini, L.; Saiful, A.S.; Makinejad, M. (2010) Characteristics of nanofibers extracted from kenaf core. *BioResources*, 5, 2556–2566.
- Jawaid, M. & Abdul Khalil H.P.S. (2011). Cellulosic/synthetic fibre reinforced polymer hybrid composites. *Carbonic Polymer*, 86, 1-18.
- Kabir, M., Wang, H., Lau, K.T., & Cardona, F. (2012). Chemical treatments on plant-based natural fibre reinforced polymer composites, *Composite*, 43, 2883-2892.

- Kumar V, Sharma N.K and Kumar R. (2013). Dielectric, mechanical, and thermal properties of bamboopolylactic acid bionanocomposites. *J Reinf Plast Compos*, 32(1): 42-51.
- Ku, H., Wang, H., Pattarachaiyakoop, N., & Trada, M. (2011). A review on the tensile properties of natural fibre reinforced polymer composites. *Composite Part B Engineering*, 42, 856–73.
- Kwon, H-J., Sunthornvarabhas, J., Park, J-W., Lee, J-H., Kim, H-J., & Piyachomkwan, K. (2014). Tensile properties of kenaf fibre and corn husk flour reinforced poly(lactic acid) hybrid bio-composites: role of aspect ratio of natural fibres. *Composite Part B Engineering*, 56, 232–7.
- Kim, J.-K., Baillie, C., & Mai, Y.-W. (1992). “Interfacial debonding and fibre pull-out stresses,” *Journal of Materials Science*, vol. 27, 3143–3154.
- Krishnaprasad, R., Veena, N.R., Hanna, J., Maria, Rathish Rajan, Mikael Skrifvars & Kuruvilla, Joseph. (2009). Mechanical and thermal properties of bamboo microfibril reinforced polyhydroxybutyrate biocomposites. *Journal of Polymer Environment*, 17, 109-114.
- Leman, Z., Sapuan, S.M., Saifol, A.M., Maleque, M.A., & Ahmad, M. M. H. M. (2008). “Moisture absorption behavior of sugar palm fibre reinforced epoxy composites,” *Materials and Design*, vol. 29, 1666–1670
- Mahjoub, R., Yatim, JM., Mohd Sam, AR., & Hashemi, SH. (2014). Tensile properties of kenaf fibre due to various conditions of chemical fibre surface modifications. *Construction Building Material*, 55, 103–13.
- Meon, MS., Othman, MF., Husain, H., Remeli, MF., & Syawal, MSM. (2012). Improving tensile properties of kenaf fibres treated with sodium hydroxide. *Procedia Engineering*, 41, 1587–92.
- Mutasher, SA., Poh, A., Than, AM., & Law, J. (2011). The effect of alkali treatment mechanical properties of kenaf fibre epoxy composite. *Key Engineering Material*, 6, 471– 472.
- Nishino, T., Hirao, K., Kotera, M., Nakamae, K., & Inagaki, H. (2003). Kenaf reinforced biodegradable composite. *Composite Sciences Technology*, 63, 1281–6.
- Nishino, T., Hirao, K., Kotera, M., Nakamae, K., & Inagaki, H. (2003). “Kenaf reinforced biodegradable composite,” *Composites Science and Technology*, vol. 63, 1281–1286.
- Nair K, Thomas S, Groeninckx G.(2001). Thermal and dynamic mechanical analysis of polystyrene composites reinforced with short sisal fibres. *Comp Sci Technol* ;61:2519–29.

- Nirmal, U., Hashim, J., Lau, S. T. W., My, Y., & Yousif, B. F. (2012). "Betelnut fibres as an alternative to glass fibres to reinforce thermoset composites: a comparative study," *Textile Research Journal*, vol. 82, 1107–1120.
- Nirmal, U., Yousif, B. F., Rilling, D., & Brevern P. V. (2010). "Effect of betelnut fibres treatment and contact conditions on adhesive wear and frictional performance of polyester composites," *Wear*, vol. 268, 1354–1370
- Okubo, K., Fujii, T., & Yamamoto, Y. (2004). Development of bamboo – based polymer composites and their mechanical properties. *Composites Part B*, 35, 337 – 383
- Pothan, L. A., Oommen, Z., & Thomas S. (2003). "Dynamic mechanical analysis of banana fibre reinforced polyester composites," *Composites Science and Technology*, vol. 63, 283–293
- Panthapulakkal, S., & Sain, M. (2007). "Studies on the Water Absorption Properties of Short Hemp –Class Fibre Hybrid Polypropylene Composites". *Journal of Composite Materials*, Vol. 41, 1871 – 1883
- Ren, X., Qiu, R., Fifield, LS., Simmons, KL., & Li, K. (2012). Effects of surface treatments on mechanical properties and water resistance of kenaf fibre-reinforced unsaturated polyester composites. *Journal Adhesion Science Technology*, 26, 2277–89.
- Saheb, D.N., & Jog J.P. (1999). Natural fibre polymer composites. *Advance Polymer Technologies* 18 ,351-363
- Salleh, FM., Hassan, A., Yahya, R., & Azzahari, AD. (2014). Effects of extrusion temperature on the rheological, dynamic mechanical and tensile properties of kenaf fibre/ HDPE composites. *Composite Part B Engineering*, 58, 259–66.
- Salleh, Z., Taib, YM., Hyie, KM., Mihat, M., Berhan, MN., & Ghani, MAA. (2012). Fracture toughness investigation on long kenaf/woven glass hybrid composite due to water absorption effect. *Procedia Engineering*, 41, 1667–73.
- Saha A, Das S, Bhatta D, Mitra B.(1999) Study of jute fibre reinforced polyester composites by dynamic mechanical analysis. *J Appl Polym Sci* ;71:1505–13.
- Sun, R.C.; Sun, X.F.; Fowler, P.; Tomkinson, J.(2002) Structural and physico-chemical characterization of lignins solubilized during alkaline peroxide treatment of barley straw. *Eur. Polym. J.*, 38, 1399–1407.
- Shibata, Shinichi, & Yong Cao, IF. (2006). Lightweight laminate composites made from kenaf and polypropylene fibres. *Polymer Testing*, 25, 142–8.

- Sukyai, P., Sriroth, K., Lee, B-H., & Kim, HJ. (2012). The effect of bacterial cellulose on the mechanical and thermal expansion properties of kenaf/polylactic acid composites. *Application Mechanical Material*, 117–119, 1343–51.
- Sgriccia, N., Hawley, M.C., & Misra, M. (2008). “Characterization of natural fibre surfaces and natural fibre composites,” *Composites Part A: Applied Science and Manufacturing*, vol. 39, 1632–1637
- Shenoy, M.A. & Melo, D. J. D. (2007). “Evaluation of mechanical properties of unsaturated polyester/gum/hydroxypropyl guar gum composites”. *EXPRESS Polymer Letters*, Vol. 1-9, 622- 628
- Thiruchitrambalam, M., Alavudeen, A., Athijayamani, A., Venkateshwaran, N., & Elaya Perumal, A. (2009) Improving mechanical properties of banana/kenaf polyester hybrid composites using sodium lauryl sulfate treatment. *Material Physic Mechanical*, 8, 165–73.
- Tajvidi, M. & Takemura, A., (2010). “Recycle natural fibre polypropylene composites: water absorption/desorption kinetics and dimensional stability”. *Polymer Environment*, Vol. 18, 500-509
- Thwe, M.M. & Liao, K. (2003). Durability of Bamboo-Glass Fiber Reinforced Polymer Matrix Hybrid Composites. *Composites Science and Technology*, 63, 375 – 387
- Yakubu, Dan-Mallam, Mohamad Zaki Abdullah, & Puteri Sri Melor Megat Yusoff. (2014). The effect of hybridization on mechanical properties of woven kenaf fibre reinforced polyoxymethylene composite. *Polymer Composite*, 8.
- Yousif, BF., Shalwan, A., Chin, CW., & Ming, KC. (2012). Flexural properties of treated and untreated kenaf/epoxy composites. *Material Design*, 40, 378–85.