



**EFFECT OF CHEMICAL TREATMENT ON  
MECHANICAL AND WATER ABSORPTION  
PROPERTIES OF KENAF-GLASS FIBRE MAT  
REINFORCED UNSATURATED POLYESTER  
HYBRID COMPOSITES**

by

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A thesis submitted in fulfilment of the requirements for the degree of  
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# **EFFECT OF CHEMICAL TREATMENT ON MECHANICAL AND WATER ABSORPTION PROPERTIES OF KENAF-GLASS FIBRE MAT REINFORCED UNSATURATED POLYESTER COMPOSITES**

## **Abstract**

In this study, hybrid composites of kenaf fiber mat (KFM) and glass fibre mat (GFM) reinforced unsaturated polyester (UPE) composites were prepared by using hand lay-up technique and continue with compression moulding. The KFM was chemically treated with hydrogen peroxide ( $H_2O_2$ ) at 6% in order to improve the interfacial adhesion between fibre and UPE matrix. Fourier transform infra red (FTIR) characterization was used to investigate the changes of fibers chemical constituents after chemical treatment. Mechanical and water absorption studies of untreated and peroxide treated KFM reinforced UPE-GFM composites were characterized by flexural properties (flexural strength and modulus), water uptake and thickness swelling, respectively. The result showed that, peroxide treatment of KFM caused a significant increase in the flexural properties of UPE composites. For water absorption study, it was observed that the peroxide treated KFM showed lower water uptake and thickness swelling in comparison to those of untreated KFM filled UPE-GFM composites.

# KESAN RAWATAN KIMIA TERHADAP SIFAT MEKANIK DAN PENYERAPAN AIR BAGI GENTIAN KENAF-GENTIAN KACA TERANYAM DIPERKUKUH KOMPOSIT HIBRID POLIESTER TAK TEPU

## Abstrak

Dalam kajian ini, komposit hibrid mat gentian kenaf (KFM) dan gentian kaca tikar (GFM) diperkukuhkan poliester tak tepu (UPE) komposit telah disediakan dengan menggunakan teknik tangan dan dengan acuan mampatan. KFM telah dirawat secara kimia dengan hidrogen peroksida ( $H_2O_2$ ) pada kadar 6% untuk meningkatkan lekatan antara muka antara serat dan UPE matriks. Pencirian Fourier infra merah (FTIR) digunakan untuk mengkaji perubahan struktur kimia bahan gentian selepas rawatan kimia. Kajian sifat mekanik dan penyerapan air bagi sampel KFM diperkukuhkan komposit UPE-GFM sebelum dan selepas rawatan peroksida masing-masing dicirikan oleh sifat-sifat lenturan (kekuatan dan modulus lenturan), pengambilan air dan pembengkakan ketebalan, Hasilnya menunjukkan bahawa, rawatan peroksida keatas KFM menyebabkan peningkatan yang ketara dalam sifat-sifat lenturan komposit UPE. Untuk kajian penyerapan air, telah diperhatikan bahawa KFM yang dirawat dengan peroksida menunjukkan pengambilan air dan ketebalan bengkak yang lebih rendah berbanding dengan sampel yang tidak dirawat.

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## LIST OF ABBREVIATION

E-GF	E- glass fibre
GFM	Glass fibre mat
KFM	Kenaf fibre mat
LICA	Neopentyl (diallyl)oxy, tri (dioctyl) phosphate titanate
MEKF	Methyl ethyl ketone peroxide
PMC	Polymer matrix composite
UK	Untreated kenaf fibre
PK	Hydrogen peroxide treatment
NFPC	Natural fibre polymer composites

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background of Study

The demand for green product in the field of composite was increased due to confine the usage space of synthetic materials in the engineering applications. A huge changed over on the usage of natural fibres all over the world was reported for the production of composites (Alavudeen et al., 2014). Natural fibres like sisal, kenaf, jute, banana, coir have been used as reinforcement materials for constructive parts in aerospace, automobile and other areas. It is also for ordinary application like consumer goods, furniture, low-cost housing, and decking, railing and civil structures. The new materials with outstanding mechanical properties can get through the combination of both the fibre and matrix (Al-Bahadly, 2013).

Natural fibres can be defined as substances that produced by plants and animals that can be spin into filament, threat or rope. Low density, low cost, specific strength, economic viability, environmental friendly, biodegradability, renewability, abundantly available and enhanced energy recovery are the good qualities for natural plant fibres (Chen et al., 2013; Li et al., 2007). Natural fibres also gain the interest in the world for fabrication of composite application. Bast fibres such as jute, flax, hemp, ramie or kenaf are suitable for replacement of inorganic fibres for reinforcing polymer materials (Chen et al., 2013).

The main drawback of natural plant fibres for composites consists of the incompatibility between the hygroscopic natural fibres and the hydrophobic polymeric matrices. To improve the interfacial adhesion between natural fibre and

polymers, there are several methods that have been reported such as silane, maleated polypropylene/polyethylene, peroxide, neopentyl(diallyl)oxytri(dioctyl) phosphate titanate (LICA), sodium hydroxide (NaOH) (Chen et al., 2013; Li et al., 2007)

Hybrid is a concept that presence of two or more reinforcement that filled materials in a single matrix. Hybrid composite have behaviour that weighed sum of the individual components (Othman, 2014). Hybrid composites have unique features that can be used to meet various design requirements in a more economical way than conventional composites. This behaviour gives more beneficial balance between the advantages and disadvantages of natural fibres. Hybrid composites have proven to make a balance effect within the fibres to include in the composite materials. For the replacement of high price and non-renewable synthetic fibre, the combination of renewable and synthetic materials appear to be the magnificent structural materials which come from natural fibre that is have reasonable chance and abundant reinforcement (Atiqah et al., 2014).

The idea of new composites can be use through the combination of different types of material properties attributed to increment in demand for unique materials (Othman, 2014). A good approach can be achieve through the hybridization of natural fibre especially kenaf and synthetic fibres (Atiqah et al., 2014). Hybridization can improve the mechanical properties and moisture resistance of the composites.

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## 1.2 Problem Statement

Natural fibres have their own disadvantages. The drawback of natural fibres are their high moisture absorption, low dimensional stability and poor wettability, low thermal stability during processing and low strength and stiffness compared to synthetic fibres (Sapuan et al., 2016). To overcome this problem, the natural fibres will be combined together with synthetic fibres as hybrid reinforcement. Through this combination, the weaknesses of natural fibres could be supported by synthetic fibre.

The interfacial adhesion between fibre-matrix plays an important role in this research. To improve the interfacial adhesion between fibre surface and polymer matrix, chemical treatment can be applied as it can increase the fibre strength and modify the fibre surface. As a result, the water absorption of composite could be reduced and mechanical properties could be enhanced.

## 1.3 Objectives

1. To fabricate kenaf-glass fibre mat (KFM-GFM) reinforced unsaturated polyester (UPE) hybrid composites using compression moulding.
2. To improve the kenaf-glass fibres interfacial adhesion by using commercially available chemical treatment
3. To study the effectiveness of chemical treatment method in enhancing the mechanical and water absorption properties of KFM-GFM filled UPE composites.

#### **1.4 Expected Outcomes**

From this research, the use of hybrid reinforcement is expected to produce more beneficial balance properties between the advantages and disadvantages of natural and synthetic fibres in UPE matrix. For example, the use of GFM partly replacing half of KFM is expected to improve strength and stiffness properties of the composites. Moreover, the use of chemical treatment is expected to further enhance the mechanical and water absorption properties of the composites.

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Composites

A composite is a material that produced by the combination of two or more materials. Composite can be formed by two basic materials which are reinforcement components and matrix or binder. The matrix or binder can be act as load transfer and the reinforcing element functioned as load carrier (Othman, 2014; Sanjay et al., 2015).

The form of particle, fibre and structural are the categories of the reinforcement material that spread in the matrix phase. Polymer matrix composite, ceramic matrix composite and metal matrix composite is involved in common engineered composite materials. The good physical, chemical, mechanical and thermal properties of composite materials will be produced through the different combination in component properties.

In numerous applications fibre reinforced polymer matrix had been usually used because of the superior advantages of natural fibre over synthetic fibres and good properties. It is also has low weight, low lost, less damage in fabrication equipment and good in mechanical properties.

## **2.2 Hybrid Composites**

The word 'hybrid' is arise from Greek-Latin and can be found in different scientific scopes. In the case of polymer composites, hybrid composites are these approach in which one type of reinforcing material is embraced in a mixture of distinct matrices (blends) or two or more reinforcing and filling materials are existing in a single matrix or both approaches are integrated (Othman, 2014).

### **2.2.1 Types of Hybrid Composites**

There are respective types of hybrid composites distinguish as interplay or tow-by-tow, in which tows of the two or more components types of fibre are combining together in regular or nonspecific manner. Core-shell, in which one material is sandwiched in the middle of two layers of another, also familiar as sandwich hybrid. Then, the alternate sheets of the two or materials are stacked in the orderly manner are known as laminated. The constituent fibres are made to mix as undirected as possible are known as firm mixed hybrids, in which no over-concentration of any one type is exist in the material. Lastly, additional kinds, like those reinforced with ribs, pultruded wires, thin veils of fibre or amalgamation of the above (John et al., 2009).



## 2.3 Matrix

The matrix is a major portion of a fibre- reinforced composite. It provides a barrier opposed to adverse environments, keep the surface of the fibres from mechanical corrosion and it transfers load to fibres through shear stresses at the interface (Aziz, 2011). There are two types of matrix that is thermoset and thermoplastics (Al-Bahadly, 2013; Pickering et al., 2016). The matrix has an amount of critical purpose. It can attach the reinforcement together, maintain the form of a component and transfers the applied load to the reinforcing fibres. It also can secure the reinforcing fibres form degradation caused by abrasion or environmental attack. Matrix can contributes remarkable to the mechanical properties of structural polymer composites, acting to withstand delamination between plies of reinforcement and to retard fibre buckling during compression (Davallo et al., 2010).

### 2.3.1 Thermoset

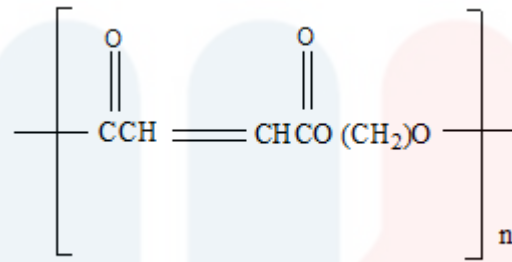
Thermoset resin is clarify as a plastic material which is originally a liquid monomer or a pre-polymer, which cured by either application of heat or catalyst to became an infusible and indissoluble material. The covalent bond linking is having in thermosets polymers in three dimensions (3D). These links prohibit the chains from sliding past one another cause in a higher modulus and enhance creep resistance. Through a curing process, the liquid resin is changed to a hard rigid solid by chemical cross-linking which involves the application of heat and the incorporation of curing agents or hardeners. Once cured, a compact bound 3D network structure is fabricated in the resin and thus the resin cannot be melted, reshaped or reprocessed

by heating. The impregnation process was followed by the shaping and solidification during the composite production.

Brittle at room temperature and have low fracture toughness are the drawback of the thermoset resin. On the other hand, the advantages of thermoset resin are high thermal stability, chemical resistance, high dimensional stability and also high creep properties. Unsaturated polyesters, epoxies, vinyl-esters and phenolic are the most common thermosetting resin that used in the composite fabrication. Generally, the thermoset are more fragile than the thermoplastic (Al-Bahadly, 2013).

### **2.3.2 Unsaturated Polyester Resin**

Unsaturated polyester resin is the condensation products of unsaturated acids or anhydrides and diols with/without diacids. It will provide a site for subsequent cross-linking through the unsaturation present in this type of polyesters. By using free radical initiators, these resins are compounded with varied fillers, reinforcements and cured to yield thermoset articles having a broad range of chemical and mechanical properties depending upon the option of diacids, diols, cross-linking agents, initiators and other additives. As an important matrix material, the versatility in the properties of the final thermoset product associated with comparatively low cost has renewed the interest in these resins for wide range of application (Dholakiya, 2012). The dimensional stability and affordable cost are the advantages of the UPE. Other benefits include ease in handling, processing and fabricating. High corrosion resistant and fire retardants are the some of the special formulation of UPE. The balance between performance and structural capabilities are the best value of this resin (Bagherpour, 2012).



**Figure 2.1:** Chemical structure of UPE

#### 2.4 Natural Fibres as Reinforcement Materials

Natural fibres are the fibres that are not synthetic or manmade. Plants or animals are the sources of the natural fibres. The use of natural fibres from both resources, renewable and non-renewable such as oil palm, sisal, flax and jute to produce composite materials. The plant, which manufacture cellulose fibres can be categories into bast fibres( jute, flax, ramie, hemp and kenaf), seed fibres (cotton, coir and kapok), leaf fibres (sisal, pineapple and abaca), grass and reed fibres ( rice, corn and wheat) and core fibres (hemp, kenaf and jute) as well as all other kinds (wood and roots). Table 1 show the most common and commercially natural fibres in the world and world fabrication (Mohammed et al., 2015).

**Table 2.1:** World Production of Natural Fibres (Mohammed et al., 2015).

<b>Fibre source</b>	<b>World production (10<sup>3</sup> ton)</b>
<b>Bamboo</b>	30.000
<b>Sugar cane bagasse</b>	75.000
<b>Jute</b>	2300
<b>Kenaf</b>	970
<b>Flax</b>	830
<b>Grass</b>	700
<b>Sisal</b>	375
<b>Hemp</b>	214
<b>Coir</b>	100
<b>Ramie</b>	100
<b>Abaca</b>	70

#### **2.4.1 Kenaf**

Rashdi et al (2009) reported more than 75 percent of worldwide kenaf production had been a source of bast fibre in India and China. That kenaf (*Hibiscus cannabinus*) is originated in Africa. The raw of kenaf fibre can be found from outer bark which is containing a bundle of lignocellulose fibres. Lignin, cellulose and pectin are a part of component in the kenaf fibres.

Kenaf fibres also have possible as an another for partial replacement of conventional materials or synthetic fibres (Patel and Patel, 2014). Kenaf is that up to 40 % of the stalk yield usable fibres roughly double that of the jute, hemp/flax that have a beneficial qualities to form the fibre quite economical. From the seed to

height of 3.6m to 4.3m (12 ft to 14ft) in 5 to 6 months the plants can increase in size(Rashdi et al., 2009). The Figure 2.1 shows the kenaf plant.



**Figure 2.2:** Kenaf plant ([www.tiyota-boshoku.com](http://www.tiyota-boshoku.com))

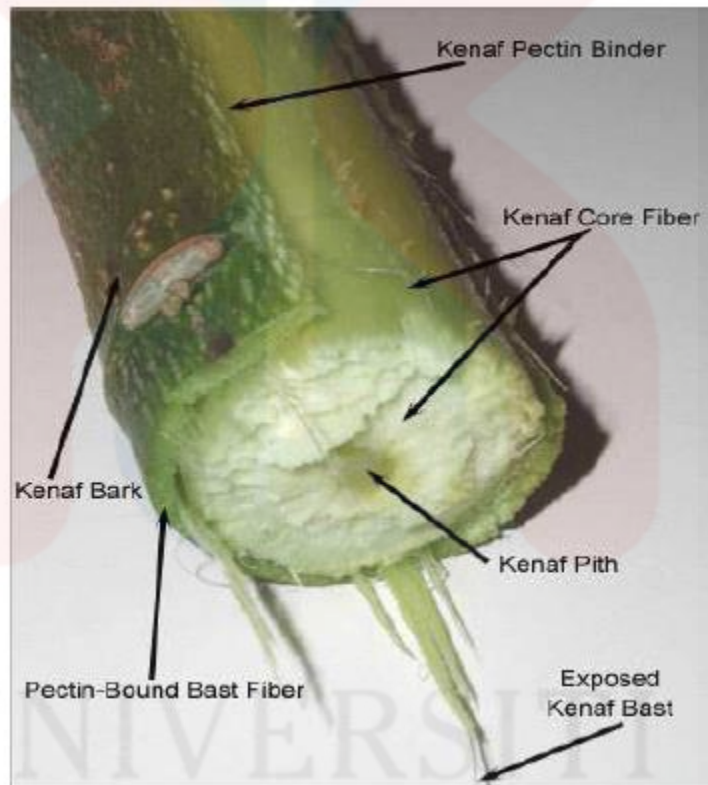
#### **2.4.2 Characterization of Kenaf**

From the outer bark of the raw kenaf fibre, content a bundle of lignocellulosic fibres. The size of the fibres bundle depends on the number of the optimum cells in each bundle that is present in the middle of the optimum cells. It is also have roughly 0.7% of cellulose, 21.6% of lignin and pectin and other components. To separate the fibres the lignin must be removed (Rashdi et al., 2009)

The stalk has three layers, a phloem that mention as the ‘bast’ is an outer cortical, the xylem is an inner woody (core) tissue layer and a thin central pith layer that made up of sponge-like tissue with mainly non-ferrous cells (Samotua et al., 2015). The kenaf fibre mat is show in Figure 2.2 and Figure 2.3 show the physical appearance of kenaf.



**Figure 2.3:** Kenaf Fibre Mat



**Figure 2.4:** Exposed physical appearance of kenaf (Samotua et al., 2015)

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## 2.5 Glass Fibre

E-glass, S-glass and C-glass are the three main categories of the glass fibres. The E-glass is classified for electrical use and the S-glass for high strength. The C-glass is uncommon for civil engineering implementation because it is for high corrosion resistance. The E-glass is the most usual reinforcement material used in civil and industrial structures from out of the three glass fibres. The glass fibre advantage and modulus can degrade with raise the temperature. It is designed to perform satisfactorily through the glass material creeps under a sustained load. The fibre itself is regarded as an isotropic material and has a beneath thermal expansion coefficient than that of steel (Bagherpour, 2012).

Due to their high specific strength and specific stiffness, glass fibres reinforced composites have become interesting structural materials not only in weight sensitive aerospace, automobile industries but also in marine, armor, railways, civil engineering structures and sports (Rao et al., 2011).

## 2.6 Chemical Treatment

The adhesion between the fibre surface and the polymer matrix can be improved by the chemical treatment of the fibre. The chemical treatment not only modifies the fibre surface but also increase the fibre strength. There are several chemical treatment including alkali, silane, acetylation, benzoylation, acrylation, meleated coupling agent, isocyanates, permanganate, peroxide, mercerization, latex coating and gamma irradiation treatment.

### 2.6.1 Peroxide Treatment

Peroxide is a specific functional group or a molecule with the functional group ROOR that containing the divalent ion that used in organic chemistry. Several organic and inorganic peroxides are useful as bleaching agents, as initiators of polymerization reactions and in the preparation of hydrogen peroxide and other oxygen compounds.

Hydrogen peroxide is a chemical compound with the formula  $H_2O_2$ . In its pure structure, hydrogen peroxide is a colourless liquid, rather more viscous than water but it's normally used as a solution for safety reasons. Hydrogen peroxide is a compound with an oxygen-oxygen single bond.

### 2.6.2 Effect of Peroxide Treatment on Mechanical Properties

Natural fibres can be handling with  $H_2O_2$  which is extensively applied in the textile production.  $H_2O_2$  can produce discolouration of fibre as an oxidizing bleaching agent. Incorporation of bleached fibre into polymeric composite can be achieved for the better physical appearance of composite. The dissociation of per hydroxyl anion ( $HOO^-$ ), equation 2.1 depends on bleaching of  $H_2O_2$  which mostly occurs under alkaline conditions:



In the case of lignocellulosic fibre, the colour of the fibre is due to the lignin component. To comprise the fibre, the bleaching action takes place when the nucleophile ( $HOO^-$ ) attacks the carbonyls and conjugated carbonyl group. Fibre surface treatment can strengthen the mechanical performance of polymeric



composites as well as the improvement in the physical appearance. With only one treatment procedure, the improvement in both physical appearance and mechanical properties can be achieved (Razak et al., 2014).

## **2.7 Water Absorption**

Natural fibres work properly as reinforcement in polymers. However, the main incapability of the application of natural fibres is their susceptibility to moisture. The interface adhesion between the fibres and the polymer matrix depends on the mechanical properties of polymeric composites.

The natural fibres are abundant of cellulose, hemicellulose, lignin and pectin, all of which are hydroxyl groups. It is normally hydrophilic sources and strong polar whilst polymers show significant hydrophobicity. Therefore, there are major challenges of appropriateness between the matrix and natural fibres. Water absorption occurs and decreases gradually into the bulk of the matrix at the composite materials' outer layers. An increased weight of wet profiles, a conceivable decline in their strength, and increment in their deflection, swelling and causing pressure on nearby structures are the results of generally high water intake by composite materials. These can trigger warping, buckling, bigger chances of their microbial inhabitation, freeze and unfreeze induced the destruction of mechanical characteristics of composite materials (Ab Ghani and Ahmad, 2011; Mohammed et al., 2015).

Mohammed et al (2015) showed the effect of coupling agent such as maleic anhydride polyethylene and chemical treatment like bleaching, acetylation and alkali treatment on reduction moisture absorption of NFPCs. During the chemical

treatment, the surface of the fibres is cleaned to ensure there no impurities which increase the fibre surface roughness and inhibit the moisture absorption through the removal of the coat of OH group of fibre as seen in equation 2.2 below:



## CHAPTER 3

### MATERIALS AND METHODS

#### 3.1 Materials

From this study, the materials used for this study is kenaf fibre mat (KFM) in the form of sheet, glass fibre mat (GFM), unsaturated polyester resin (UPE) as a matrix, methyl ethyl ketone peroxide (MEKP) act as catalyst, cobalt naphthalene as an accelerator. Hydrogen peroxide treatment will be used for this chemical treatment. The woven E-glass fibre (E-GF) type TGFM-450 chopped strand mat supplied by the district supplier for the fabrication of hybrid composite in this experiment.

#### 3.2 Composite Preparation

There are types of samples which are treated and untreated samples with different composition. At a ratio 60:40, the unsaturated polyester resin was mixed with hardener. The composition and the designation of the hybrid composites for this investigation are shown in Table 3.1.

**Table 3.1:** Composition and designation of the hybrid formulations

Designation	Sample	Composition of hybrid formulation
Untreated	S1	UPE (100% UPE + 2% MEKP + 1% cobalt)
	S2	UPE + GF (60% UPE + 40% GF)
	S3	UPE + UK (60% UPE + 40% UK)
	S4	UPE + UK + GF (60% UPE + 20% UK + 20% GF)
Peroxide Treated	S5	UPE + PK + GF (60% UPE + 20% PK + 20% GF)

### 3.2.1 Hydrogen Peroxide Treatment of Kenaf Fibres

In peroxide treatment, kenaf fibre is coated with hydrogen peroxide in acetone solution for about 30 minutes after alkali pre-treatment. Peroxide solution concentration was 6% and saturated solution of peroxide in acetone was used.

### 3.2.2 Fabrication Method of Hybrid Composites

To perform the hybrid composite fabrication, the two thick mild steel plates each weighs 1kg was needed and the measurement of 150mm x 150mm x 5mm are used. By using carbide abrasive paper of grade 240, the mild steel plates are shined to smooth the surface thus ensuring better sample flatness upon production.

It is important to handle the mould surface with a release agent before sheet moulding process start to inhibit adhesion of the fibre composite to the mould and to ease removal of the fabricated pieces. The component may otherwise bond in eliminable to the mould create the composite to be scrapped. To avoid the above

problem, the release agent in a form of sheet (commercial name is Mylar sheet) was used to ensure the surface of the fibre composite is glassy upon curing. 0.01mm is the thickness of the sheet.

The mould will be close and at room temperature at almost 25°C, it was kept for curing and in the compression moulding machine, it will take time for 30 min at 0.5 barr pressure to minimise the voids. The composite will be dividing from the mould and the specimens were cut according to the ASTM standards.

### **3.3 Material Characterizations**

The composite materials can be characterized through the flexural testing, compression moulding, and water absorption and thickness swelling test.

#### **3.3.1 Flexural Test**

The three-point bending test method was used to determine the flexural strength and modulus of the composite by following ASTM D790 standard.

#### **3.3.2 Compression Moulding**

Compression moulding is normally applied to thermoset-based polymer material composites (PMC), which made up of reinforcement phase embedded in a polymer matrix to strengthen the polymer. Thermal and mechanical properties are the dominant in thermoset compression moulded composites (Ismail et al., 2015)

### 3.3.3 Water Absorption Test and Thickness Swelling

The three samples of each composite were submerging in distilled water at room temperature. After a definite period of time, the samples were removed from the water, rubbing with the clean tissue paper before the weight is calculated. The water absorption percentage was calculated by the weight disparate using the Equation 3.1.

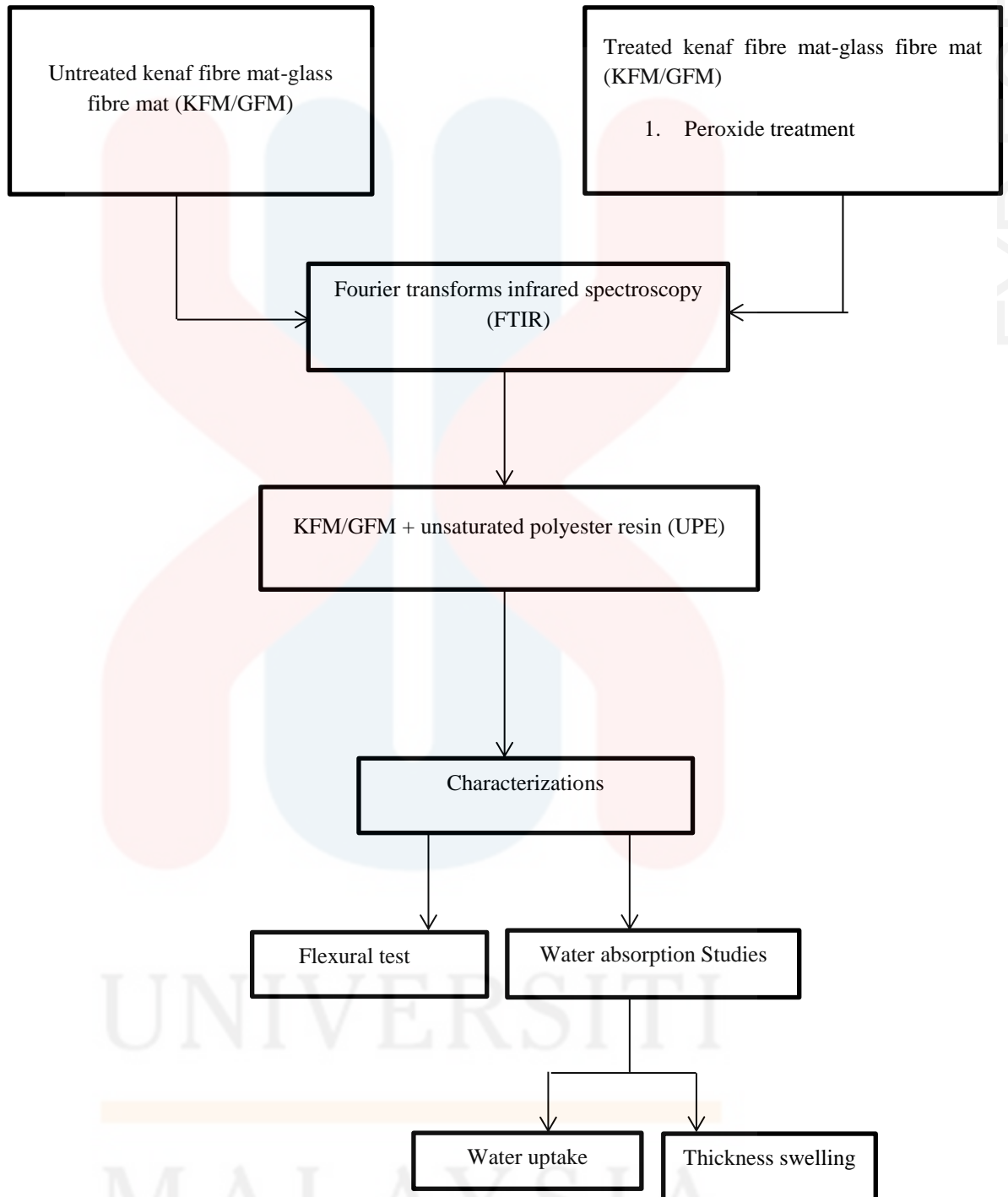
$$w_e(t) = 100 \times \left( \frac{w_t - w_o}{w_o} \right) \quad (3.1)$$

Where  $w_e$  is the relative weight change or water absorption percentage,  $w_t$  is the weight at the time,  $t$  and  $w_o$  is the initial weight at  $t=0$  and  $t$  is the soaking measure.

The thickness swelling of KFM-GFM hybrid composites is deliberated using the Equation 3.2.

$$t_e(t) = 100 \times \left( \frac{t_t - t_o}{t_o} \right) \quad (3.2)$$

Where  $t_e$  is the relative thickness percentage,  $t_t$  is the thickness at the time  $t$  and  $t_o$  is the initial thickness at  $t=0$  and  $t$  is the water immersion time (Sapuan et al., 2016).



**Figure 3.1:** Research flow chart

## CHAPTER 4

### RESULTS AND DISCUSSION

#### 4.1 Introduction

The result of the experimental work based on Chapter 3 were analysed and tabulated. All the outcomes of the experiment were discussed in this chapter. In this research, the properties of FTIR, flexural strength, flexural modulus, water absorption and thickness swelling were analysed and discussed.

#### 4.2 Functional Group Identification by FTIR

FTIR analysis was conducted to study the characterization of the functional groups of the KF before and after treated with hydrogen peroxide. Figure 4.1 showed that the broad peak at  $3328.28\text{cm}^{-1}$  and  $3280.62\text{cm}^{-1}$  attributed to hydroxyl group, H-bonded and OH stretch (Coates, 2000; Razak et al., 2014). These hydroxyl groups may be within the matching molecule (intramolecular hydrogen bonding) or they most likely exist amidst neighbouring molecules (intermolecules hydrogen bonding) (Coates, 2000; Jonoobi et al., 2010). The effect of hydrogen bonding is to trigger significant bond broadening and to lessen the mean absorption frequency. A function of the degree and strength of the hydrogen bonding will prefer the lowering of the frequency (Coates, 2000).

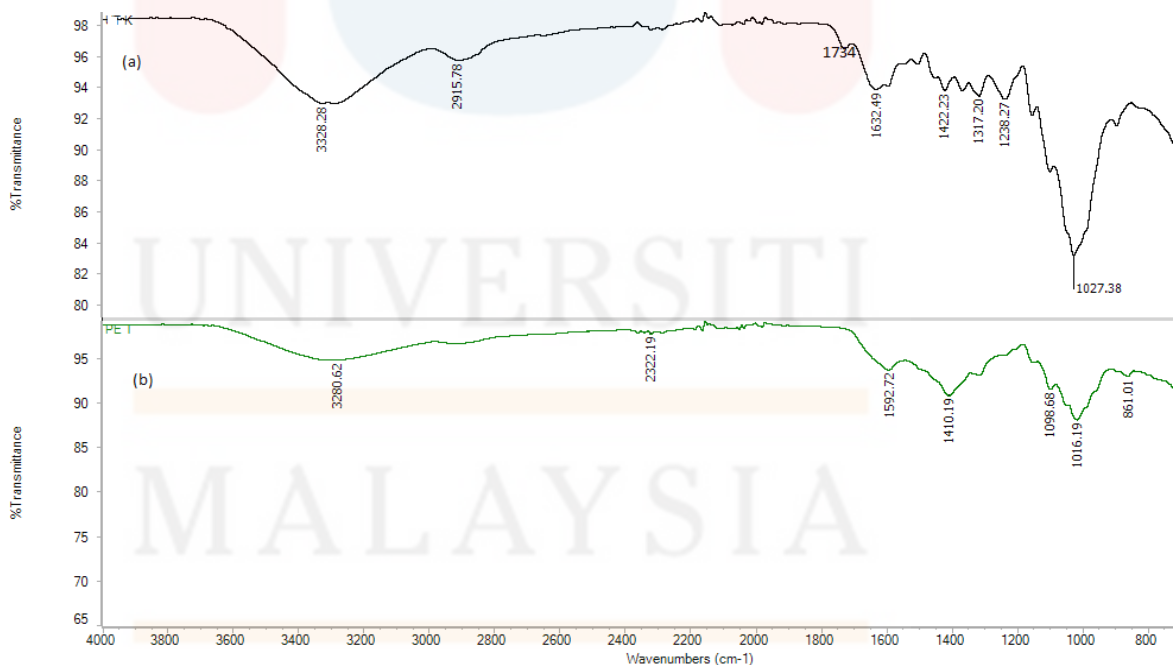
The absorption peak at  $1734\text{cm}^{-1}$  equivalent to ester carbonyl vibration which related with C=O stretching from the acetyl, feruloyl and p-coumeric acids of lignin and/or hemicelluloses (Jonoobi et al., 2010; Razak et al., 2014). After the bleaching treatment, these peak are completely vanished from the spectra as well as from those



of the nanofibers, hence suggesting the removal of lignin and most of the hemicellulose (Jonoobi et al., 2010).

A stretching peak detected at  $1632.49\text{cm}^{-1}$  for untreated kenaf fibre is attributed to carbonyl group of the acetyl ester in hemicellulose and the carbonyl aldehyde in lignin (Razak et al., 2014). After the hydrogen peroxide treatment took place, the peak disappeared due to the removal of lignin and hemicellulose (Jonoobi et al., 2010; Razak et al., 2014).

The peaks detected in the spectra of the fibres around  $1238.27\text{cm}^{-1}$  is due to C–O stretching of the aryl group in lignin. Through the chemical treatment, the disappearance of this peak from the spectra of the bleached fibres as well as from those of the nanofibers could be described by the removal of the lignin (Jonoobi et al., 2010).



**Figure 4.1:** FTIR analysis spectra of (a) untreated kenaf (b) hydrogen peroxide treatment

## 4.2 Flexural Testing

Figure 4.2 illustrated the flexural strength of UPE and its composites respectively. The highest flexural strength showed that the efficiency of stress transfer from matrix to the reinforcement. Atiqah et al., (2014) stated that the flexural behaviour of treated kenaf-UPE hybrid materials was slightly improved with higher value than the untreated kenaf-UPE. This is through a combination of compaction, matrix impregnation and curing obtained with integration of fibre composites resulting the higher flexural properties of hybrid composite.

The flexural strength of composite material increases due to introduction of reinforcements. This is due to the part played by the reinforcing fibre in support load that acts transversely to their axes. Moreover, woven fibre reinforced composite samples possessed higher flexural strength than un-woven fibre reinforced (Samotua et al., 2015).

These results indicated that the stiffness of treated kenaf-UPE composite is better than untreated kenaf-UPE composite. The combination of UPE/PK/GF hybrid composite showed higher value and could even match other composites like a kenaf-glass-epoxy hybrid composite which was studied by Atiqah et al., (2014) for the approach of automotive bumper. It can be observed that composite reinforced with treated kenaf fibre UPE/PK/GF (S5) hybrid composite has higher flexural strength compared with untreated kenaf fibre UPE/UK/GF (S4). The yarn geometry and fibre mat orientation is mostly depends on the properties of hybrid composite which have significant microstructural features (Atiqah et al., 2014). Nevertheless, in this investigation, 60% UPE matrix with 40% reinforced of kenaf-glass were compressed

through compression moulding and therefore the fibres were randomly aligned. These alignments also strengthen the flexural properties of the hybrid composite.

The UPE/GF (S2) has the higher value than the UPE/UK (S3). This is because GF more stiff and stronger than the UK.

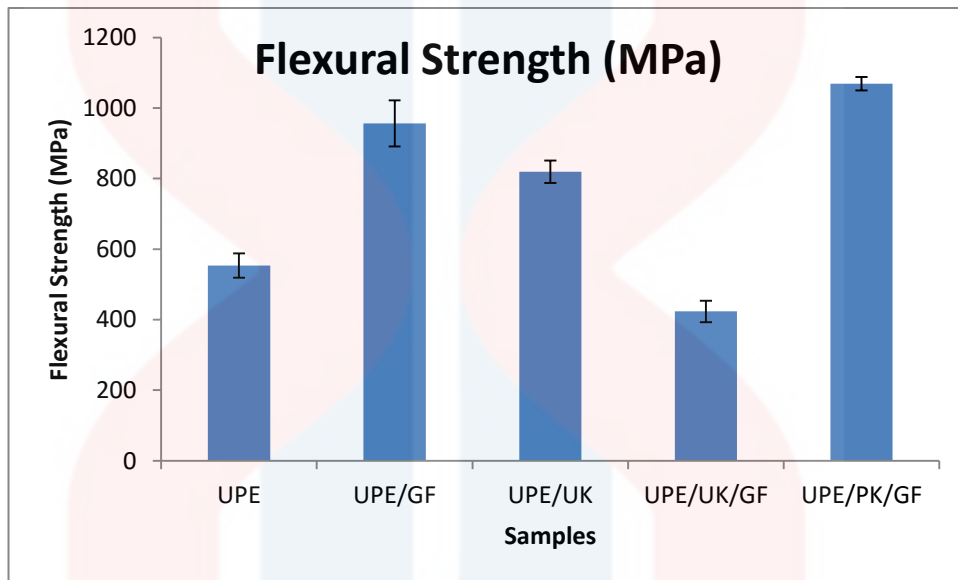
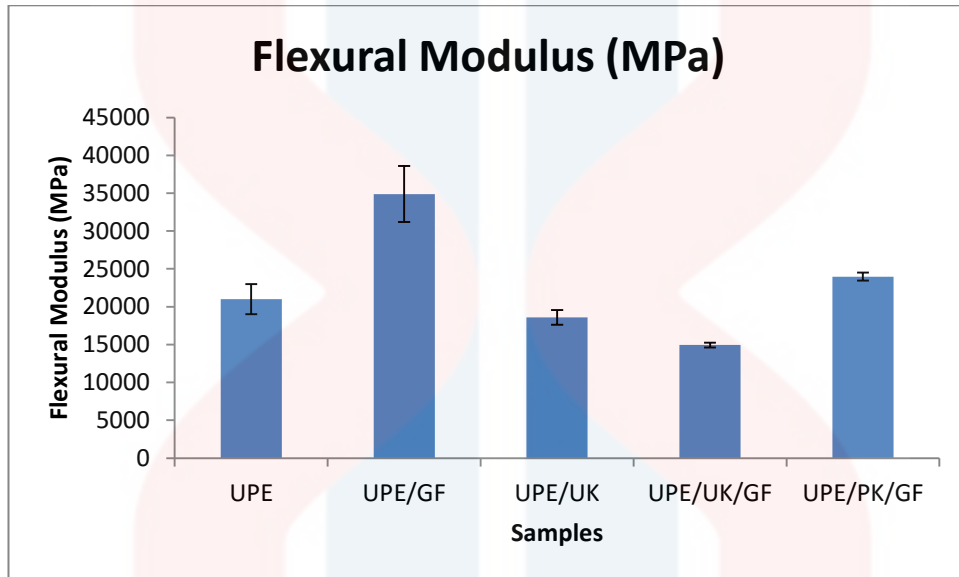


Figure 4.2: Flexural strength of UPE and its composite

According to Figure 4.3 showed that the UPE/PK/GF has higher flexural modulus than UPE/UK/GF. It is because the treated KF has a high strength than untreated KF. However, UPE/GF has highest flexural modulus because of the stiffness of glass fibre.



**Figure 4.3:** Flexural modulus of UPE and its composites

### 4.3 Water Absorption Studies

Water absorption studies can be divided into two, first is water uptake and thickness swelling.

#### 4.3.1 Water Uptake

Figure 4.4 showed the percentage of water absorption behaviour with the different composition of the composite. The curve with the different formation of UPE/GF, UPE/KF, UPE/UK/GF and UPE/PK/GF was illustrated respectively. The curve of UPE is constant and used as the comparison with other formation.

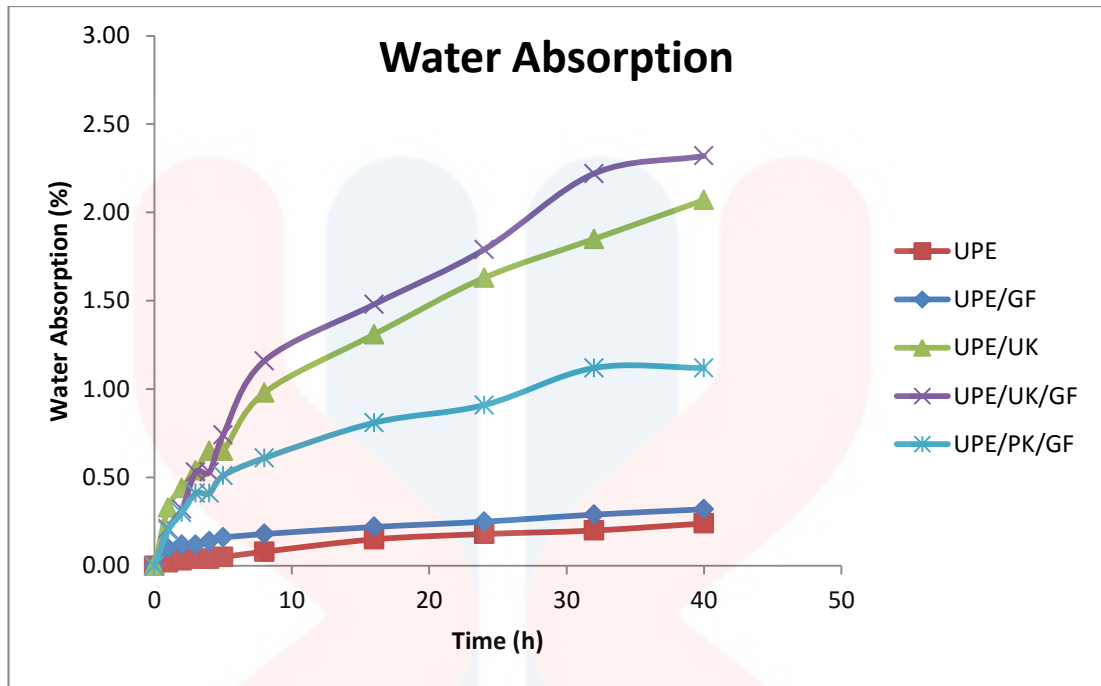
At the initial stage of absorption process, the entire composite described the water uptake increases linearly until it reached the equilibrium state. Water absorption occurs and decreases gradually into the bulk of the matrix at the composite materials surface layers (Mohammed et al., 2015).

Based on the result, the formation of UPE/UK/GF showed the highest percentage of water uptake compared to UPE/PK/GF. An increased weight of wet profiles, a conceivable decline in their strength and increase in their deflection, swelling and creating pressure on nearby structure can be determined by the high water intake of the composite materials (Mohammed et al., 2015). It is because natural fibres are connected with its hydrophilic nature, which promote the fibre to swell and ultimately rot through fungi attack. Natural fibres are hydrophilic which contain strongly polarized hydroxyl groups that can obtain from lignocellulose. These fibres, therefore, are inherently incompatible with hydrophobic thermoplastics like polyolefin. Furthermore, another problem that leads to composites with weak

interface is complication in mixing because of poor wetting of the fibre with the matrix. However, it was reported that even though the hydrophilic character of natural fibres would head to composites with weak interface but the adhesion between fibres and matrix will be improve through the pre-treatment of a natural fibres (Esther et al., 2013).

UPE/PK/GF has least water uptake than UPE/UK/GF because of the  $H_2O_2$  treatment that was applied on the KFM. Resulted to the removal of hemicellulose and lignin in the KFM. According to that, water absorption of the KFM became reduced.

Whilst, the UPE/UK has water uptake compared with UPE/GF. It is because GF is more stiff and strong than UK. As mentioned above, UK (natural fibre) is hydrophilic that have hydroxyl group. Water absorption of composites is relatively high due to the hygroscopic nature of fibre. Water absorption was supposed to be increase as the fibre content is increased (Al-Bahadly, 2013).



**Figure 4.4:** Water absorption of composites

Table 4.1 described the maximum water absorb (%) by the 5 samples in this investigation. UPE/UK/GF has higher value 2.32 % than UPE/PK/GF that has only 1.12 % of water absorption. High fibre content in the sample lead to more water penetration into the interface through the micro cracks generated by swelling of fibres causing swelling stresses which lead to composites defect.

UPE as a control sample has less value 0.24 % water absorption. It is because UPE has hydrophobic characteristic. The immersion process, changes the structure and properties of fibres, matrix and the interface between them due to the moisture uptake (Al-Bahadly, 2013).

**Table 4.1:** Maximum percentage of water absorption

Samples	Maximum Water Absorb (%)
UPE	0.24
UPE/GF	0.32
UPE/UK	2.07
UPE/UK/GF	2.32
UPE/PK/GF	1.12

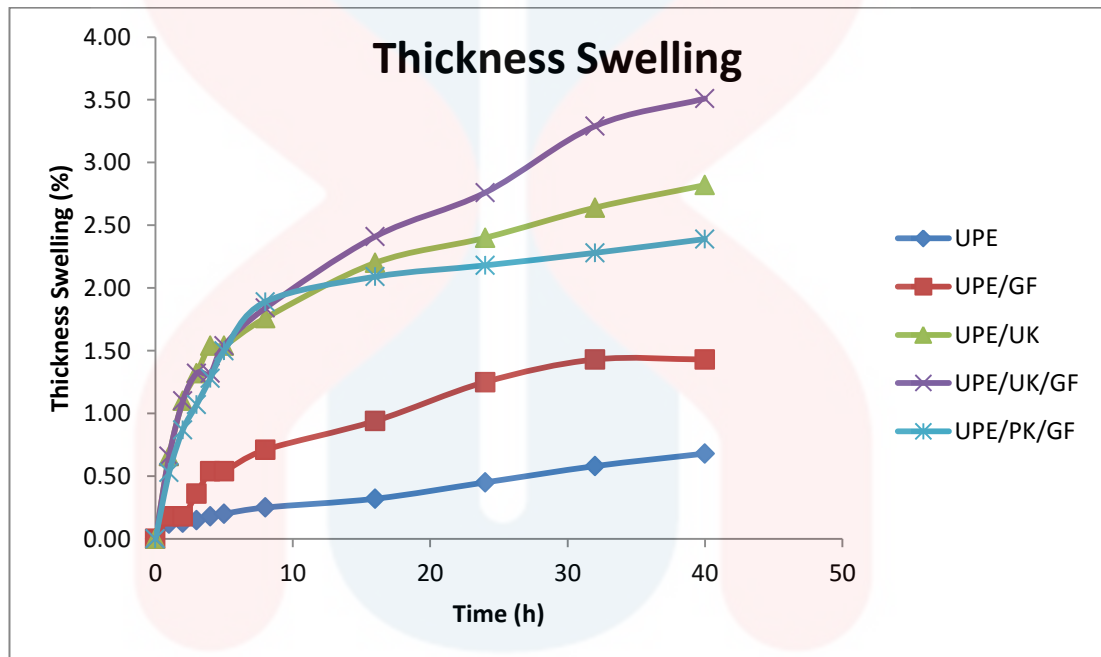
### 4.3.2 Thickness Swelling

Thickness swelling is the test where the rate percentages of thickness swell of the composites were taken. It is to determine the ability of the samples to expand in the presence of water. There was insignificant effect of thickness swelling of composites for UPE samples. The thickness swell of composites will be increase after water immersion time in the first 5 hours increased.

Depends on the results shown in the Figure 4.5 below, the UPE/UK/GF has the higher thickness swell compared to UPE/PK/GF composite. The UPE/UK/GF tends to absorb more water as the result of the thickness swelling also increases. The reduction in flexural strength is attributed to the changes appearing in the kenaf fibres and the interface between matrix and fibre as there is no reduction effect on the UPE after aging. Swelling of natural fibre as a result of extended immersion to water led to the reduction of fibres and also generated in the development of shear stress at the interface that induced de-bonding of fibre from matrix (Al-Bahadly, 2013).



However, UPE/GF has lower thickness swell than UPE/UK. Resulted that the water absorbed in the compound end up to spread through the cell wall of the sample became expand and increase the thickness. Only the natural fibre has the cell wall. The higher the fibre content, the higher the thickness swelling.



**Figure 4.5:** Thickness swelling of the composites

In addition, Table 4.2 showed the maximum percentage of thickness swelling in the specimens. As mentioned before, the UPE/UK/GF has the higher value 3.51 % of thickness swelling compared to UPE/PK/GF that has 2.39 %. It is because UPE/PK/GF does not fibre content like hemicellulose and lignin due to the chemical treatment that was applied on the KFM.

**Table 4.2:** Maximum percentage of thickness swelling

<b>Samples</b>	<b>Maximum Thickness Swelling (%)</b>
<b>UPE</b>	0.68
<b>UPE/GF</b>	1.43
<b>UPE/UK</b>	2.82
<b>UPE/UK/GF</b>	3.51
<b>UPE/PK/GF</b>	2.39

## CHAPTER 5

### CONCLUSIONS & RECOMMENDATIONS

#### 5.1 Conclusions

The unsaturated polyester resin-based hybrid composites reinforced with kenaf and glass fibre were evolved and characterised successfully. Different types of hybridization were formed which were UPE, UPE/GF, UPE/UK, UPE/UK/GF and UPE/PK/GF. The flexural strength of the kenaf-glass fibre hybrid composite for the treated 6% composition showed the highest value compared to other composites due to hydrogen peroxide process.

Furthermore, using  $H_2O_2$  treatment to improve the kenaf-glass fibres interface adhesion was successfully achieved through the composition of UPE/PK/GF hybrid composite sample. The hydrogen peroxide process enhanced adhesion between surface of the fibre and the matrix which plays an important part in improving the mechanical properties of the UPE/PK/GF hybrid composite. The properties existing for each fibre will influence the adhesion between the fibre and UPE matrix.

In addition, the effectiveness of  $H_2O_2$  treatment in enhancing the mechanical and water absorption properties of KFM-GFM filled UPE composites was improved through this investigation. It was proved by the flexural testing and water absorption studies. Flexural testing indicated the treated kenaf fibre has higher strength than untreated kenaf fibre. UPE/PK/GF showed least water uptake and thickness swelling compared to UPE/UK/GF hybrid composite. This is because of the removal of cellulose, hemicellulose and lignin in the KFM.

Addition of KF affects the water absorption behaviour of composite. The hydrophilic nature of natural fibres is responsible for water absorption. The higher the water absorbs, the higher the thickness swelling. Based on the present investigation on the developed KFM-GFM filled UPE hybrid composite, it can finally concluded UPE/PK/GF hybrid composite material is suitable for structural applications.

## 5.2 Recommendations

In future, this research should be done further with variation of characteristics used such as different length, dimensional and other characteristics. It will influence the mechanical properties of the composites. The orientation or arrangements of fibre mat also assist the composite to withstand with forces. This research also can be continued by adding materials to be hybrid with. Through the combination of this hybrid composite such as bamboo and other, hybridization of natural and synthetic fibres is very good approach to enhance the mechanical properties and water resistance of hybrid composites.

Other than that, the method also can be improved by upgrade the machines and apparatus. Later, this research can use hot compression which can spread the matrix evenly so that the result might improve and the quality might increase.

In addition, this research can be continued by improving the mechanical properties by doing some other chemical treatment on the samples. For examples, silane, mercerization and acetylation treatments. Through this investigation, the treated will show better mechanical properties than untreated fibres. It will lead the stronger bonding which might grip the fibres with UPE matrix.

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