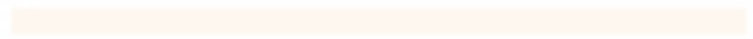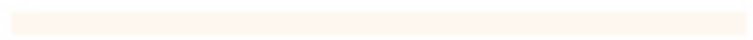# EFFECT OF CHEMICAL TREATMENT ON MECHANICAL AND PHYSICAL PROPERTIES OF NON-WOVEN KENAF FIBER MAT REINFORCED POLYPROPYLENE COMPOSITES

MUHAMMAD NAJIB IKMAL BIN MOHD SABRI
F15A0263

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FACULTY OF BIOENGIENEERING AND TECHNOLOGY
UNIVERSITI MALAYSIA KELANTAN

2019

DECLARATION

I admit that this thesis is based on my own work except quote and summary which respectively been explained their sources

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ACKNOWLEDGEMENT

Effect of Chemical Treatment on Mechanical and Physical Properties of Non-Woven Kenaf Fibre Mat reinforced Polypropylene Composites

ABSTRACT

Kenaf fibre mat (KFM) reinforced polypropylene (PP) biocomposites have been prepared using sandwich structure and hot press moulding technique. To enhance the interfacial bonding between PP and kenaf fibre, KFM was chemically treated with four different chemicals which are 5% hydrogen peroxide, 5% alkaline (sodium hydroxide), 2% silane and 5% maleated polypropylene (MAPP). Fourier transform infrared (FTIR) was used to characterize the chemical constituents in both treated and untreated kenaf fibres. Universal tensile machine (UTM) was used to determine mechanical properties (tensile and flexural strength, tensile and flexural modulus and elongation at break) of both treated and untreated KFM biocomposites. Water absorption test was also conducted in order to evaluate its water resistance performance. The result shows that, peroxide treatment demonstrated the highest performance in mechanical and water absorption properties compared to others chemical treatment. SEM morphological studies proved that almost all treated KFM showing good fibre-matrix interfacial adhesion which less fibre pull out, void and small interface gap. Thus, the chemical treatment on KFM improved fibre-matrix adhesion, which also contributed to the enhancement of mechanical properties and physical properties compared to untreated KFM.

Keywords: Chemical treatment, kenaf fibres, polypropylene, biocomposite

Kesan Rawatan Kimia terhadap Sifat Mekanik dan Fizik Biokomposit Tikar Gentian Kenaf Tidak Teranyam diperkukuh Polipropilena

ABSTRAK

Biokomposit polipropelina diperkukuh tikar gentian kenaf (KFM) telah disediakan dengan teknik struktur sandwicdan pengacuanan mampatan. Untuk meningkatkan ikatan antara muka polipropelina dan gentian kenaf, KFM telah dirawat secara kimia dengan empat bahan kimia yang berbeza iaitu 5% hidrogen peroksida, 5% alkali (natrium hidroksida), 2% silana dan 5% maleikanhidrida tercantum polipropelina (MAPP). Spestroskopi transformasi inframerah Fourier (FTIR) telah digunakan untuk mengkaji perubahan struktur atau juzuk kimia dalam kedua-dua gentian terawat dan tidak terawat. Mesin pengujianan universal (UTM) telah digunakan untuk mengkaji sifat mekanik (kekuatan tegangan dan lenturan, modulus tegangan dan lenturan serta pemanjangan takat putus) bagi kedua-dua biokomposit KFM terawat dan tidak terawat. Ujian penyerapan air turut dilaksanakan untuk menguji kadar rintangan air. Keputusan menunjukkan bahawa rawatan peroksida memberikan prestasi sifat mekanik dan penyerapan airyang terbaik berbanding rawatan kimia yang lain. Kajian morfologi SEM morfologi telah menunjukkan bahawa KFM yang terawat telah menunjukkan pelekatan gentian-matriks yang baik dengan pengurangan tarik-keluar gentian, rongga dan jurang antara muka yang kecil. Oleh itu, rawatan kimia pada KFM terbukti dapat mempertingkatkan pelekatan gentian-matriks, dan juga menyumbang kepada peningkatan sifat mekanik dan fizik dibandingkan dengan KFM tidak dirawat.

Kata kunci: Rawatan kimia, gentian kenaf, polipropilena, biokomposit

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LIST OF ABBREVIATIONS

ASTM	American Society for Testing and Materials
E_f	Flexural modulus
FTIR	Fourier Transform Infrared Spectroscopy
FE	Finite element
GPa	Gigapascal
HKFM	Hydrogen Peroxide Kenaf Fibre Mat
IR	Infrared Spectroscopy
KF	Kenaf Fibre
KFM	Kenaf Fibre Mat
LMT	Liquid Moulding Technologies
MAPP	Maleic Anhydride Polypropylene
MKFM	MAPP Kenaf Fibre Mat
MPa	Megapascal
NFPC	Natural Fibre Plastic Composite
NKFM	Sodium Hydroxide Kenaf Fibre Mat
PP	Polypropylene
RIFT	Resin Infusion under Flexible Tooling
SKFM	Silane Kenaf Fibre Mat
SEM	Scanning Electron Microscopy
UKFM	Untreated Kenaf Fibre Mat
σ_f	Flexural strength

LIST OF SYMBOLS

wt%	Weight percentage
%	Percentage
mm	Millimeter
mL	Milliliter
in	Inches
ºC	Degree celcius
h	Hour
g	Grams
kpsi	Kilo-pound per square inches
m	Weight
min	Minute
kN	Kilonewton
cm^{-1}	Reciprocal centimeter

CHAPTER 1

INTRODUCTION

1.1   BACKGROUND OF STUDY

Recently, the idea of composite materials is not a new technology thus nature is the favourite example wherein the idea of this composite materials method. There also a lot of nature that can be converted into composite material such as wood in fibrous composite and bone in collagen fibre. As the beginning of the 1960s, people can guardedly mark the provenance of the distinct discipline of composite materials after that there also has been an expanding attention for materials to are stiffer and more grounded thus far lighter in fields as incompatible as aviation, strength, as well as common development.

One of the attractive nature composite materials that can be selected and used mainly is in term of wood structure. The main role in human society activity depends on forests and the wood that been produced form them. Wood has remained a vital substance all through history on account of its one of a kind and valuable properties. Wood is recyclable, inexhaustible, and biodegradable. Numerous species are stun safe, bendable, and stable (although all wood changes measurements as it loses or picks up dampness).

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About 20 years ago, the usage of natural fibre has been a dramatic increase thus making another environment-friendly and ecological composite resources also called as "Green Composite" where structure mostly by wood fibre for example flax, cotton, kenaf, hemp, and jute. The utilization of regular strands in polymer framework composites is developing quickly to meet differing end utilizes as a part of transportation, materials, minimal effort building and other development ventures.

The material choice in kenaf (*Hibiscus cannabinus L.*) as reinforced fibre, researcher pursuit that reinforced kenaf fibre thermosets are very good to bring out two precept products which are the first is structural biocomposite such as roof structure and the another is non-structural biocomposites such as door frame (Yuhazri, Sihombing, Jeefferie, & Rassiah, 2011). The nature of natural fibre that consists of the hydrophilic component has shared higher moisture uptake of biocomposite.

1.2   PROBLEM STATEMENT

Many researches based on synthetic fibre like glass, carbon, kevlar has been utilised in polymer composites material thus synthetic fibre which is high energy consumption during manufacture and high densities properties. There are major disadvantages such as expensive in value in term of capital-intensive. The draw backs of synthetic fibre are non-biodegradable and unrecyclable although been dominate on mechanical strength. In this research, natural fibre introduced to replace synthetic fibre via biodegradable, natural fibre is derived from a renewable resource and do not have large energy requirement.

In this research, natural fibre introduced to replace synthetic fibre via biodegradable, natural fibre is derived from a renewable resource and do not have large energy requirement. Kenaf is a particularly attractive choice because of its consequent low cost and quick growth over a wide range of climatic environment. The key disadvantages of natural fibres in composites are the deprived compatibility between fibre and matrix and the relatively high humidity sorption. Previous studies (Herrera-Franco & Valadez-González, 2005; Mohanty, Misra, & Drzal, 2001) have reported that the major aspect that restraints the mechanical properties of natural fibre reinforced polypropylene as thermoplastic composites is the chemical inappropriateness between both hydrophilic lignocellulosic molecules and the hydrophobic thermoplastic molecules respectively.

Natural fibres are strongly polarized hydroxyl groups that contained in lingo-cellulose which generally hydrophilic in nature. This hydrophilic or incompatibility phenomena is infelicitous for interfacial fibre-matrix adhesion resulting in inner mechanical properties of the fibres. Hence, chemical treatments are considered in modifying the fibre surface properties meanwhile, surface adhesion of natural fibre also improvised. The research will be filled up the gap on previous research conducted by (Li, Tabil, & Panigrahi, 2007) presented a review article on the diverse chemical treatments inured to enhance the compatibility between lignocellulosic fibre and polymer matrices.

Purposely this study was conducted to make a resolution on the optimum amount of chemical that can enhance the mechanical properties of KFM by alkali, peroxide, maleic and silane treatments with 5%, 5%, 5%, ratio of (2wt%: 0.033 wt%) correspondingly.

1.3   OBJECTIVES

The objectives of the research are:

i.   To fabricate non-woven kenaf fibre mat reinforced polypropylene composites by compression molding method.

ii.   To determine the most suitable chemical treatment that can enhance mechanical and physical properties of non-woven kenaf fibre mat reinforced polypropylene composites.

1.4   SIGNIFICANCE OF STUDY

In this present study, both untreated and treated kenaf fibre mat which are sodium hydroxide, silane, hydrogen peroxide and maleic anhydride polypropylene (MAPP) are produced. In this study, kenaf fibre was chosen to reinforce by polypropylene to increase matrix fibre adhesion of biocomposite. 5%wt, 2%wt, 5%wt and 5%wt of sodium hydroxide, silane, hydrogen peroxide and maleic anhydride polypropylene (MAPP) respectively were selected in this study. A variety of chemical treatment on kenaf fibre will show diversification mechanical, chemical composition and morphological properties. Understanding the knot between untreated and chemical treated such as soaking duration and concentration also choice of natural fibres will help higher performance biocomposite function with higher service life. This study provided an important opportunity to prior the understanding of chemical treatment on fibres

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which are mostly conducted in room temperature thus contributed an increase in fibre-matrix interfacial adhesion of biocomposite than one that is untreated.

Kenaf is highly potential candidate for sustainable appliance as renewable raw material origin and cultivated widely. Large production of both conventional and hybrid vehicles are responsible for its usage. This kenaf material usually found in Malaysia, waste industry frequently used this fibre material. Introducing thermoplastic into kenaf fibre based biocomposite should make an important improvement to the field of automotive industry to decline the weight for products of NFPCs.

The results from this research will allocate an observation on treated and untreated kenaf fibre proportionately. This research will provide an incremental value on the amount of chemical portion to treat the kenaf fibre. Untreated and treated of kenaf fibre that are all reinforced with polypropylene will be examined, all of which analyses the results of tensile properties, flexural properties, failure morphology, chemical functional group and water adsorption. Data for this study were collected using softwares called OMNIC while mechanical test were carried out according to ASTM D 790 and ASTM D 3039 standards, whereas FTIR analysis is one according to ASTM E168-06 and ASTM E1252-98 standards.

CHAPTER 2

LITERATURE REVIEW

2.1   NATURAL COMPOSITE

About 100 years ago, all natural fibres such as sisal, banana, jute, oil palm, kenaf, recycled jute and coir have been used globally in matrix natural fibre-reinforced composites. The characteristic filaments are minimal effort, lightweight, high particular quality, inexhaustibility, and biodegradability and these can progressively guarantee their utilization for the creation. In some technical applications, natural fibre composites combine good mechanical properties with low specific mass (Gassan, 2002). Most naturally occurring fibers can be used as composites, yet major in applications that involve low pressure. The fibres are fundamentally comprised of an unbinding, crystalline cellulose microfibril reinforced amorphous lignin, and hemicelluloses matrix. The main constituent of any plant fiber is cellulose (Berthelot, 1999).

Natural fibres are non-abrasive towards molding also mixing equipments, which can contribute to cost reductions on equipment maintenance. Processing of natural fibres is environmental friendly where offer better working conditions hence reduced risk of respiratory problems. Currently, siren calls toward eco-friendly, natural composites are increasingly being considered as an environmentally friendly substitute for synthetic fibres on the reinforcement of polymer based composites (Sahari et al., 2012).

Moreover, they generally contain large amounts of the hydroxyl group that makes them polar and hydrophilic.

2.1.1 Polymer Composites

A pure polymer does not usually have requisite mechanical strength for application in various fields but with the support of fibre can substantially enhanced the mechanical properties where fibre provides high strength through their internal and external components in fibre which are hemicelluloses, lignocelluloses and others component. Growth trend to operation biocomposite as fillers and reinforced in plastic composites. Various studies indicated that the matrix interaction plays a vital role in important transfer between the matrix and the fibres, thereby affecting the mechanical properties of composites (del Rey et al., 2017). Attraction on their highly specific stiffness, flexibility during processing and low cost thus more than 80% increment demands for the utilization of plastics as important raw materials which are thermoplastic.

The higher content of natural fibre, the more environmental friendly is the composite. Recent years, a lot of product have build up with composite that been innovating with variety of polymer. For example, company named Milliken introduced a new line of kayaks made with Tegris, a 100% polypropylene thermoplastic composite. The material is fully recyclable and impact resistant. Impact resistance is also needed in water sports, such as jet ski, where the bottom composite panel is subject to slamming all the time. In advance, Diabhas developed a fully recyclable thermoplastic core material for use in interior panelling, floors and exterior panels for trains, trams, buses

and coaches. China Railway Long Dragon wants to refurbish interior trim components used on its national railway system in advance of the 2008 Beijing Olympics.

2.2   POLYPROPYLENE THERMOPLASTIC

The latest research and expansion results managing with natural-fibre-reinforced plastic demonstrate conceivable for partial substitute of inert fibres in interior components of cars and trucks for example linings, parcel racks, and column trim. Initially, polypropylene is an addition a lot of monomer propylene that produced in a variety of structure and can be easily reshaped to desirable shape. Unlike liquid thermosetting resins, thermoplastic polymers are in solid state of aggregation and are assimilated with the fibers in moulded state. The polymer matrix in fibre-reinforced composite material binds together with fibres, transfer applied to these fibres and conserve the fibre from adverse effect cause by the environment. The matrix also protects the fibres from any other damage when formation of shape or a profile onto composite material.

Polypropylene has been chosen as the matrix material in this study due to its good property range, low processing temperature and fairly good fibre wettability (Jayaraman, 2003). Moreover, part can be easily joined and made by heating usually with compression moulding. Next thermoplastic can be recycled or remolded if there is undesired surface or shelf-life at its limit. Lastly time length of molding cycle is very short compared to thermoset matrix. The structure is either semi-crystalline or amorphous. There are only two thermoplastics that amenable to natural fibre reinforcement which are polyethelene (PE) and polypropylene (PP).

The main obstacle in the use of plastic is poor compatibility between the fibers and the matrix and the inherent high moisture absorption, simultaneously cause dimensional changes of the fibers that guidance to micro-cracking of the composite and downgrading of mechanical properties. The process temperature must below the decomposition temperature of cellulose, which is ~$190℃$. Generally, thermoplastic processing temperature higher than thermosets.

2.3   KENAF FIBRE

Harvesting is initial step to get kenaf fibre by remove bark from the stem perpendicular retting process that occurs either by bioretting, water retting or chemical retting. Kenaf fibre is extracted from the plant *Hibiscus cannabinus sp.* (Monti, 2013) agree that kenaf has numerous uses such as fabrics and biocomposites and also an annual non-food fiber crop. Kenaf fibre also acknowledged an essential bast fibre crop with numerous industrial applications. The kenaf filaments are composites of principally cellulose, lignin and hemicelluloses. The main reinforcing element in the filaments is crystalline cellulose, while others consist of the hemicellulose and lignin can be as binding matrix (Caulfield, 2001). Kenaf fibre output is about 0.33 million worldwide per year which is estimated.

Over 30% of the stem can be used for diverse high-value fibre application in aerospace and automotive. Absence of silica content is one of advantage of the kenaf fibre which is critical in reducing abrasiveness to the processing equipment. Kenaf fibre has been found to be an important source of fibre for composites and various industrial applications. Kenaf offers many significant advantages including a short harvest period and no chlorine bleaching (Eirich, 1976).

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Kenaf fibre is lack of silica content which is crucial in reducing abrasiveness to the processing equipment (Eirich, 1976). There are three main factors of kenaf as source of fibre substance which are rapid growth, ecological flexibility and zero–waste.

2.3.1 Non-woven Kenaf Fibre

Non-woven fibre described as a sheet or web of directionally or inconstantly oriented fiber, pledged by friction, unity or adhesion, contribute economical reinforcement for composites. Non-woven gives a simple with minimal cost of natural fibre which limits the degree in optimizing the geometry and packing arrangement. This non-woven fibre laid randomly in the texture plane for most non-woven network, the in-plane properties of such non-woven composites are less anisotropic than other types of fibre composites, for example, a woven composite fibre. Broadly function of non-woven in the automotive industries, textiles and fibre board as substitute for petroleum-based plastic plus glass fiber reinforced composite. It is normally bulkier and varies in thickness with fibre type and construction. Production of kenaf polypropylene non-woven composite of fibre has not been paid much contemplation so far.

2.3.2 Woven Kenaf Fibre

Woven fibres principally produce by multiple fill and warp weaving method and usually consist of two sets of interfacing yarn to form 3-dimensional fabric structure. This structure regularly held by mechanical bound, compression stress. To construct a

nice woven, wooden frame must be used. The warp yarn is arranging on the wooden frame. Next weaving process which is weft yarn must passing over warp yarn and underneath consecutively until weaving process complete. The parameter and strength of the fibre contrast depending on the amount of warp yarn the weft yarn travel across. Function of weft yarn is to create a strong edge on the fibre or fabric during process of turning and returning back to create another row. The finite element (FE) technique has been broadly used in the foreseeing the motorized response of woven fibre composites.

2.4   COMPOSITE FABRICATION METHOD

Combination of two or more constituent materials with naturally different in physical and chemical ingredients thus the final product produces completely a different characteristics from the individual components. In the research, natural fibre or kenaf fibre build up with three major elements such as lignin, cellulose and hemicelluloses while polypropylene build up by addition a lot of monomer propylene. Widely application as in alternative composite material especially in building and construction is highly plausible with both lightweight and low cost as its main driving force (Akil et al., 2011). Using natural reinforcement in plastics is effortless but optimistic advent to advance the mechanical properties of composite materials, which had been recognized since around early on 1900s (Jawaid & Abdul Khalil, 2011). The melt polypropylene (PP) works like glue that bonds the intersected kenaf fiber (Hao, Zhao, & Chen, 2013).

2.4.1   Hot Press

Hot press or compression moulding is a technique that used to formulate kenaf fibre with polypropylene (PP). This technique is conventional technique for producing polymer mould composites. Study on kenaf-PP composites compression technique proved to have a superior tensile and flexural strength when compared to other compression molded natural fibre composites such as sisal and coir reinforced thermoplastic (Faruk, Bledzki, Fink, & Sain, 2012). Moreover, there is one more obstacles to be addressed in the invention of kenaf fibre reinforced composites regards rough fiber handling.

Compression moulding is very popular in the manufacture of natural fibre composites by reason of its high reproducibility and low cycle time. The first step is to make the material charge by sandwiching kenaf mat saturated with PP powder where the upper and bottom layer is PP. The kenaf-PP composites compression molded which utilizes a layered sifting of PP powder. The composite made by pressing the material between hot plates of a compression moulding press at 180⁰C for 2 minutes (Murali & Pannirselvam, 2011) at 10 MPa.

Experiment conducted by (Zampaloni et al., 2007) state that two different composite manufacture, one with long kenaf with approximate 130 mm (5.1 in) while the other using shorter with approximate length of 20 mm (0.79 in). Polypropylene sheets firstly are made by compression molding a polypropylene (PP) powder on a Carver Laboratory Press. A layer of 10 g of PP powder heats to $190℃$ under minimal pressure for 3 minutes. The pressure increases to 69 MPa (10 kpsi) for a period of 10 minutes, then inflation until 165.5 MPa about 5 minutes more. Before release and remove, melted PP allows to cold to $100℃$ still under pressure. After hot-pressing, the

panels were transferred to a brace of cold plates for cold-pressing for 8 minutes to get a sleek surface.

2.4.2 Injection Moulding

Thermoplastic matrix composites can also require injection moulding such as Liquid Moulding Technologies (LMT) and Resin Infusion under Flexible Tooling (RIFT) which thermoplastic systems supplied as low viscosity monomers and polymerized in-process is normally (Summerscales, Virk, & Hall, 2013). The technique can produce multiplex geometric components with functional elements fast and also in a great number. The problems encounter when using injection moulding firstly melt flow viscosity increased as sisal fibres were introduced (C & Regiment, 1999; Fung, Li, & Tjong, 2002). Secondly interfacial bonding between sisal fibre and polypropylene are low thus high injection temperature necessary to unable the high melt viscosity of the fibre and polypropylene composites. Injection moulding shows superior overall strength due to adhesion and distribution of the fibres.

In contrast, injection moulding also offers various points of advantages for example minimal wrapping and shrinkage, economics of scale and utilization of reused materials as compared to compression moulding. The moulded sisal fibre and polypropylene composites give increment to the darkening tone and odor discharge. Moreover, there are suggest a few pre-treatment techniques to beat the broke interfacial bonding between sisal fibre along with polypropylene, for example, alkaline treatment, heat treatment and conceivable arrangement is coupling agent treatment, maleic anhydride.

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As summary, compression moulding picks up as moulding technique for conduct my research. This is because kenaf and polypropylene need a superior tensile and flexural strength when compared to other compression molded natural fibre composites. In other hand, the low cycle time and high reproducibility are the positive factor to conduct experiment by using compression or hot press moulding.

2.5   UNTREATED KENAF FIBRE MAT

Side effect by untreated fibres and the polymer matrix is insufficient adhesion that can lead to debonding with age (Gassan, 2002). Raw kenaf fibres are only hand-washed to remove dirt or sand from the surface later let the surface dry. Kenaf fibre which there is no modification or alteration by any chemical factor to improve interface adhesion property between the fibres so it is in original stated or natural fibre form will continuously undergo quality analysis to determine progress or their primitive structure next fabrication or compression moulding held on fibre. Compression moulding will held in form of sandwich where untreated kenaf fibre matting of layering each other. Polypropylene that combined with MAPP will be the coverage of the kenaf fibre, upper and lower layer of sandwiched form. Before conducted SEM analysis, untreated kenaf also combined with MAPP.

2.6   TREATED KENAF FIBRE MAT

Initially, four chemical treatments would conducted in which alkaline treatment with 5% of NaOH, silane treatment with 2% concentration ratio of (95% ethanol with 5% water solution) follow by addition of acetic acid to adjust pH in range 4.5 until 5.5, maleic anhydride grafted polypropylene (MAPP) treatment about 5% and hydrogen peroxide treatment portion of 5% thus all the treatments will be organized in appropriate condition.

Alkali treatment literature from (Sreekala, Kumaran, & Thomas, 1997) reported that 5% NaOH amount is better enough as a treat for tensile strength than 10%. (Valadez-Gonzalez, Cervantes-Uc, Olayo, & Herrera-Franco, 1999) research outcome stated that silane treatment diluted in a water and ethanol mixtures with the ratio (2wt%:0.033 wt%) for 3 hours also keep at room temperature. Maleic treatment research by (Amran et al., 2014) approve that 5% of MAPP can have a better mechanical property for both tensile and modulus strength lastly research on peroxide whose conducted by (Razak, Ibrahim, Zainuddin, Rayung, & Saad, 2014) implement that this 5% hydrogen peroxide treatment caused boost in surface roughness and crystallinity index of the kenaf fibre due to the evacuation of hemicelluloses and lignin after the bleaching treatment.

The state chemical treatments are in the optimum parameter or measurement thus and so this research would identified the best chemical treatment among those four treatments. Compression moulding also would held in form of sandwich where all treated kenaf fibre matting of layering each other.

 Polypropylene that combined with MAPP would be the coverage of the kenaf fibre, upper and lower layer of sandwiched form. Briefly both untreated and treated

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KFM would check up their quality analysis on FTIR analysis lastly characterization on three factors which are morphology; SEM analysis, mechanical properties; tensile and flexural and physical properties, water absorption and thickness distension.

2.6.1 Sodium Hydroxide Treatment

Mercerization or alkaline treatment uses specifically sodium hydroxide to remove a certain amount of lignin, wax, and oils integument the external surface of the fibre cell wall as well as one of the most used chemical method. Definite amount of lignin wax and oils layering the exterior surface of the fibre cell wall remove, depolymerizes the indigenous cellulose structure and exposes tiny length crystallites affect by this treatment. Inclusion of aqueous sodium hydroxide (NaOH) to natural fibre coordinated the ionization of the hydroxyl group to the alkoxide.

$$Fibre - OH + NaOH \rightarrow Fibre - O - Na + H_2O \qquad (2.1)$$
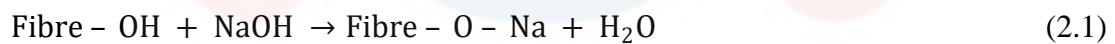
Potential chemical bonding also been exposed by cellulose on the surface of the fibre with matrix material (Li et al., 2007). The dispersal of fibre in the matrix enhances accordingly thus resulted in decline of the agglomeration of the fibre. A high alkali concentration may deployment the native cellulose and delignify the fibre extremely, which know how to worsely influence the strength of the fibre. The fluctuation of the mechanical and physical properties may affect by alkaline treatment for both surface and texture of kenaf fibre. There are several effects of alkaline treatment on the fibre. Firstly, increment surface roughness can result in surpass mechanical interlocking and

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for instance amount of cellulose gain, exposed on the fiber surface, thus increasing the total of possible reaction sites (Valadez-Gonzalez et al., 1999).

Reduction of hydrophilic hydroxyl groups affected the fibre drizzle resistance properties is increased. (Sreekala et al., 1997) reported that 5% sodium hydroxide (NaOH) amount is better enough as a treat for tensile strength than 10%. This treatment will take part in a vital role for accomplishing the ideal competence of the fibre as far as the adhesive attributes of the fibres are troubled. (Asumani, Reid, & Paskaramoorthy, 2012) conducted an experiment on concentrated NaOH from 1% to 8% in intervals of 1% by mass were used. Kenaf fibre immersed in the NaOH solution for 24 h at 45°C. Afterward the mats washed with running tap water later absorbed in distilled water containing 1% acetic acid to counterbalance the remaining NaOH molecules. These mats were then dried up in an oven at $45°C$ for a day.

As summary method for alkaline treatment (NaOH) with amount of 5% is be chosen to further on researcher cause by the material is easily can get and the optimum amount thus the achievable condition which is dry in room temperature is the way of dry the mats. If concentration of alkali is extreme can causes excess delignification of natural fiber resulting damage fibre.

2.6.2 Silane Treatment

Silane coupling agents may pony up hydrophilic properties to the interface (Faruk et al., 2012). The presence of moisture, hydrolysable alkoxy groups can form silanols consequently hydroxyl groups of fibre cell easily reacts one end of silanol while another end reacts with the matrix functional group. Some major advantages of silane as

coupling agent are economically and commercially available other than have a great number of functional groups which can be customized as a purpose of the matrix to be used.

Kenaf fibre would undergoes surface treatment of the filler carry out in solution where silane must be diluted in ethanol solution (95% ethanol with 5% water solution) pursue by addition of acetic acid to adjust pH in range 4.5 until 5.5 to yield a 2% final concentration. While (Valadez-Gonzalez et al., 1999) comes up with 1% silane and 0.5% dicumyl peroxide, weight up percentage in relation to fibre. The silane and dicumyl peroxide dissolved in a mixture (90% methanol and 10% water) for their hydrolysis.

According to (Asumani et al., 2012) 5% by of three-aminopropyltriethoxysilane (weight of silane relative to the weight of kenaf mat) is dilute in a 50% aqueous solution of methanol. The pH of the solution is maintain between 4 and 5 using acetic acid. Again the mats are immerse in the solution for 4 h at a temperature of 28°C lastly wash again with distilled water before been dried in the oven at 45°C for 12 h. Silane solutions in a water and ethanol mixture by concentration of 0.033% and 1% were also agreed by (Valadez-Gonzalez et al., 1999) to luxury henequen fibers and oil palm fibres.

The reaction schemes were given as follows (2.2) and (2.3) showed the hydrocarbon chains transferred by the application of silane hamper the swelling of the fibre by creating a crosslinked network due to covalent bonding between the matrix and the fibre. As summary method for silane with amount of 2% is be chosen to further on researcher cause by the material is easily can get and the optimum amount.

$$CH_2CHSi(OC_2H_5)_3 \xrightarrow{H_2O} CH_2CHSi(OH)_3 + 3C_2H_5OH \qquad (2.2)$$

$$CH_2CHSi(OH)_3 + Fibre-OH \rightarrow CH_2CHSi(OH)_2O-Fibre + H_2O \qquad (2.3)$$
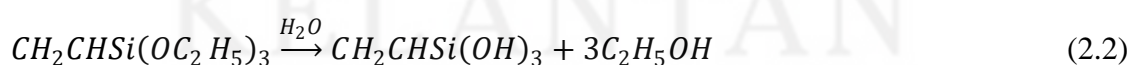
2.6.3   Hydrogen Peroxide Treatment

Organic chemistry highlights that peroxide is a specific functional group or a molecule with the functional group ROOR encompassing the divalent ion O – O. Organic peroxides lean to decompose easily to free radicals of the form RO; RO then rebounds or reacts with the hydrogen group of the matrix and cellulose fibers. Commonly natural fibre can be treated with hydrogen peroxide (H_2O_2) which is extensively utilized in textile industry.

$$RO - OR \rightarrow 2RO \tag{2.4}$$

Theoretically nucleophile (HOO^-) will attack the carbonyls and adjoin carbonyl groups that comprise the fibre. Speedy decay of a peroxide treatment yielding free radical that can react with the hydroxyl group of the fibre hence the matrix result a good fibre adhesion along the composite interphase.

As an outcome, of peroxide treatment affect the hydrophilicity of the fibres by decrease value and the tensile properties increased.

2.6.4   Maleic Anhydride grafted Polypropylene as Coupling Agent

Late 1970s, several groups of researchers cited that MAPP has the efficiency to improve the compatibility between the fibre and plastic matrix. Usage of MAPP with satisfactorily high molecular weight and anhydride content appreciably improve the fibre-matrix bonding. The interfacial adhesion between wood fibre and plastic or thermoplastic, the addition of a coupling agent is one of the ways to improvise and

enhance both mechanical and physical of fibre. Cellulose fibres that treated with hot MAPP copolymers provide covalent bonds across the interface (Figure 2.1).

MAPP categories as a familiar coupling agent for polypropylene (PP) based composites by natural fibre. Good transfer interface by entanglement chain of MAPP and possessed the highest mechanical properties. Fibre matrix bond and enhances the mechanical properties of the biocomposites noticeably better. Coupling agent for PP based composites well known as MAPP either from natural fibre (Gassan, 2002)(Sanadi, Hunt, Caulfield, Kovacsvolgyi, & Destree, 2002).

Previous research approved that 5wt% of MAPP can have a better mechanical property for both tensile and modulus strength. 5wt% MAPP yielded the optimum value for the composites in term of tensile strength and modulus. MAPP have a higher modulus or cost and a higher specific modulus if the composites being manufactured than sisal, coir, and even E-glass (Faruk et al., 2012). Significantly showed by previous research that a superior tensile strength of up to 74 MPa, achieved with the highest fibre loading of 60% by weight or 49% approximately by volume, compared to uncoupled composites and unfilled polypropylene (PP) (Sanadi et al., 2002).
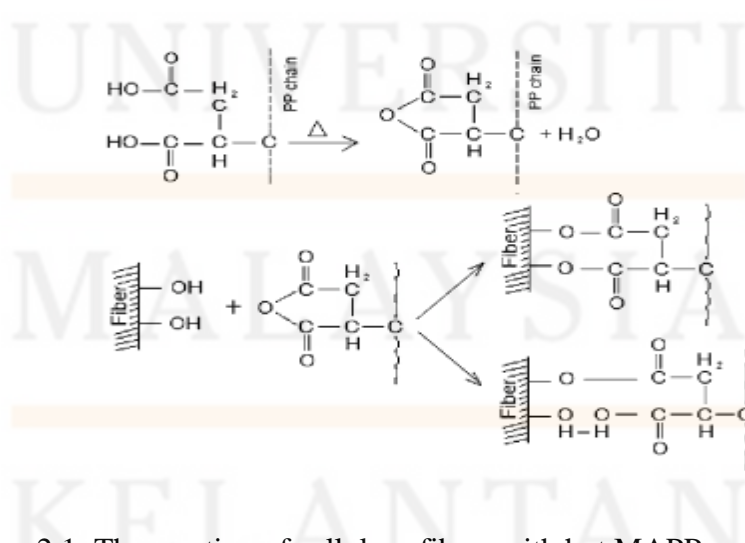
Figure 2.1: The reaction of cellulose fibres with hot MAPP copolymers

Source:(Li et al., 2007)

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CHAPTER 3

MATERIALS AND METHODS

3.1   MATERIALS

The materials that were used in this study are the kenaf fibre mat (KFM). The KFM were collected from local plantation sources which were located at Penang. The matrix selected for the study included polypropylene (PP). Four different types of chemical which were silane, maleic anhydride polypropylene (MAPP) from OVERAC CA100, sodium hydroxide and hydrogen peroxide used separately as treatment on the KFM. All measurements were taken according to the size of the KFM for five characterizations to be carried out. The mechanical properties such as strength and moduli of both flexural and tensile specimens also physical properties likes water absorption of untreated and treated composites should be pointed, respectively.

3.2   METHODS

Sample preparations were separated into two different groups: untreated and treated KFM. Four types of chemical treatments were used to treat the KFM before the composite samples were fictitious using hand lay-up and hot press technique.

3.2.1 Fibre Treatment

Dried kenaf fibre mat (KFM) were soaked in three different type of chemical which were sodium hydroxide, hydrogen peroxide and silane solution. First dried KFM was soaked in 250mL of diluted water and 5wt% NaOH solution while second dried KFM was soaked in 250mL of diluted water, silane and ethanol mixture within ratio of (2wt%: 0.033wt%). Both NKFM and SKFM were using cleaned basin with duration of 3 hours at room temperature. NKFM and SKFM kept overnight dried in room temperature after treatment. Third, dried KFM was soaked in 5wt% hydrogen peroxide (H_2O_2) with the help of water bathto maintain temperature range of 80ºC. Initial and final pH for the treated HKFM was recorded in Table 3.1. HKFM was washed with diluted water to remove impurities before hot-oven for 2 days at 60ºC.

Table 3.1: Differences of pH value on various parameters

Parameter	pH value
Pure hydrogen peroxide (pHi)	3.8
KFM and hydrogen peroxide before heated	5.5
KFM and hydrogen peroxide after heated	5.3

3.2.2 Composite Preparation

Dried kenaf fibre mats (KFM) were cut using scissor and dimension of 150 mm long, 150 mm width and 3 mm thick were taken. The matrix–fibres were about ratio 30:70 for KFM-PP composites. To manufacture the treated and control sample of KFM were sandwiched thoroughly with polypropylene (PP) except for maleic anhydride

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polypropylene (MAPP) where it would immersed in both PP. The PP poured onto mold of 2 mm thick for the both layer. KFM was sandwiched by two sheets of PP, hot-press compression machine would pressed in the PP into KFM. For making KFM-PP composite material, the mass was easily determined by simple weighing using electronic balance.

Table 3.2: Preparation of composites for untreated and treated KFM-PP.

Sample design	Composite formulation (wt%)		Treatment (wt%)			
	PP	KFM	NaOH	Silane	MAPP	Hydrogen Peroxide
UKFM	70	30	-	-	-	-
NKFM	70	30	5	-	-	-
SKFM	70	30	-	2	-	-
MKFM	70	30	-	-	5	-
HKFM	70	30	-	-	-	5

The series of designation was depicted in Table 3.2. The sides of stainless steel mold were spread with release agent to prevent tight sticking of PP on the mold. The mold and the specimen were closed slowly, to avoid misplacement or inaccurate position thus forming a good interaction in the mold shape, and then placed into compression moulding machine. A compression moulding machine branded GT-7014-H was operated at 200°C. Steel mold was initially preheated on upper platen by released a gap between steel mold and platen steel about 8 minutes. Then, steel mold pressed for 5 minutes. Exchange position of the mold took place in separate press of the machine and cooling was done for 8 minutes under maintained pressure in room temperature.

3.3 CHARACTERIZATIONS

3.3.1 Flexural Test

Flexural test measured the force requisited to bend a beam under three-point loading conditions. The flexural test was performed in accordance to ASTM D 790 procedure, using Instron universal testing machine (Testometric M500-50T) with span – to – depth ratio of 16:1 and tested at cross head speed of 5 mm/min. Test samples in rectangular form having 127 mm x 12.7 mm x 3 mm were used for each of samples for depicted. The samples were prepared by cutting from sandwiched KFM in rectangular form using handsaw. A sample rested horizontal on a support span and the load was applied to the centre by the loading nose producing three-point bending at a detailed rate. The samples were placed onto two supports having a 50 mm span length between the supports. A load capacity of 50 kN used to implement this test and conducted in laboratory environment.

All the statistical indicative results were obtained as the arithmetic mean and the standard deviation of three test samples of treated, untreated KFM composites and pure polypropylene. Load – deflection curves determined the flexural strength and modulus. The flexural strength, σ_f and modulus, E_f were measured using the equations (3.1) and (3.2):

$$\sigma_f = (3PL)/2bd^2 \tag{3.1}$$

$$E_f = L^3m/4bd^3 \tag{3.2}$$

Where P = load applied, L = span length, d = thickness of samples, b = width of samples and m is the slope of the initial straight-line portion of the load-deflection curve.

3.3.2 Tensile Test

Tensile test engaged escalating the sample in machine and subjecting it to tension at an even rate. ASTM D 5083 referred as procedure to perform the test using Instron universal testing machine (Testometric M500-50T), tested at cross head speed of 10 mm/min, and conducted 50 kN in laboratory condition. Test samples were prepared by probing from KFM in rectangular form using handsaw. The samples which were 150 mm x 20 mm x 3 mm initially polished and cleaned well by abrasive paper so that the samples did not have bumps.

All the statistical indicative results were obtained as the arithmetic mean and the standard deviation of three samples of treated, untreated KFM composites and pure polypropylene. Tensile strength, elongation at break and tensile modulus were then recorded for all samples. The tensile strength, elongation at break and tensile modulus were measured using the equations below:

$$s = \sqrt{(\sum \bar{X}^2 - nX^2)(n-1)} \qquad (3.3)$$

s = estimated standard deviation

X = value of single observation

n = number of observations

$\bar{X}$ = arithmetic mean of the set of observations.

Figure 3.1: Fracture tensile samples

3.3.3 Fourier Transform Infrared Spectroscopy

Infrared spectra of each sample were collected in the range of 4000cm^{-1} to 500cm^{-1} using FTIR spectrophotometer which is Thermo Scientific Nicolet iN10. FTIR was operated to recognize functional groups and its molecular bond granted in KF by effect of sodium hydroxide, silane, hydrogen peroxide and MAPP. Nicolet iN10 ranged infrared spectra of each samples at room temperature. Sample with thickness 3 mm can easily operated on the operation bench of Nicolet iN10. The IR spectrum can get through the samples which obtained with laser of infrared projected.

3.3.4 Scanning Electron Microscopy

Scanning electron microscopy (SEM) conducted by Jeol JSM-IT100 provided an examination technique of surface morphology of fibres and fracture surfaces of NFPCs.

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Fibre-matrix interfacial reciprocal action that occurred after treatment of the fibre were examined. In this study, SEM was accomplished at an acceleration voltage of 3 kV and a magnification ranged from x100 to x500 was used to collect SEM images of the fracture surfaces of the tensile samples. For all tensile cleavage surface samples were fit out in dimension of 6 mm x 6 mm x 3 mm, the selected composite cleavage surface was fixed on the stub using copper tape to decrease amount of charging affect by SEM.

Figure 3.2: Sample preparation for SEM

3.3.5 Water Absorption

Concernedly composites structural applications focus on wood fibre; kenaf fibre implements on water absorption behaviour of the composite. Water absorption effect on behaviour of kenaf fibre will reach equilibrium at a certain specific immersion time. The samples must be in standard form and measurement which is flexural test mould of mass and dimension then were weighed regularly at 1, 2, 3, 24, 48 up to 72 h would took in water respectively. Similarly referred to Figure 3.3, the samples were immersed in water at room temperature furthermore the samples were positioned inside a container of distilled water (pH 7). The samples would weighted every 1, 2, 3, 24, 48 up to 72 h by removed water on the surface with a clean dry cloth continuously. The

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research flowchart of the preparation and characterization of KFM-PP biocomposite was presented in Figure 3.3

Figure 3.3: Sample immersion in distilled water

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                    ┌─────────────────────┐
                    │   Kenaf fibre mat   │
                    │        (KFM)        │
                    └─────────────────────┘

         Randomly obtained from kenaf plantation

              Cut into required dimension

                    ┌─────────────────────┐
                    │    Untreated KFM    │
                    └─────────────────────┘

         Undergo four different chemical treatments

   NaOH        Silane    Maleated Polypropylene    Hydrogen Peroxide
                              (MAPP)

                    ┌─────────────────────┐
                    │     Treated KFM     │
                    └─────────────────────┘

          ┌──────────────────────────────────────┐
          │ FTIR (Progress of chemical treatment) │
          └──────────────────────────────────────┘

                       Fabricating

          ┌──────────────────────────────────────┐
          │ 1. Compress polypropylene            │
          │    (PP) moulding onto KFM at         │
          │    ratio 30:70                       │
          │ 2. Temperature about 200ºC           │
          └──────────────────────────────────────┘

                    ┌─────────────────────┐
                    │  Characterizations  │
                    └─────────────────────┘
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Stage 1

Stage 2

Stage 3

| Chemical Functionology FTIR analysis | Morphological properties by Scanning Electron Microscope (SEM) | Mechanical properties testing (flexural, tensile, impact) | Physical properties testing (water absorption) |

Analysis, evaluation and comparison of all the optimum parameter for a chemical treatment that treated KFM between tensile strength and modulus, flexural strength and modulus, mass loss and reinforcement surface morphology.

Stage 1: Material preparation (Process based on experimental set up including collecting and grinding followed by chemical treatment of KFM)
Stage 2: Fabricating the KFM-PP composites (30:70)
Stage 3: Analysis, evaluation and comparison of the obtained experimental data

Figure 3.4: Research flowchart of the preparation and characterization of KFM-PP biocomposites

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CHAPTER 4

RESULTS AND DISCUSSION

4.1   FOURIER TRANSFORM INFRARED SPECTROSCOPY ANALYSIS

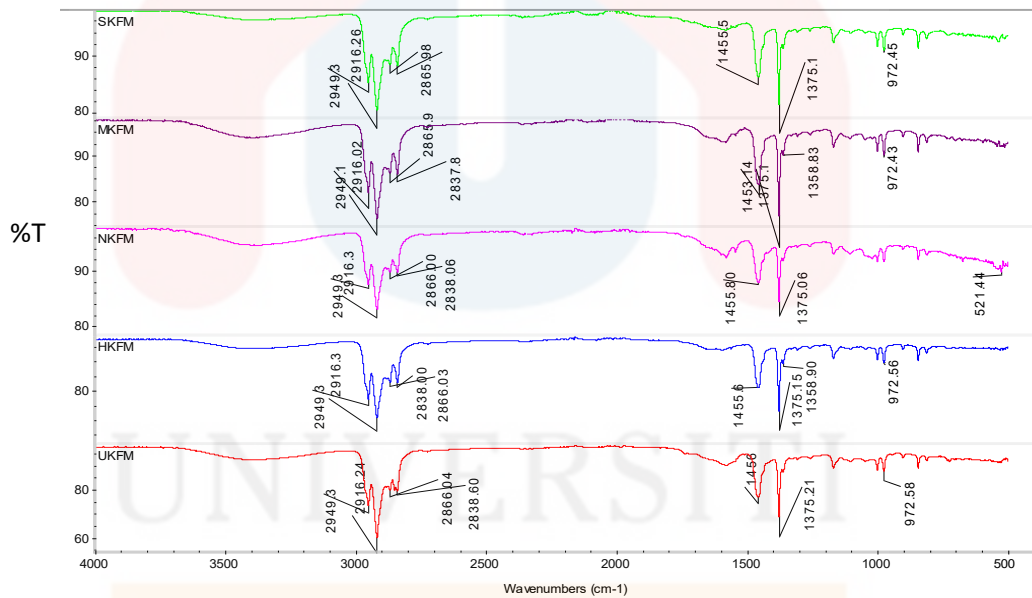### 4.1.1   FTIR Analysis of Untreated and Treated Kenaf Fibre reinforced PP.

Figure 4.1: FTIR spectra of untreated and treated KFM reinforced PP composites

Figure 4.1 showed FTIR spectra of untreated and treated KFM. A broad absorbance peaks in the range $3400 - 3300$ cm^{-1} region were represented stretching O-H groups due to moisture content where hydroxyl easily found in cellulose, hemicelluloses and lignin (Fatah et al., 2014). The peaks for both treated and untreated around 2900 –

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2800 cm^{-1} were indicated C – H stretching vibrations of CH$_2$ and CH groups respectively (Jonoobi, Harun, Shakeri, Misra, & Oksmand, 2009; Saharudin et al., 2018). Lignin is known to contain carboxyl groups represented by carboxyl vibrations between 1750 and 1550 cm^{-1} in the FTIR spectra (Fels, Zamama, & Hafidi, 2015).

Basically peak at 1102.43 cm^{-1} in the UKFM was related to the C-O stretching of the aryl group in lignin (Jonoobi et al., 2009). This disappearance was believed due to the removal of lignin after the chemical treatments. For both treated and untreated KFM, in the range 3000 – 2800 cm^{-1} which represented aliphatic hydrocarbons yet signalled for existence of methane, due to cracking methoxyl(OCH_3-) and methyl$(CH_3 -)$(Nine, Tran, Tung, Kabiri, & Losic, 2017). Furthermore, absorption bands at 1500 cm^{-1} were related to C=O stretching for ketone or aldehyde compounds, $C - O - C$ bend stretching for the groups of phenols, respectively (Nine et al., 2017).

A strong band at 1605 - 1578.21 cm^{-1} assigned for Ar – C = C – H stretching vibration. The disappearance of the bands at 3056 and 915 cm^{-1} indicated to CH_3 asymmetric and symmetric stretching vibrations respectively (Morent, De Geyter, Leys, Gengembre, & Payen, 2008). Appearance of band 1101.80 cm^{-1} is feature for C – N stretching ambience (Nikolic et al., 2010) thus confirm the CH_3 symmetric and asymmetric stretching vibrations respectively into the corresponding polymer as well as crosslinking process which located in MKFM. Range wavelength from 1450 to 1355 cm^{-1} points to stretching of C – O – H bonds in carboxylic acids (Krumins, Klavins, Seglins, & Kaup, 2012). The presence of carboxylic acids in UKFM is more expressed and characteristic which in range 1580 – 1550 cm^{-1} than in other four treated KFM, this zone represented stretching of C – O bonds in carboxylic groups or ethers.

UKFM absorption band at 1375.21 cm^{-1} is owing to CH$_2$ bending lignin whereas the broad peak at 1166.40 cm^{-1} is caused by C – O unfolded bond structure from the functional group of alcohol (cellulose, hemicellulose and lignin). Similarity, there is a slight reduction in the intensity of the bands at 719.93 cm^{-1} in comparison with NKFM and SKFM. Peak band of UKFM stated in medium value, 1455.70 cm^{-1} while NKFM; 1455.80 cm^{-1}.

As summarize, functional group such as carboxylic acids, aryl and carboxyl in lignin had been major factor that altered the interfacial bond on natural fibre surface thus can be conclude that UKFM and NKFM were somewhat have practically properties correspond to strength of the KF. Figure 4.2 stated below showed the correlation for infrared spectroscopy for most of chemical compound.
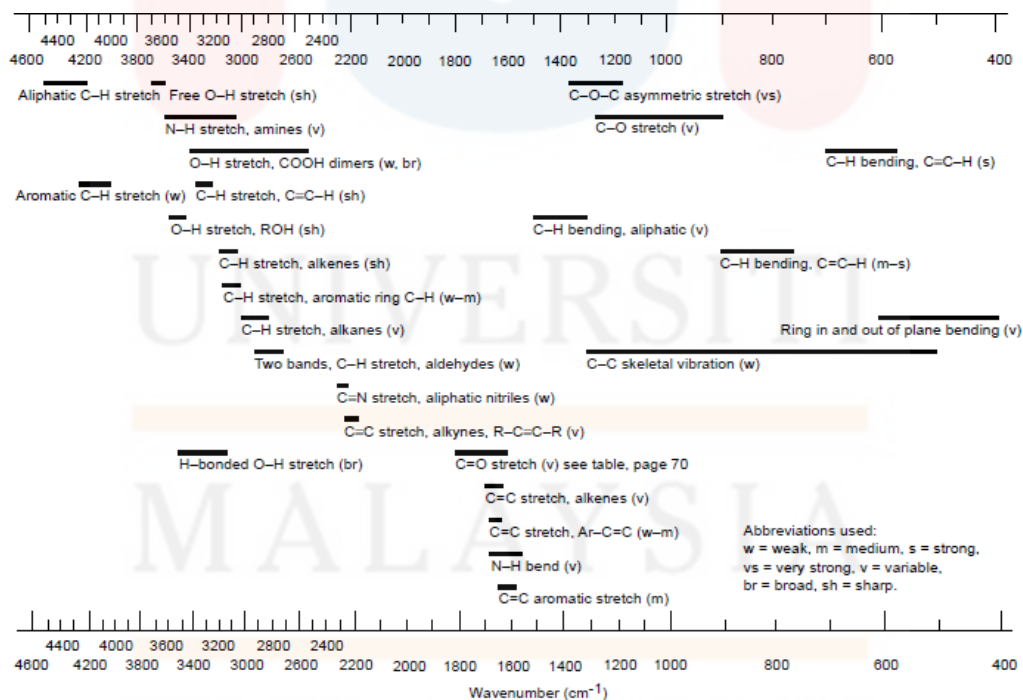

Figure 4.2: Correlation table for infrared spectroscopy, FTIR

Source: (Royal Society of Chemistry, 1992)

4.2 MECHANICAL PROPERTIES OF KFM-PP BIOCOMPOSITES

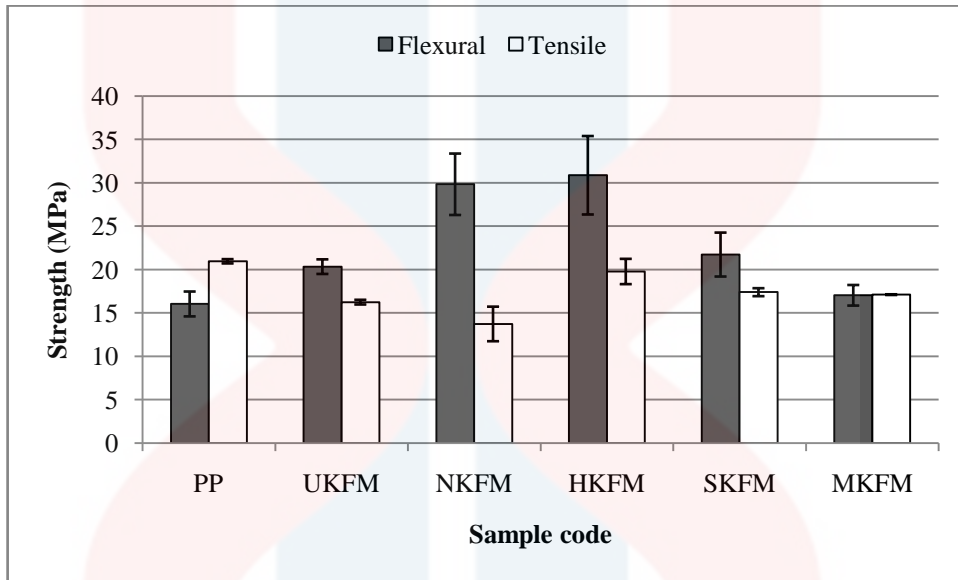### 4.2.1 Tensile and Flexural Strength

Figure 4.3: Tensile and flexural strength for pure PP, untreated and treated KFM-PP.

Figure 4.3 shows the measured average tensile and flexural strength for PP, untreated and four samples of treated KFM-PP biocomposite. The tensile strength of pure PP decreased through the incorporation of KFM. From Figure 4.3, it showed that, pure PP (20.962 MPa) demonstrated the highest tensile strength compared to all untreated UKFM (16.251 MPa) and treated biocomposite HKFM (19.785 MPa), SKFM (17.399 MPa), MKFM (17.117MPa) and NKFM (13.739 MPa). This might be due to the presence of KFM disrupted PP entanglement chain which reduce the toughness properties.

The tensile strength of biocomposites illustrated in Figure 4.3 informed that there has been low deflection on NKFM from untreated UKFM which cause by damage of fibre due to high concentration of NaOH consequently reduce the tensile strength of

the fibre (Edeerozey, Akil, Azhar, & Ariffin, 2007). Tensile strength of NKFM indicated 72.9% lower than other three treated KFM. The interface between fibre and matrix was related to the load share from the matrix to the fibre was proved by this study beneficially. HKFM gain a great tensile properties was cause to terrific fibre-matrix interaction and physical bonding supported by (In, 2017) which this treatment can react with hydroxyl group of the fibre and the matrix resulting in good fibre-matrix adhesion along the composite interphase. The increment of tensile strength indicative on acceleration and coalescence of the cracks in KFM and PP matrix (Hao, Zhao, Jiang, Yuan, & Chen, 2012).

Figure 4.3 also displays the measured average flexural strength of PP, untreated and four treated KFM-PP biocomposites. Hydrogen peroxide treatment HKFM viewed highest (30.88 MPa) flexural strength, NKFM (29.839 MPa), SKFM (21.736 MPa), UKFM (20.342 MPa), MKFM (17.041 MPa) while pure PP viewed as the least flexural strength (16.042 MPa).

The flexural strength of biocomposites as interpreted in Figure 4.3 reduced by variation of chemical treatment and amount of water held with same fibre content. Flexural strength of MKFM indicated 88.1% lower than other three treated KFM. Pure PP get stiffer and loss of elasticity due to decrement of flexural strength cause of incompatible during chain – scission. The flexural strength of the biocomposite increased linearly at different chemical treatment and increased more higher than comparable tensile strength obtained in this study. Such a degree, MKFM and NKFM are two different chemical compounds which were cannot withstand in flexural strength and tensile strength cooperatively compared to other treated KFM.

This experiment bolded in the flexural strength was increased with increasing bonding surface area because of better adhesion between fibre and matrix. As the chemical compound changed, the variation of bonding interaction on surface area dispersed widely for tensile strength and flexural strength. Due to overload amount of water in the fibre and insufficient amount of bonding, bonding mechanism of fibre and matrix were not effectively be transferred by loads from one end to another hence reduction formed in flexural strength and tensile strength of the biocomposite. When the bonding between fibre and matrix was increased, tensile and flexural strength increased proportionally and strong bonding transferred loads from one end to another.
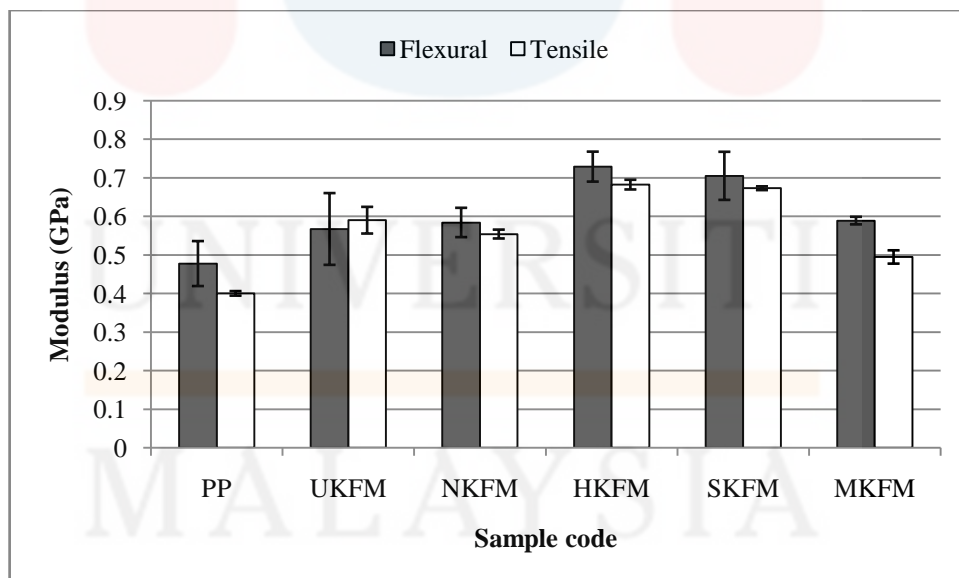
4.2.2 Tensile and Flexural Modulus

Figure 4.4: Tensile and flexural modulus for pure PP, untreated and treated KFM-PP.

Figure 4.4 illustrated an average tensile and flexural modulus for PP, untreated along with four samples of treated KFM – PP biocomposite. The tensile modulus

increased from pure PP (0.4002 GPa), MKFM (0.4946 GPa), NKFM (0.5541 GPa), UKFM (0.5900 GPa), SKFM (0.6729 GPa) and while the highest tensile modulus was HKFM (0.6823 GPa).

Figure 4.4 informed that tensile modulus of biocomposites with varieties of chemical treatment. It can be observed that the tensile modulus was rise by the implementation of varieties of chemical treatments at the same fibre content. Tensile modulus of HKFM was at the high peak with 98.74% than other KFM-PP biocomposites.

Due to better rigidity, values of HKFM biocomposite are much higher than UKFM biocomposite. Additionally, it approved that HKFM or bleached KFM biocomposite exhibited better tensile modulus at each fibre loading compared to UKFM biocomposite (Razak et al., 2014). In contrast, this higher value of tensile modulus was altered by the fibres that support the stress from the matrix.

In Figure 4.4, average tensile and flexural modulus for PP, untreated and four samples of treated KFM-PP biocomposite. Whereas, flexural modulus increased from pure PP (0.4775 GPa), UKFM (0.5673 GPa), NKFM (0.5841 GPa), MKFM (0.5890 GPa), SKFM (0.7051 GPa) and while the highest tensile modulus was HKFM (0.7289 GPa).

Based on Figure 4.4, there are varieties of treated KFM, untreated KFM and pure PP. Flexural modulus was generally improved by implementation of variance chemical compound onto the same fibre content. The KFM treatment by hydrogen peroxide in room temperature greatly increased the flexural modulus and flexural strength.

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In consideration of hydrogen peroxide treatment on the flexural modulus, the results formed was the flexural modulus of biocomposites that was 96.1% higher than untreated, because of steep interaction and great dispersion of the fibre in matrix like so this increment affected good compatibility between fibre and matrix (Razak et al., 2014). The result showed that surface alteration by chemical treatment by maleic anhydride polypropylene (MAPP), MKFM has less influence value on flexural strength and tensile modulus compared to flexural modulus and tensile strength, respectively.

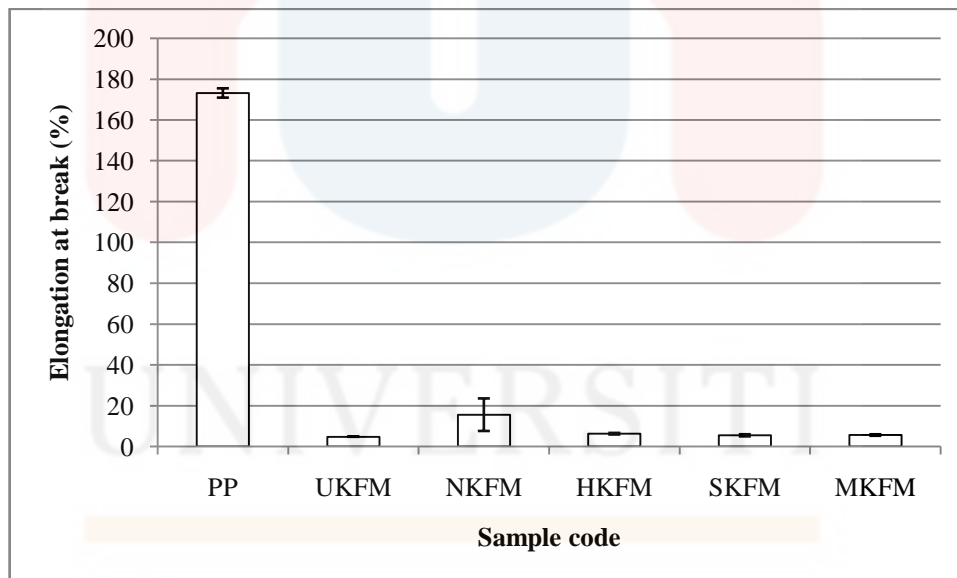### 4.2.3   Tensile Elongation at Break

Figure 4.5: Tensile elongation at break for pure PP, untreated and treated KFM-PP.

Figure 4.5 illustrated an average tensile elongation at break for PP, untreated and four samples of treated KFM-PP biocomposite. The tensile elongation at break decreased from pure PP (173.148%), NKFM (15.542%), HKFM (6.231%), MKFM (5.579%), SKFM (5.439%) and while the lowest was UKFM (4.755%).

The results proved that variation of chemical treatments at the same fibre content declines the elongation at break of the biocomposite by cause of KF and pure PP are rather brittle and stiff. Common expectation which is the biocomposites had lower elasticity than pure PP. Differences chemical compounds made the breakage of the biocomposite increased. Fracture behavior gave better of bond strength between fibres and matrix in the biocomposite (Kongkaew & Choawanklang, 2015).

Furthermore, the decline in fundamental elongation at break was cause by loading of KF initially can eliminate structural principle of pure PP. Most treated KFM originated fast breakage can interpretation of good fibre-matrix adhesion otherwise was the progressive decline on the elongation at break can be attributed to a reduction in deformability of the inflexible interface between fibres and matrix.

4.3   SEM ANALYSIS

4.3.1   Morphology of KFM-PP Biocomposites

Basically on Figure 4.6a, a pure polypropylene (PP) where at x100 magnification in high vacuum. (Bashree et al., 2016) presented that presence of plastic deformation in the form of matrix fibrillation on the surface is believed to be corresponded for a high value in toughness – related properties. Figure 4.6b shows SEM micrographs obtained from untreated kenaf fibre that featured the interfibrillar voids on the centre of samples. Declination thus poor penetration or transferred into fibre-matrix also can be accredited to voids ensuing from of the lack of matrix material.

Kenaf fibre treated with hydrogen peroxide on the surface at x500 magnification in high vacuum condition seen that most of fibre imbedded deeply inside the PP matrix thus indicate the increase of fibre-matrix adhesion between KFM and PP matrix, a main factor for the improved strength, modulus and toughness properties (Bashree et al., 2016).
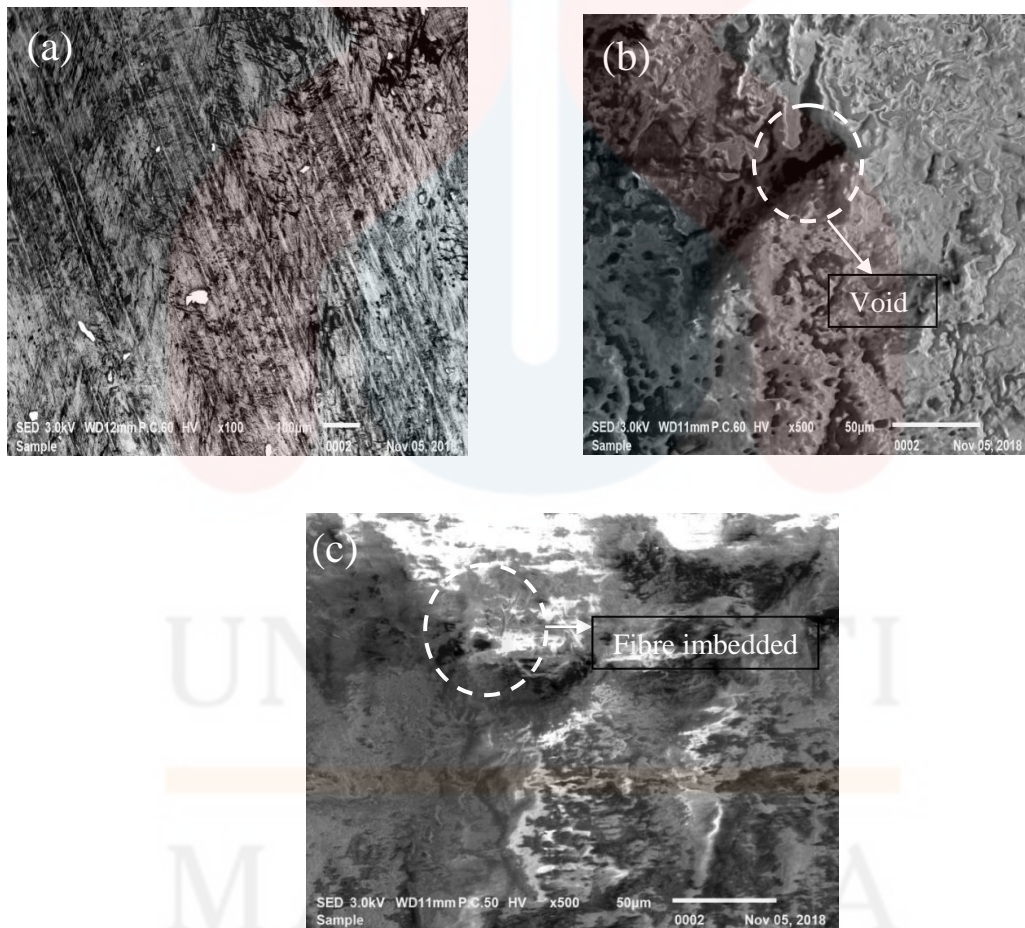

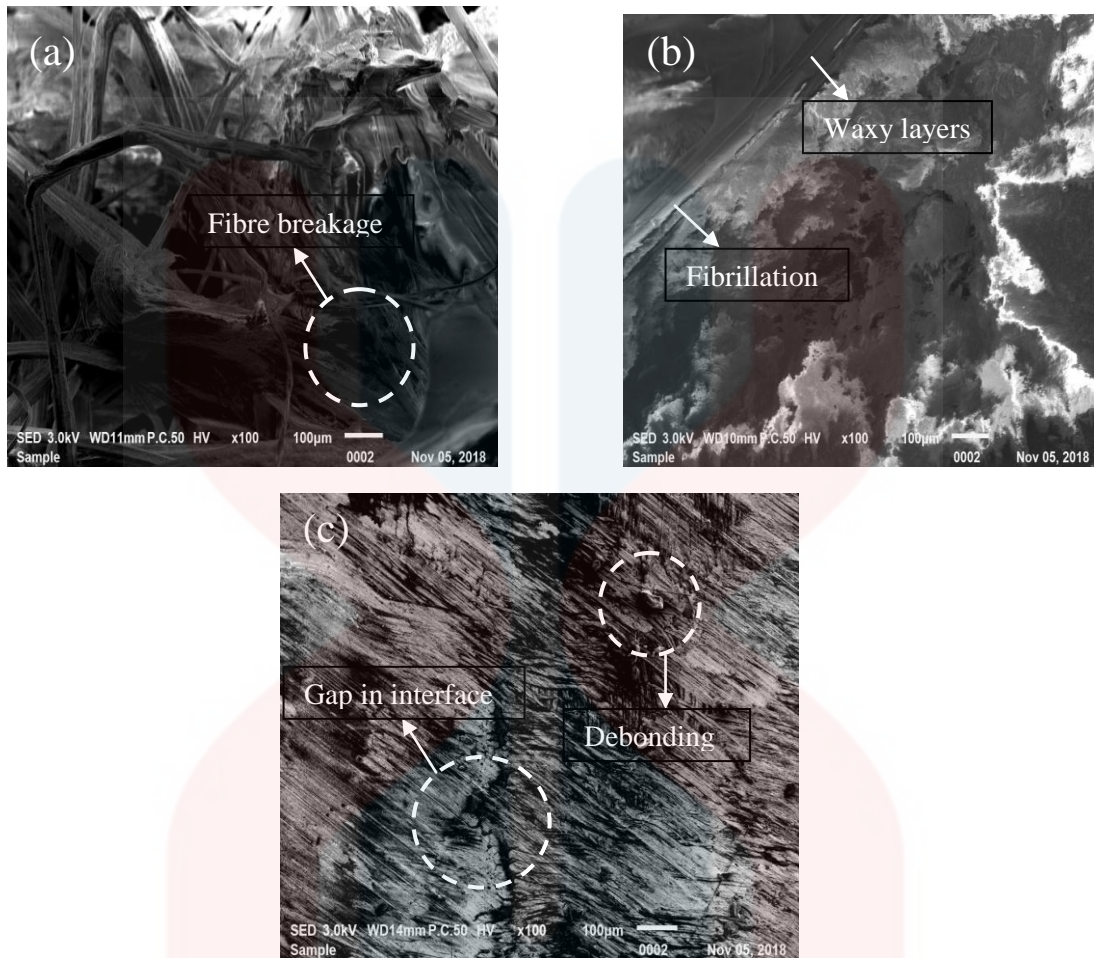Figure 4.6: SEM micrograph of (a) Polypropylene (PP), (b) UKFM and (c) HKFM

Figure 4.7: SEM micrograph of (a) NKFM, (b) SKFM and (c) MKFM

SEM image on the surface at x100 magnification depicted in Figure 4.7a was NKFM where a lot of fibre pulls – out basically cause by cracks that propagated between kenaf fibre and PP matrix interfaces. This phenomenon can resulted to greater extent of delamination. Asumani et al.(2012) agreed that Figure 4.7a fracture is predominately characterized by fibre breakage.

The fibrillation seen on SKFM biocomposite on the surface at x100 magnification in high vacuum condition Figure 4.7b attributed to optimum tensile

strength because of significant stress can impregnate through the fibre and break the untreated fibre bundle down into smaller ones by dissolution of the hemicelluloses.

Diversely on both Figure 4.6 (a) – (c) and Figure 4.7 (a) – (c) SEM observations determined that was a significance and considerable differences in the fibre-matrix interaction between untreated, chemical treated and pure polypropylene. There are similarities on MKFM and UKFM have debonding surface and voids which were engaged for irrelevant on tensile and flexural strength.

NKFM specimen where 5wt% (Figure 4.7a) showed a fibre breakage, capillarity and transport via micro cracks become active thus water flow involved along fibre-matrix interfaces and a process of diffusion through the bulk matrix (Maslinda, Abdul Majid, Ridzuan, Afendi, & Gibson, 2017). There is a huge void on UKFM specimen (Figure 4.6b) compared to SKFM and MKFM specimens (Figure 4.6 (b) – (c)) thus the low interfacial interaction of fibre – matrix likewise tensile strength by UKFM specimen. Lastly, HKFM morphological showed in Figure 4.6c showed the optimum condition of fibre-matrix adhesion which improved on strength, modulus and toughness properties.

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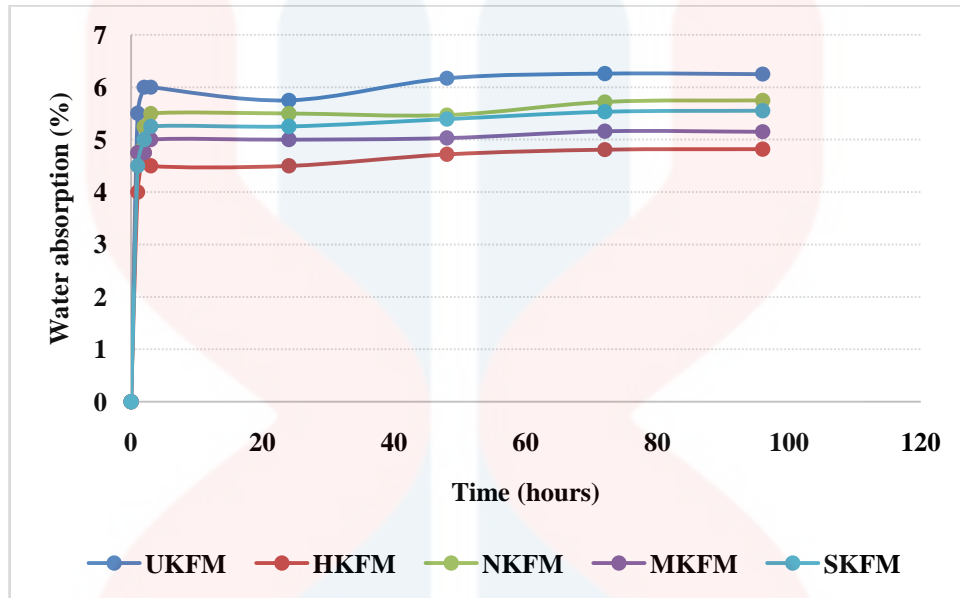
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4.4 WATER ABSORPTION

Figure 4.8: Water absorption percentage at room temperature for untreated and treated KFM-PP

Figure 4.8 viewed water uptake in duration of 1, 2, 3, 24, 48, 72 up to 96 h for both treated and untreated KFM – PP at room temperature. The maximum percentage gainUKFM (6.25 %), HKFM (4.82 %), NKFM (5.75 %), MKFM (5.15 %) and SKFM (5.55 %). The water absorption process for all samples, which hardly absorbs any water, is decline at the beginning 3 h then increased slowly plus linearly approaches saturation after prolonged time. Equally, the early and final pace of water absorption percentage increased for all KFM-PP biocomposite samples as the fibre volume part rise. Based on (Ververis, Georghiou, Christodoulakis, Santas, & Santas, 2004) there is about 43% of cellulose content in kenaf can contributed to water penetrating into the interface through the micro cracks induced by swelling of fibres creating swelling stresses leading to composite failure. As the composite damaged due to crack, capillarity and transport via micro cracks become active (Maslinda et al., 2017). Without chemical treatment,

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untreated UKFM showed the highest water uptake. The presence of chemical treatment significantly reduced water uptake by almost 30%.

The application of peroxide (HKFM) showed the most effective water uptake reduction then followed by maleated PP (MKFM), silane (SKFM) and alkaline (NKFM). Other study done by Afzaluddin, Jawaid, & Sapuan (2018) also obtained the same result since the surface modification for both HKFM and MKFM undergo bond formation stage and condensation that induced fibre-matrix structures form the reaction hydroxyl groups. Hence percentage of water absorption reduced due to moisture induced interfacial cracks at an accelerated rate as result of degradation (Maslinda et al., 2017) beside improved interfacial adhesion between KFM and PP as matrix supported by chemical treatments on the kenaf fibre.

As summarize, correlation between water absorption with flexural and tensile test were theoretically less water penetrating into the interface through the micro cracks induced by swelling of fibres implemented high toughness on both flexural and tensile of the KFM-PP biocomposites.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 CONCLUSION

This study set out to fabricate KFM – PP composites by applying sandwich skin (PP) and core (KFM) structure and compression moulding techniques. The present research was designed to analyze the effect of chemical treatment on mechanical and physical properties of KFM – PP biocomposites. The following conclusions can be drawn from the present research.

Result of SEM observation showed that better surface interfacial interaction between KFM and PP matrix although either untreated or treated sample was still showed debonding, void and fibre pull out. In contrast HKFM showed a magnificent improvement by just fibre imbedded and immersed deeply with PP matrix in their morphology. The contribution of this research has been to confirm hydrogen peroxide treatment (HKFM) displayed the best result to a significant enhancement of mechanical fulfillment such as tensile and flexural properties of KFM – PP biocomposites.

Physical test essentially approved in the water absorption mechanism which HKFM demonstrated the most effective water uptake reduction then followed by MKFM, SKFM, NKFM and lastly UKFM. Finally, HKFM can be concluded as treated kenaf fibre that had the optimum interfacial bond strength with encompassed toughness,

most likely spread over the interfacial area which can be investigated through tensile strength, flexural strength and water absorption mechanism in this research result.

5.2   RECOMMENDATIONS

These findings favored the following insights for future research:

i)   The introduction of filler as surface acetylation for better and induced the production cost furthermore can overcome hydrophilic character of natural fibre.

ii)   Control of water humidity, pressure and time during processing to manufacture good composites. The stainless-steel mould must frequently clean and avoid moisture along with natural fibre.

iii) Average reading of test samples must repeat four to seven times thus reading can be accurately and elimination of standard error of measurement so that data trends can be nearly visible.

iv)   Proper mechanism for reduction of charge in SEM test such as gold coating or copper tape must be given on the sample in order to achieve a better surface morphology image.

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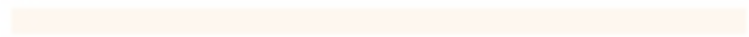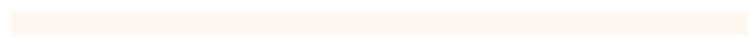
APPENDIX A

Equipments

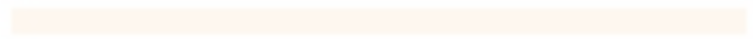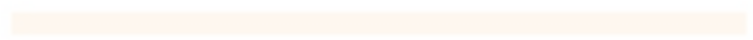|	
Thermo Scientific Nicolet iN10 Infrared Microscope and iZ10 FT-IR Spectrometer	Jeol JSM-IT100 Scanning Electron Microscope
Testometric M500-50T	Hot press bonding GT-7014-H
Handsaw	