MECHANICAL, THERMAL AND MORPHOLOGICAL STUDIES OF EPOXIDIZED PALM OIL / UNSATURATED POLYESTER / MONTMORILLONITE COMPOSITES

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DEDICATION

‘For my lovely Mom and Dad, my sisters and my brothers’

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ABSTRACT

Nanocomposite consisting of natural fibre reinforced thermoset/vegetable oil/montmorillonite (MMT) nanofiller resin is among the latest technologies in nanocomposites production. It has contributed to better environment and increase the sustainability of the composite materials. To tap the abundance of palm oil in Malaysia, this study was conducted to produce a novel composite of kenaf fibre reinforced unsaturated polyester (UPE)/epoxidized palm oil (EPO)/MMT by resin transfer moulding (RTM) followed by oven curing. Benzoyl peroxide was used as an initiator and the composition was kept constant at 1.5 phr. The compositions of UPE/EPO resin were varied at 90/10, 80/20, 70/30 wt % and the MMT nanoclay were varied at 1, 1.5 and 2 phr for each formulation. The effects of different loading ratios of EPO and MMT in UPE/EPO/MMT and the different RTM mould angles in kenaf fibre reinforced UPE/EPO/MMT composite were studied in particular mechanical, morphology, thermal, and water absorption properties of the resin and composite. Pre-characterisations of UPE/EPO resin were carried out using Fourier transform infrared spectroscopy and differential scanning calorimetry. The results revealed that the oxirane rings of EPO were successfully opened and the resin successfully crosslinked at applied curing temperature. The addition of EPO has caused a decrease in glass transition temperature, tensile, flexural properties and an increase in elongation at break and impact strength, which indicated an increase in mobility and toughness of UPE chain. UPE/EPO blends also exhibited higher thermal stability than neat UPE. The addition of MMT nanoclay enhanced the mechanical and thermal properties of the resin. The optimum balance properties were obtained for UPE at 10 wt % EPO and 1.5 phr MMT nanoclay loadings. The best formulation was then carried out at 0º, 30º and 45º RTM mould angle with kenaf fibre reinforcement. 30º RTM mould angle gave the highest mechanical and thermal properties of the resulted kenaf fibre reinforced UPE/EPO/MMT composite.

ABSTRAK

Nanokomposit yang terdiri daripada gentian semulajadi yang diperkukuhkan dalam resin termoset/minyak sayuran (VO)/pengisi nano montmorilonit (MMT) adalah antara teknologi terkini dalam penghasilan komposit. Ia telah menyumbang kepada persekitaran yang lebih baik dan meningkatkan kemampanan bahan-bahan komposit. Untuk memanfaatkan lambakan minyak sawit di Malaysia, kajian ini telah dijalankan bagi menghasilkan satu komposit baru gentian kenaf bertetulang poliester tak tepu (UPE)/minyak sawit terepoksi (EPO)/MMT oleh acuan pemindahan resin (RTM) dan diikuti oleh pengawetan ketuhar. Benzoil peroksida telah digunakan sebagai pemula dan komposisinya telah digunakan pada kadar tetap, 1.5 bahagian peratus resin (phr). Komposisi adunan UPE/EPO telah diubah pada 90/10, 80/20, 70/30 % berat dan tanah liat nano MMT telah diubah pada 1, 1.5 dan 2 phr bagi setiap formulasi. Kesan nisbah muatan EPO dan MMT yang berbeza dalam UPE/EPO/MMT dan sudut acuan RTM yang berbeza dalam gentian kenaf bertetulang UPE/EPO/MMT komposit telah dikaji secara khusus dalam sifat-sifat mekanikal, morfologi, penyerapan haba, dan air bagi resin dan komposit. Pra-pencirian UPE/EPO resin telah dijalankan dengan menggunakan spektroskopi inframerah transformasi Fourier dan kalorimeter pengimbasan pembezaan. Keputusan telah menunjukkan bahawa cincin oxiran EPO telah berjaya dibuka dan resin telah berjaya terpaut silang pada suhu pengawetan yang digunakan. Penambahan EPO menyebabkan penurunan suhu peralihan kaca, tegangan, sifat lenturan dan peningkatan dalam pemanjangan pada waktu rehat dan kekuatan impak, yang menunjukkan peningkatan dalam kekuatan dan mobiliti rantaian UPE. Penambahan EPO juga menghasilkan kestabilan haba yang lebih tinggi daripada haba kemas UPE resin. Di samping itu, penambahan tanah liat nano MMT menunjukkan peningkatan dalam sifat-sifat mekanikal dan haba resin. Sifat optimum keseimbangan telah diperoleh bagi UPE pada 10 % berat EPO dan 1.5 phr muatan tanah liat nano MMT. Formulasi resin yang terbaik ini kemudiannya diproses pada sudut acuan RTM 0º, 30º dan 45º dengan tetulang gentian kenaf. Sudut acuan RTM 30º memberikan sifat-sifat mekanikal dan haba tertinggi di dalam gentian kenaf bertetulang UPE/EPO/MMT komposit.

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LIST OF ABBREVIATIONS

UPE	-	Unsaturated polyester resin
EPO	-	Epoxidized palm oil
MMT	-	Montmorillonite nanoclay
BP	-	Benzoyl peroxide
UPE/EPO	-	Unsaturated polyester/epoxidized palm oil
UPE/MMT	-	Unsaturated polyester/montmorillonite
UPE/EPO/MMT	-	Unsaturated polyester/epoxidized palm oil/montmorillonite
FWHM	-	Full width half maximum
PMC	-	Polymer matrix composite
CMC	-	Ceramix matrix composite
MMC	-	Metal matrix composite
FRP	-	Fibre reinforced polymer
PHA	-	Polyhydroxyalkanoates
PHB	-	Polyhydroxybutyrate
PHVB	-	Poly(3-hydroxybutyrate-co-3-hydroxyvalerate)
PLA	-	Polylactic acid
PCL	-	Polycaprolactone
PEA	-	Polyesteramides
PBSA	-	Polybutylene succinate adipate
EML	-	Epoxidized methyl linseedate
EMS	-	Epoxidized methyl soyate
PGO	-	Pongamia glabra seed oil
ESO	-	Epoxidized soybean oil
EAS	-	Epoxidized allyl soyate
AESO	-	Acrylated epoxidized soybean oil
KF	-	Keratin feather fibre
RTM	-	Resin transfer moulding

VARTM	-	Vacuum assisted resin transfer moulding
SRIM	-	Structural resin injection moulding
LCM	-	Liquid composite moulding
FTIR	-	Fourier transform infrared
DSC	-	Dynamic scanning calorimetry
TGA	-	Thermogravimetric analysis
DMA	-	Dynamic mechanical analysis
XRD	-	X-ray diffraction
SEM	-	Scanning electron microscopy
TEM	-	Transmission electron microscopy
ASTM	-	American standard testing material
HDT	-	Heat distortion temperature
MEKP	-	Methyl ethyl ketone peroxide
NaOH	-	Natrium hydroxide
KBr	-	Calcium bromide
IUPAC	-	International Union of Pure and Applied Chemistry

LIST OF SYMBOLS

wt %	-	Weight percent
e.g.	-	For example
i.e.	-	That is
ºC	-	Degree celsius
m	-	Meter
mm	-	Millimetre
mm/min	-	Millimetre per minute
h	-	Hour
cm	-	Centimetre
cm^3	-	Centimetre cube
nm	-	Nanometre
m^2	-	Metre square
m^2/g	-	Metre square per gram
%	-	Percent
˚C/min	-	Degree celsius per minute
GPa	-	Giga pascal
KPa	-	Kilo pascal
MPa	-	Mega pascal
Pa.s	-	Pascal second
L	-	Liter
kg	-	Kilogram
M	-	Molar
mg	-	Milligram
in	-	inch
J	-	Joule
kV	-	Kilovolts
mA	-	Milliampere

J/g	-	Joule per gram
phr	-	Part per hundred resin
R	-	Gas constant [8.314 J/K.mol]
E	-	Activation energy
T	-	Absolute temperature
k	-	Permeability
P	-	Pressure
ρ	-	Density
Tg	-	Glass transition temperature

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Composites are one of the wide spread areas that have been applied present due to the high performance of composites compared to neat polymer. Both thermoset and thermoplastic composites have widened the applications and improved the performance of polymer industries. Many composite industries have generated significant economic activity in many countries. Main industries that have applied composites materials include automotive, construction, furniture, aircraft, boat, chemical, electrical and sports industries (Richard, 2005). The manufacturers, designers, and engineers recognized the ability of composite materials to produce high quality and cost-effective products that are high strength, light weight, corrosion resistant, flexible in design and durable. These advantages are reflected in the high demand for composite products.

In order to increase the environmental concern of composite product, natural fibres have been used rather than synthetic fibres. Replacing synthetic fibre (such as glass fibre, carbon fibre and aramide fibre) with natural fibre has both environmental and economic advantages (Williams and Wool, 2000). Among the common natural fibre is kenaf. Kenaf, known by its scientific name as *Hibiscus Cannabinus, L.* has been cultivated for use in ropes and animal consumption for the last 4,000 years. Kenaf fibre has been produced mostly for textiles, gunny sacks and, to a certain extent, paper. Recently, kenaf fibre is also being applied in composite field. Due to the increase in application of kenaf fibre, the crop has begun to attract much attention and interest of many countries. China, Indonesia, Myanmar, Thailand and United States of America are the traditional growers of kenaf for the world market. Malaysian Government is also heavily promoting the development of kenaf as the next major industrial crop for the country in line with its policy of developing new sources of economic growth. Up to February 2011, the Government had spent RM50 million under the purview of the Ministry of Plantation Industries and Commodities to develop kenaf through research and development in upstream and downstream areas (Khan, 2011). In 2012 and 2013, the federal government disbursed a total of 33 and 48 million RM respectively under the New Plant Kenaf Program (MPIC) which was developed to manage and develop the kenaf plant in Malaysia (Annual Report, 2013).

In composite industries, natural fibres including kenaf fibre are used as an alternative to form green composite, replacing the synthetic fibre commonly used previously. Many researchers have reported on natural fibre reinforced petroleum-based thermoplastic (Kozlowski *et al.*, 2004; Tajvidi *et al.*, 2006) and thermoset (Mohamed Yusoff, 2009; Trujillo *et al.*, 2010; Jawaid *et al.*, 2012; Adriana *et al.*, 2013) polymer. Even though natural fibre contributed to a better environment, natural fibre reinforced polymer resin composites are not sufficiently eco-friendly because of the petroleum-based source and the non-biodegradable nature of the polymer matrix (Mohanty *et al.*, 2002a). This phenomenon indicates that the development of natural fibre reinforced renewable source based resin is important. Other than solving the environmental issues, the composite from natural fibre and renewable source based resin is also increased the sustainability of the composite materials (Zhu *et al.*, 2004). Thus, developing the bioresin from renewable sources is a clear future prospect.

Over the past decade, natural oils, as a feedstock for thermosetting resin has gained a lot of attention due to their availability and low cost (Pfister and Larock, 2010). However, it was found that the use of plant oil based resin in composites has been limited, due to their lower mechanical and thermo-physical properties compared to synthetic resin. A promising compromise between environmental friendliness and performance of bio-based resin was obtained by replacing part of a petroleum based resin with natural plant oil (Dutta and Karak, 2005). The most reported starting materials for bio-based thermoset resin are soybean oil (Haq *et al.*, 2008; Nava and Brooks, 2010; Tan and Chow, 2011a), linseed oil (Haq *et al.*, 2011), rapeseed oil and nahar oil (Dutta *et al.*, 2004; Mahapatra and Karak, 2004; Dutta and Karak, 2005). In addition, corn, olive and canola oil are being used as the starting raw material for bio-based resin (Williams and Wool, 2000). As reported by previous studies, the additions of plant oil in petroleum based thermoset resin has improved the toughness, barrier and thermal properties of the resulting bioresin system (Dutta and Karak, 2005; Haq *et al.*, 2008, 2009b, 2011). However, according to Haq *et al.* (2009a, 2009b), increase in toughness performance generally against the stiffness performance of the material. Thus, some filler needs to be added in order to have proper balance on toughness-stiffness (Haq *et al.*, 2008, 2009b) and improve certain properties of the bioresin.

Layered silicate nanoclay has been discussed in past decades as reinforcement filler in polymer nanocomposites industries. Polymers reinforced with nanoclays have been shown to exhibit enhancements in mechanical, thermal and barrier properties at low concentration. As reported in previous study, the MMT addition in vegetable oil based bioresin has recovered the decrease in stiffness, thermal and barrier properties of the bioresin due to addition of plant oil and it leads to improved stiffness-toughness balance of the bioresin (Miyagawa *et al.*, 2006b). Thus, the addition of nanoclay has leads to improved the stiffness-toughness balance of the resin and produced a polymer nanocomposite with similar or enhanced properties compared to the neat polymer (Haq *et al.*, 2008, 2009a, 2009b, 2011). This condition holds a great promise for the resin to be used in wide composite applications.

Palm oil is the world's highest yielding oil crop, which is abundantly available in South East Asian countries, especially in Indonesia and Malaysia (Biron, 2004). Malaysia is the second largest producer of palm oil in the world after Indonesia. According to the report from United States Department of Agriculture (USDA) (2015), 86 % of world production of palm oil in 2014/15 was dominated by

Malaysia and Indonesia. Palm oil is mostly used in food as cooking oil, shortening and margarine, while in oleochemicals industry, functionalized palm oil are used in manufacturing soaps, cosmetics, detergents, lubricants, glues, and also as raw material for biodiesel. In the last few decades, palm oil applications in the pharmaceutical, engineering and industrial areas have been explored by many researchers. However, the use of epoxidized palm oil as resin in composite matrix applications have not been explored and fully realized. Widening the applications of palm oil will give a significant return to the agriculture sector together with the environment. Due to the sustained availability of palm oil in Malaysia, development of new products from palm oil derivatives such as epoxidized palm oil (EPO) has been accorded priority in Malaysia. Consequently, it is beneficial to partly combine the functionalized palm oil to petroleum based thermoset resin to form a new biobased resin.

Unsaturated polyester resin (UPE) is one of the most important thermosetting polymers, which are being used widely in Fibre Reinforced Plastic (FRP) applications. UPE resin are more commonly used than other thermoset resin due to their attractive price per properties ratio (Biron, 2004). In addition, unsaturated polyester resin also possess some good properties such as good mechanical and electrical properties, fairly good heat and creep behaviors, aesthetic, choice of rigidities, resistance to a great number of chemicals, resistance to light, weathering and water in spite of surface deteriorations. It is also easily used in the preparation of composite materials due to the simplicity and flexibility to modify their chemical structure that will allow changes in viscosities and curing time to be used in large variety of applications (Nava and Brooks, 2010) including house-wares, transportation, sports equipment, electrical industries and many more.

This study was focused on developing a new resin by partially blended EPO in UPE as a matrix in kenaf fibre reinforced composites. To improve the loss stiffness of the resin matrix system due to the addition of EPO, MMT nanoclay filler was also added. Kenaf fibre reinforced UPE/EPO/MMT resin was then processed by using resin transfer moulding (RTM).

1.2 Problem Statement

Fibre reinforced unsaturated polyester resin (FRP) is highly preferred in material industries including marine, automotive, sanitary-ware, furniture industries and many more. It was due to its advantages i.e. light in weight, low cost, high strength and elasticity, good thermal and electrical properties, rigid, fairly good heat and creep behaviour and excellent resistance to corrosion. However, the major concern of FRP is its disposal. FRP is very difficult to dispose since it consist of non-biodegradable petroleum based polyester resin and synthetic fibre. Unlike thermoplastic composite, FRP is not considered environmental friendly since it non-biodegradable, cannot be reused and recycled. The only method to dispose FRP is by cutting it into small fragments and sends to the landfill. This disposal method is considered unsustainable for long run. In order to preserve the environment and to reduce the solid waste volume in landfill, the production of bio-based composite is reinforced.

Bio-based composites have received extra attention in recent years. For example, there are many studies conducted to investigate the use of natural fibres to replace synthetic fibres in polymer composites. It was proven that the natural fibres reinforced unsaturated polyester (UPE) composites enhance better environmental characteristics of the composites (Williams and Wool, 2000). However, the composite does not sufficiently eco-friendly due to the non-biodegradable nature of the synthetic UPE resin matrix (Mohanty *et al.*, 2002a). Thus, the study of polyester based on renewable resource resin to replace synthetic based resin is needed. The synthetic UPE resin was synthesized from chemical reaction of diacid and diols, which are both petroleum based. The depletion of petroleum resources coupled with increasing environmental regulations are acting synergistically to provide the impetus for new materials and products that are compatible with the environment and independent of fossil fuels. However, synthesizing new UPE based resin directly from the vegetable oil has some limitations. This is mainly because the vegetable oil based UPE resin does not show properties of rigidity and performed low mechanical strength which was not suitable for most composite applications especially for structural applications (Miyagawa *et al.*, 2006a; 2007). Therefore, partially blend vegetable oil with the synthetic UPE resin is required.

Resin transfer moulding (RTM) is one of the common thermoset composite processing method applied in industries. It was due to its advantages i.e. low pressure involves, short production time, perform two sided finishing product, and low processing cost as low skill level operator and less labour service needed. Even though RTM is an advanced processing method, the main concern using RTM is because of the resin flow properties. Poor resin flow properties may affect to the poor fibre wetting which consequently will drop the performance of the resulted composite materials. Thus, attaining a good resin flow with good fibre matrix impregnation was the main focus in RTM processing. RTM mould angle position is one of the factors that has a significant effect on the resin flow properties. However, there are very limited study conducted on the effect of gravitational (RTM mould angle) on the fibre wetting and properties of the resulted composite. In fact, there was no study reported on the effect of mould angle on the natural fibre reinforced modified thermoset/vegetable oil resin.

The production of new composite consists of natural fibre and UPE/vegetable oil resin processed by RTM is a new venture that can be explore. Because of the availability of palm oil in Malaysia, development of new products of eco-friendly UPE resin composites from palm oil derivatives such as EPO has been of particular interest in this project. Thus, the objective of this research is to explore the potential of EPO to be replaced partially in UPE as a matrix in natural fibre reinforced composite, in particular kenaf fibre. In order to improve the stiffness properties of the new resin, MMT nanoclay filler was added. The best formulation of UPE/EPO/MMT blend resin was reinforced with kenaf fibre and the composite prepared using RTM at 0, 30 and 45º mould angle. Following are some of the questions that need to be answered to expand this research area.

1) What are the effects of blend ratio of UPE/EPO/MMT resin on thermal characteristic and curing behavior as well as its resistance to moisture absorption?

2) What are the effects of blend ratio of UPE/EPO/MMT resin on the mechanical and thermophysical properties of the polymer resin such as tensile, flexural, impact strength, storage modulus and tangent delta?
3) What are the dispersion and morphology of EPO and MMT nanoclay inclusion in UPE and the blend of EPO/UPE resin?
4) What are the mechanical, thermal and morphological properties of kenaf fibre reinforced UPE/EPO/MMT composite prepared by using RTM process?

It was reported that previously that replacing some of an UPE with vegetable oil has contributed to increase the toughness and altered the thermal properties of the neat UPE resin (Xia and Larock, 2010). However, the increase in toughness seriously lowers the stiffness properties of the new UPE resin. Therefore, the addition of MMT nanoclays was important to improve the stiffness properties of the UPE/vegetable oil resin (Haq et al., 2008, Tan and Chow, 2010). The addition of MMT may also improve the barrier properties between the vegetable oils added and the UPE (Rigoberto Burgueno et al., 2007). The modified UPE/EPO resin reinforced kenaf fibre processed with higher RTM mould angle was provided better fibre wetting and increase the properties of the resulted new composite.

1.3 Objectives of Study

This research aims at developing environmentally friendly composite using UPE/EPO/MMT blend as matrix with the reinforcement of kenaf fibre. The objectives of this study are as follows:

1) To investigate the effect of EPO blends on chemical structure and curing behaviour of UPE resin.
2) To characterise the effects of EPO content on the mechanical, thermal, thermophysical, morphology and water absorption properties of UPE/EPO resin.

3) To characterise the effects of MMT content on the mechanical, thermal, thermophysical, morphology and water absorption properties of UPE/EPO/MMT resin.
4) To investigate the effects of different RTM mould angle on the mechanical, thermophysical and morphology properties of kenaf fibre reinforced UPE/EPO/MMT composite.

1.4 Scope of Study

In order to achieve the objectives of the research, the following activities were carried out:

The first part of the study, the modified UPE/EPO resin were prepared with various amounts of EPO (10, 20 and 30 wt %). In this step, the EPO was added into UPE resin and cured in an oven at 100 ºC (2 hours) and 160 ºC (2 hours) as referred to previous study (Miyagawa *et al.*, 2006a; Miyagawa *et al.*, 2007; Haq *et al.*, 2009a, Haq *et al.*, 2011). The chemical structure of the cured UPE/EPO blend resin was then characterised using Fourier transform infrared (FTIR). In order to understand the curing behaviour of the UPE/EPO blend resin,the uncured UPE/EPO blend resin sample was heated in the diffrential scanning calorimetry (DSC) and the enthalpy was evaluated. Next, the effect of different EPO content in the UPE/EPO resin on the physical and mechanical properties of the UPE/EPO resin were evaluated by water absorption, tensile, flexural and impact test. The thermal and thermophysical behaviour of the UPE/EPO were also evaluated by using thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA) test. Lastly, the morphology of the UPE/EPO blend was evaluated using scanning electron microscope (SEM) test.

The second part of the study, MMT filler was added into the UPE/EPO resin system as reinforcement. The UPE/EPO/MMT resin were prepared with various amounts of MMT (1, 1.5 and 2 phr). The physical and mechanical properties of the UPE/EPO/MMT resin were evaluated by water absorption, tensile, flexural and impact test. The thermal and thermophysical properties of the UPE/EPO/MMT resin were characterised using thermogravimetric analysis (TGA) and dynamic mechanical analysis (DMA) test. The distribution of the MMT nanoclay filler in the system was also being evaluated by using scanning electron microscope (SEM) and transmission electron microscopy (TEM) test. The best UPE/EPO/MMT resin formulations was chosen by selecting the best stiffness-toughness balanced properties of the new fabricated UPE/EPO/MMT resin for further use in the preparation of kenaf fibre reinforcement composite.

The third part of the study, the kenaf fibre reinforced UPE/EPO/MMT composite was prepared by using resin transfer moulding (RTM) processing technique at different mould angle (0º, 30º and 45º). The mechanical properties of the kenaf fibre reinforced UPE/EPO/MMT composite at different RTM mould angles were evaluated by determining tensile, flexural and impact properties. The thermophysical and morphology properties of kenaf fibre reinforced UPE/EPO/MMT composite at different RTM mould angles were also characterised by using dynamic mechanical analysis (DMA) and scanning electron microscope (SEM). Finally, the conclusion has been made on the best RTM mould angle for kenaf fibre reinforced UPE/EPO/MMT composites.

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