MECHANICAL PROPERTIES OF HYBRID RECYCLATE GLASS FIBRE - MONTMORILLONITE NANOFILLER REINFORCED POLYMER NANOCOMPOSITE

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DEDICATION

This thesis is dedicated to my father, who taught me that the best kind of knowledge to have is that which is learned for its own sake. It is also dedicated to my mother, who taught me that even the largest task can be accomplished if it is done one step at a time.

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ABSTRACT

Glass fibre reinforced polymer (GFRP) waste quantity is reported to increase every year and contributing to the majority of composites waste. The awareness for a greener world has led to the recycling of GFRP waste. Previous studies focus on extracting the recycled fibres from GFRP waste and reintroducing the reclaimed fibres back into various types of polymer matrix to produce new composite materials. However, there is a lack of study conducted on the potential use of recycled fibres hybridized with nanoclay. This research highlights on recycled glass fibre (rGF) as a potential reinforcement in polymer composite, as green alternative to virgin glass fibre. The aim of this study was to investigate the effect of montmorillonite (MMT) nanoclay on the mechanical properties of rGF – unsaturated polyester (UP) composites. The selected parameters were rGF fibre sizes and loading, MMT weight percentage and the hybridization effect of rGF with MMT. The grades of rGF were raw (unsieved), fine and coarse, while the fibre loading ranges were from 5% to 45% by weight. Various MMT nanoclay weight percentage ranges from 0.5% to 7% were studied. The rGF samples were prepared using mechanical grinding, prior being fabricated by hand lay-up and followed by compression moulding. The effects of varying experimental parameters were observed in composites tensile, flexural, and compression properties. Mathematical formulae for every mechanical properties including all interaction factors were developed. The results from tensile and flexural tests revealed that rGF size was the most significant factor influencing its strength. The tensile strength of 30% coarse rGF was the highest among all samples while coarse rGF at 35% fibre loading showed the best flexural strength. For hybrid rGF-MMT composites, tensile strength showed improvement at 0.5% MMT inclusion. The flexural test demonstrated that the inclusion of rGF increases the flexural modulus by approximately 300% compared to pure UP. The addition of rGF and MMT was also improved the compression properties of composites by about 200% compared to pure UP. The morphology analysis showed that a good adhesion between rGF-resin bonding was observed at low MMT percentage. Transmission electron microscopy of UP-MMT nanocomposites showed that at 1 wt.% MMT, the nanoclay is well dispersed. Based on the results, hybridization with MMT nanoclay can be a green alternative solution to improve mechanical properties of rGF-UP composites and at the same time providing recycling solution to GFRP waste.

ABSTRAK

Kuantiti sisa polimer bertetulang gentian kaca (GFRP) dilaporkan meningkat setiap tahun dan menyumbang kepada sebahagian besar sisa komposit. Kesedaran terhadap kelastrian dunia telah mendorong aktiviti kitar semula sisa buangan FRP. Kajian- kajian terdahulu memberi fokus pada pengekstrakan gentian kitar semula dari sisa buangan FRP dan gentian tersebut akan dimasukkan semula ke dalam matriks polimer untuk menghasilkan bahan komposit baru. Walau bagaimanapun, kajian yang sangat kurang dilakukan mengenai potensi penggunaan gentian kitar semula yang dihibridisasi dengan tanah liat nano. Penyelidikan ini memberi tumpuan terhadap gentian kaca yang dikitar semula (rGF) sebagai penguat berpotensi dalam komposit polimer sebagai alternatif hijau untuk gentian kaca tulin. Tujuan kajian ini adalah untuk mengkaji kesan tanah liat nano montmorillonite (MMT) terhadap sifat mekanik komposit rGF - poliester tak tepu (UP). Parameter yang dipilih adalah ukuran dan peratusan berat gentian rGF, peratusan berat MMT dan kesan hibrid rGF dengan MMT. Gred rGF terdiri daripada mentah (tidak diayak), halus dan kasar manakala peratusan berat gentian yang dikaji adalah antara 5% hingga 45% berat. Pelbagai peratusan berat tanah liat nano MMT antara 0.5% hingga 7% telah dikaji. Untuk penyediaan sampel, rGF telah dikisar secara mekanikal, sebelum difabrikasi dengan teknik bengkalai tangan dan diikuti dengan kaedah pengacuanan mampatan. Pelbagai parameter eksperimen telah dianalisa terhadap sifat tegangan, lenturan, dan mampatan komposit. Formula matematik pada setiap sifat mekanikal yang dibangunkan meliputi kesemua faktor interaksi. Hasil dari ujian tegangan dan lenturan menunjukkan bahawa saiz rGF adalah faktor yang paling bererti yang mempengaruhi kekuatannya. Kekuatan tegangan bagi 30% rGF kasar adalah tertinggi di antara semua sampel sementara rGF kasar pada 35% serat menunjukkan kekuatan lenturan terbaik. Untuk komposit hibrid rGF-MMT, kekuatan tegangan menunjukkan peningkatan pada kemasukan 0.5% MMT. Ujian lenturan menunjukkan bahawa penambahan rGF meningkatkan modulus lenturan sebanyak 300% berbanding UP tulen. Untuk hasil ujian mampatan, penambahan rGF dan MMT meningkatkan sifat mampatan komposit sebanyak 200% berbanding UP. Analisis morfologi menunjukkan bahawa MMT pada peratusan rendah menghasilkan rekatan yang baik antara rGF-resin. Mikroskopi elektron transmissi bagi kompositnano UP-MMT menunjukkan bahawa pada 1 wt.% MMT, tanah liat nano menyerak dengan baik. Berdasarkan hasil kajian, hibridisasi dengan tanah liat nano MMT boleh menjadi penyelesaian alternatif hijau untuk meningkatkan sifat mekanik komposit rGF-UP dan pada masa yang sama memberikan penyelesaian kitar semula kepada sisa GFRP.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
CrGF	-	Coarse rGF-UP Composites
EoL	-	End-of-Life
EDX	-	Energy Dispersive X-ray Analysis
EuCIA	-	European Composites Industry Association
FRP	-	Fibre Reinforced Polymer
FrGF	-	Fine rGF-UP Composites
GFRP	-	Glass Fibre Reinforced Polymer
MEKP	-	Methyl Ethyl Ketone Peroxide
MMT	-	Montmorillonite
PHR	-	Part per Hundred Resin
PMC	-	Polymer Matrix Composite
PNC	-	Polymeric Nano-Composites
rGF	-	Recycled Glass Fibre / Recyclate
rGFRP	-	Recycled Glass Fibre Reinforced Polymer
rGFUP	-	Recycled Glass Fibre Unsaturated Polyester
ROM	-	Rule of Mixture
SEM	-	Scanning Electron Microscope
SMC	-	Sheet Moulding Compound
TGA	-	Thermogravimetric Analysis
TEM	-	Transmission Electron Microscope
UP	-	Unsaturated Polyester
XRD	-	X-ray Diffractometer

LIST OF SYMBOLS

ε	-	Normal strain
σ_{Com}	-	Tensile strength of composite
σ_{cl}	-	Tensile strength of MMT clay
σ_{fib}	-	Tensile strength of fibre
σ_{m}	-	Tensile strength of matrix
σ_{nc}	-	Tensile strength of nanocomposite
E_{com}	-	Tensile modulus of composites
E_{fib}	-	Tensile modulus of fibre
E_{m}	-	Tensile modulus of matrix
E_{nc}	-	Tensile modulus of nanocomposites
M_{f}	-	Mass of glass fibre recyclate
M_{m}	-	Mass of matrix
v_{c}	-	Volume of composite
v_{cl}	-	Volume fraction of MMT clay
v_{f}	-	Volume of fibres
v_{m}	-	Volume of matrix
W_{f}	-	Weight of fibre
W_{m}	-	Weight of matrix
W_{MMT}	-	Weight percentage of montmorillonite nanoclay
W_{rGF}		Weight percentage of recycled glass fibres
W_{UP}	-	Weight percentage of unsaturated polyester
ρ_{f}	-	Density of glass fibre recyclate
ρ_{m}	-	Density of matrix
$\Delta\forall$	-	Change of water level
P	-	Force
ζ	-	Shape factor of MMT nanoclay
σ	-	Tensile strength
E_{C}	-	Compressive modulus
σ_{C}	-	Compressive strength

σ_f	-	Flexural strength
E_f	-	Flexural modulus
A	-	Cross section area
E	-	Tensile modulus
L	-	Support span
k	-	Coefficient for types of recyclates
l	-	Fibre length
V	-	Volume fraction of fibre

LIST OF APPENDICES

CHAPTER 1

INTRODUCTION

1.1 Research Background

Fibre reinforced polymer (FRP) composites are unique because they offer engineers and designers the flexibility in selecting the wide selection of reinforcing fibres, fillers, additives, binding resins and manufacturing process to produce an engineered material with desired definitive specifications (Gibson, 2016). The main advantages of using FRP composites over other conventional materials are high strength-to-weight ratios, non-conductive, non-corrosive, non-magnetic, good electrical insulation and wear resistant (Vinson and Sierakowski, 2012).

Every year, it is projected that over six megatons of plastic composites are produced worldwide, mostly consists of glass fibre reinforced polymers (GFRP). It is estimated that about 1 million tons of GFRP produced annually in Europe (Nash et al., 2019). In United Kingdom, the GFRP production represents about 140,000 tonnes, while the carbon fibre composite production represents around only approximately 2,000 tonnes. It was estimated one billion tonnes of GFRP waste is generated in Europe annually and the amounts of GFRP waste are increasing yearly (Rybicka et al., 2016).

Increased GFRPs usage has led to mounting pressure to resolve issues relating to composite waste (Abu Hassan et al., 2016). For instance, GFRP sheet moulding compound (SMCs), is one of the most commonly used composite materials, its total waste generates 0.4 million tonnes annually. Composites materials from glass fibres and carbon fibres are relatively new in commercial usage and therefore the current composites recycling technologies are still in development stage and requires further research. Environmental factors are seen to be probably the most critical element affecting the composites industries, with the issues of recycling having the greatest impact.

Composites made from thermoset polymer resins such as epoxies and unsaturated polyesters are technically challenging to recycle since the thermoset polymer matrixes are cross-linked and cannot be re-melted like thermoplastic polymers. There are many recycling options available for FRP composites waste include mechanical grinding, fluidised-bed processing, pyrolysis and supercritical water processing (Meng et al., 2017; Naqvi et al., 2018; Kim et al., 2019; Shuaib and Mativenga, 2016). Most of the technique has been demonstrated inside the laboratory except for mechanical recycling which has been commercialized (Mamanpush et al., 2019; Yazdanbakhsh et al., 2018).

The strength of recycled glass fibres is lower compared to virgin glass fibres (Rouholamin et al., 2014). The deterioration of recycled fibres is one of the major factors that discourage FRP manufacturers to recycle or reuse the composites scraps. Recent studies show that hybrid glass fibre composites that uses nanofillers such as montmorillonite (MMT) nanoclay can improve the mechanical properties of composites (Sagar and Palanikumar, 2016; Prabhu et. al., 2019). The addition of MMT nanoclay could possibly give the same positive result for recycled glass fibre composites.

The research on nanoclays fillers in polymer nanocomposites has attracted considerable interest due to its many potential improvement in thermo-mechanical properties, fire and gas resistance of the developed nanocomposites. The nanoclay fillers has excellent dispersion in the polymer matrix at nanometer scale and high surface-to-volume ratio which resulted in improvement of mechanical and physical properties of nanocomposites as compared to the pure polymers (Müller et al., 2017; Laatar et al., 2016).

Previous studies focus on the technology of recycling and extracting the fibres from FRP waste. The reclaimed fibres were added back to various types of polymer matrix and produced into a new composites material. In this study, the reclaim fibres will not only be reintroduced into new polymer matrix but will also be hybridize with nanoclay. The effect of nanoclay hybridization with glass fibre recyclates polymer composites will be evaluated by its quasi-static mechanical properties such as tensile and flexural properties. Compression properties of glass fibre recyclates polymer composites will also be highlighted in this study, as not many research has been done on the compression properties of glass fibre recyclate composites.

1.2 Problem Statement

Abundant usage of GFRP applications has generated large amount of waste which ends up in landfills. A sustainable recycling mechanism is critically necessary to effectively recycle the GFRP waste. From past studies, mechanical recycling technology of GFRP has shown to be an eco-friendly method compared to thermal and chemical recycling technologies. However, the recovered glass fibres from mechanical recycling of GFRP only maintain half of its original tensile strength compared to virgin glass fibre (Rouholamin et al., 2014; Palmer 2009). Reintroducing the recovered fibres into composites resulted in inferior mechanical properties.

Understanding the characteristics of the GFRP recyclates is essential in determining the composites mechanical properties. Characteristic of GFRP waste from septic tanks fabrication scraps has not been investigated. The effect of raw recyclates at various fibre loading on the tensile and flexural properties of recyclate reinforced unsaturated polyester composite need to be establish.

Raw GFRP recyclates tends to include powder particles and impurities which inhibit good composite fabrication. The raw recyclates need to be sieved to remove the impurities and powder particles and to grade the fibrous recyclate form into different fibre length grades. The effect of sieved GFRP recyclates at different fibre length and various fibre loading percentages in unsaturated polyester composite on tensile and flexural properties need to be determined.

The high surface-to-volume ratio of montmorillonite (MMT) nanoclay among other nanoparticles geometries has the potential to improve the mechanical and physical properties of nanocomposites. However, the optimal percentage of MMT loading in unsaturated polyester nanocomposites need to be identified as the dispersion of MMT in polymer plays a major role in influencing the mechanical properties of nanocomposites. Therefore, inclusion of MMT loading ranging from 0.5 wt.% to 7 wt.% need to be study in determining optimal MMT loading for tensile and flexural properties of unsaturated polyester nanocomposite.

Based on a previous study (Prabhu et. al., 2019), the incorporation of MMT can improve the mechanical properties of virgin glass fibre reinforced composites. The hybridization of MMT nanofillers with recycled glass fibres can potentially overcome the degraded strength of recycled glass fibres reinforced polymer composites. The combination effect of MMT and GFRP recyclates at various percentages on tensile and flexural properties of hybrid composites requires further evaluation.

Lack of studies was found regarding the compression properties of GFRP recyclates composites. Pure unsaturated polyester polymer composites have low compression strength and modulus reading. Most studies combine the use of sand and aggregates to improve the compression performance of polyester polymer composites but lack of studies was performed using recyclates exclusively with polyester polymer composites. The effect of sieved GFRP recyclates at different fibre length and various fibre loading percentages in sieved GFRP unsaturated polyester composite on compression properties requires further investigation. Investigation on MMT reinforced polyester nanocomposite need to be done using various MMT loading. The combined effects of GFRP recyclates and nanoclay at various percentages on compression of hybrid composites need to be assessed.

1.3 Objectives of Research Project

In the light of the above, the aim of this project is to investigate the potential use of montmorillonite nanoclay in order to enhance the mechanical properties of recycled glass fibre reinforced unsaturated polyester composites. The specific objectives are:

1. To determine the physical characteristic of raw glass fibre recyclates (rGF) and its effect at different fibre loading on the tensile and flexural properties of raw rGF reinforced unsaturated polyester composites.

2. To determine the effect of different fibre sizes and different fibre percentages of sieved rGF on the tensile and flexural properties of sieved rGF reinforced unsaturated polyester composites.
3. To investigate the effect of MMT nanoclay at different percentages on the tensile and flexural properties of MMT nanoclay reinforced unsaturated polyester nanocomposites.
4. To evaluate the hybridization effect of MMT nanoclay percentages on the tensile and flexural properties of sieved rGF reinforced unsaturated polyester composites.
5. To evaluate the effect of rGF fibre percentages, rGF fibres sizes and the hybridization effect of MMT percentages on the compression properties of sieved rGF reinforced unsaturated polyester composites.

1.4 Scope of Research

This thesis covers the topic of polymer composites focusing on the issue of composite recycling and sustainable composite materials. The study emphasized on the recycling of glass fibre reinforced polyester (GFRP) waste specifically from the scraps of septic tanks fabrication. The GFRP was recycled using mechanical recycling method. The recycled GFRP (rGF) was reintroduced in a new composite at different fibre size grades. The type of matrix selected for this study is limited to unsaturated polyester resin. The mechanical performance of the test samples was evaluated under three different types of testing which are tensile, flexural and compression test. The parameters that were studied are fibre size and fibre loading of the rGF. The mechanical tests provide data regarding the tensile properties, stress versus strain curve graph, flexural properties, compression properties and compression stress versus compression strain curves of the rGF reinforced UP composites samples. Morphology study on the test samples was perform using scanning electron microscope (SEM) to observe the interfacial bonding between rGF and polymer resin. Data such as fibre distribution, fibre breakage, fibre-matrix debonding, fibre pull-out, matrix cracking and porosity of the composites was observed.

Polymer nanocomposite was studied specifically on the use of montmorillonite nanoclay reinforced unsaturated polyester (UP-MMT) composites. The prepared composite samples were tested for tensile, flexural and compression properties which generate data on tensile strength, Young's modulus, tensile strain, stress versus strain curve graph, flexural strength, flexural modulus, compression strength, compression modulus and compression stress versus compression strain of the UP-MMT nanocomposites material. These data were analysed based on the MMT weight percentages. Mathematical model using Halpin-Tsai equation was produced based on the plotted experimental tensile and compression data. Transmission electron microscope (TEM) and X-ray diffraction (XRD) were used to provide information regarding MMT nanoclay distribution in UP-MMT nanocomposites samples.

Hybrid polymer nanocomposites to be studied in this thesis include the use of rGF and MMT nanoclay. The samples of rGF and MMT were produced after the optimal fibre percentage and MMT loading. The prepared hybrid nanocomposite samples were tested for tensile, flexural and compression properties which generate data on tensile strength, Young's modulus, tensile strain, stress versus strain curve graph, flexural strength, flexural modulus and compression strength, compression modulus and compression stress versus compression strain curve of the hybrid rGF-MMT nanocomposites material. Mathematical model using modified Halpin-Tsai equation were produced based on the plotted experimental tensile and compression data.SEM image was used to observe the interfacial bonding between rGF and UP resin at different MMT percentage.

1.5 Significance of Research

The hybridization of montmorillonite nanofillers with recycled glass fibre reinforced composites has the potential to increase the mechanical properties of the composites. The data on tensile, flexural and compression properties of recycled GFRP composites hybridized with nanoclay can be used by engineers and other researchers to develop a useful product and further investigates potential application of recycled GFRP. Mathematical model on the mechanical properties of recycled GFRP-MMT hybrid composites in relation to fibre loading that was produced by this study can be used by other researcher as a guideline. In general, a practical composite product developed based from this study will encourage FRP manufacturer to involve in composites recycling rather than landfilling or incinerating their FRP waste. This study will indirectly promote the recycling of GFRP scraps, reuse of GFRP scraps and reducing the consumption of new glass fibre.

1.6 Thesis Framework

This thesis is divided into five major chapters. The first chapter will present the overall background of the study, problem statement, research objectives, scope and the significance of this research. Chapter 2 discussed the literature review based on previous studies of related topics including GFRP and its waste management and recycling technologies. The literature review includes past studies which use recycled GFRP in composites. Polymer nanocomposites and its hybrid with conventional fibres were reviewed in this chapter. Chapter 3 elaborated on the research methodology. Materials properties used in this study, sample formulations, sample fabrication, testing standards and analysis method was highlighted in this chapter. Chapter 4 reported the experimental data on tensile, flexural and compression properties. Mechanical test results analysis was supported by microstructure analysis. The mathematical models generated based on the composites parameters studied. Chapter 5 concluded the research findings based on research objectives and recommendation for future research to be explored.

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2. **Umar Abdul Hanan**, Shukur Abu Hassan, Mat Uzir Wahit, Ahmad Raizal Md Rozali, Hj. Kamarul Bahrin Mohamad and Mohd Arif Mat Lazi (2016), "Mechanical properties of glass fibre waste/kenaf core reinforced unsaturated polyester eco-friendly composites." Journal of Built Environment, Technology and Engineering, 1: 273-281.

3. **Umar Abdul Hanan**, Shukur Abu Hassan, Mat Uzir Wahit and Siti Khalijah Jamal (2016), "Mechanical Recycling of GFRP Waste as Reinforcement in Unsaturated Polyester Composites." In: 6thInternational Graduate Conference on Engineering, Science and Humanities (IGCESH). Johor Bahru.

4. Shukur Abu Hassan, Mat UzirWahit, **Umar Abdul Hanan** and Siti Khalijah Jamal (2017), "Tensile Properties of Hybrid Glass Fibre Recyclate-Montmorillonite Nanofiller Composites." In Wong King Jye and Zaini Ahmad (Ed.), Structural Composites: Mechanical Properties and Behaviour (pp. 67-81). Johor Bahru:UTM.

5. Behzad Abdi, Shukur Abu Hassan, Mat Uzir Wahit and **Umar Abdul Hanan** (2014), "Tensile and Flexural Properties of Recycled Glass Fibre Reinforced Polyester Composites." Materials Research Innovations Vol. 18 Suppl. 6 pp.S6-1-S6-3. (Indexed in Scopus)