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EFFECT OF NANOCLAY AND SILICA AEROGEL ON MECHANICAL, THERMAL AND PHYSICAL PROPERTIES OF SUGAR PALM FIBREREINFORCED UNSATURATED POLYESTER COMPOSITES

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By

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September 2018

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With the increasing rate of technological developments, the use of specialized materials, such as composites, is continuously increasing. Conventional composites are prepared from synthetic raw materials which are proving to be hazardous to the environment and measures need to be taken to reduce their effects on the environment. Reinforcing natural fibres in composites is a potentially viable solution to this problem and can result in enhanced properties as well. Sugar palm fibres are also anticipated to be used in a similar scope, however, there is a need for improvement in properties before they can be successfully employed in relevant applications. This study focused on enhancing the usability of Sugar palm fibre in polymer composite with the addition of fillers. This study investigated the effect of filler loading on sugar palm fibre reinforced unsaturated polyester composite. Naturally existing woven sugar palm fibres were used in this study as reinforcement and unsaturated polyester was used as the matrix. Nano-fillers namely, nanoclay and silica aerogel were infused in the sugar palm fibre reinforced polyester composite. Composites were prepared with hand layup process followed by the hydraulic hot press, which pressed the mould for 30 minutes at 80 °C. Different weight percentages of nanoclay content (NC) and silica aerogel content (SAC) were used to prepare the composite. The filler loadings of 0, 1, 2, 3, 4 and 5 % were used for both the fillers. Mechanical, thermal and physical tests were performed on the fabricated composites and the results were analysed. Tensile, flexural and impact tests were performed according to respective ASTM standards. Almost all the composites with infused fillers showed higher tensile strength and 4 % NC mixed composite showed the highest tensile strength and modulus improvement of 58 % and 12 % respectively. Flexural properties of nanoclay based composites also showed higher improvement compared to silica aerogel based composites. A 54 % increase in flexural strength and 42 % maximum increase in flexural modulus was achieved with the addition of nano-fillers compared to composite without any filler. Both nanoclay and silica aerogel were able to achieve better and similar impact strength but at different filler loading. 2 % for nanoclay and 3 % for silica aerogel achieved almost 20 % improvement in impact strength compared to 0 % composite. SEM images of the impact fracture composite were discussed and analysed. For thermal analysis, dynamic mechanical analysis (DMA) and thermogravimetric analysis (TGA) were performed. Addition of both the fillers improved the dynamic mechanical and thermal properties of the composite. The optimum concentration for both the fillers to achieve the best thermal performance was found to be between 2 % to 3 %. To determine the physical properties, water absorption and thickness swelling investigation of the composites were carried out. The composites were immersed in distilled water for up to 13 weeks to analyse long-term water absorption and thickness swelling characteristics of the composites. Composites with nanoclay restricted and reduced the moisture uptake and dimensional deformation when immersed in water while silica aerogel infused composites showed higher levels of water absorption and thickness swelling compared to composites without additives. This study concluded that the addition of studied fillers can significantly improve the properties of sugar palm reinforced composites, making it more usable for a broader range of tertiary applications, where currently conventional composites are employed.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

KESAN NANOCLAY DAN SILIKA AIRGEL PADA SIFAT MEKANIKAL, HABA DAN FIZIKAL PADA KOMPOSIT - KOMPOSIT GENTIAN ENAU DIPERKUAT POLIESTER TAK TEPU

Oleh

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Dengan perkembangan teknologi yang semakin pesat, penggunaan bahan khusus seperti komposit juga semakin meningkat. Komposit konvensional diperbuat daripada bahan mentah sintetik terbukti berbahaya kepada alam sekitar. Oleh itu, langkah-langkah perlu diambil untuk mengurangkan kesannya terhadap alam sekitar. Menguatkan serat semulajadi dalam komposit adalah penyelesaian yang berpotensi untuk menyelesaikan masalah ini dan boleh menghasilkan sifat yang dipertingkatkan juga. Gentian enau juga dijangka digunakan dalam skop yang sama. Walaubagaimanapun, terdapat keperluan untuk meningkatkan sifat sebelum mereka dapat digunakan dengan berkesan dalam aplikasi yang relevan. Kajian ini memberi tumpuan kepada peningkatan kegunaan gentian enau dalam komposit polimer dengan penambahan pengisi. Kajian ini menyiasat tentang kesan pemuatan nano filler pada komposit gentian enau diperkuat poliester tak tepu. Gentian semulajadi yang ditenun dengan semulajadi telah digunakan dalam kajian ini sebagai penguat dan poliester tak tepu yang digunakan sebagai matriks. Pengisi Nano iaitu, nanoclay dan silika airgel diselitkan dalam komposit gentian enau diperkuat poliester tak tepu. Komposit disediakan dengan proses tangan diikuti oleh mesin menekan panas hidraulik yang menekan acuan selama 30 minit pada suhu 80°C. Peratusan berat kandungan kandungan nanoclay (NC) dan silika airgel (SAC) yang berbeza digunakan untuk menyediakan komposit. Muatan 0, 1, 2, 3, 4 dan 5 % telah digunakan untuk kedua-dua pengisi. Ujian mekanikal, haba dan fizikal dilakukan pada komposit dan hasilnya dianalisa. Ujian tegangan, lenturan dan kesan mekanikal dilakukan mengikut piawaian ASTM masing-masing. Hampir kesemua komposit dengan pengisi menunjukkan kekuatan tegangan yang lebih tinggi. Campuran komposit NC 4 % menunjukkan kekuatan tegangan tertinggi dan peningkatan modulus sebanyak 58 % dan 12 % masing-masing. Ciri-ciri fleksural komposit berasaskan nanoclay juga menunjukkan peningkatan yang lebih tinggi berbanding komposit berasaskan silika airgel. Peningkatan kekuatan lenturan sebanyak 54 % dan peningkatan maksimum 42 % dalam modulus flexural dicapai dengan penambahan pengisi nano berbanding komposit tanpa pengisi. Kedua-dua nanoclay dan silika airgel dapat mencapai kesan kekuatan yang lebih baik dan serupa tetapi pada pemuatan pengisi yang berbeza. 2 % untuk nanoclay dan 3 % untuk silika airgel mencapai hampir 20 % peningkatan dalam kekuatan kesan berbanding komposit 0 %. Imej SEM komposit akibat ujian fraktur dianalisa. Untuk analisis haba, ujian analisis mekanikal dinamik (DMA) dan analisis termogravimetrik (TGA) telah dilakukan. Penambahan kedua-dua pengisi mampu meningkatkan kestabilan haba komposit. Kepekatan optimum bagi kedua-dua pengisi untuk mencapai prestasi yang terbaik adalah antara 2 % hingga 3 %. Penyiasatan bengkak terhadap komposit telah dijalankan bagi menentukan sifat-sifat fizikal, penyerapan air dan ketebalan. Komposit telah direndam dalam air suling selama 13 minggu untuk menganalisis penyerapan air jangka panjang dan ciri bengkak komposit. Kadar peningkatan komposit dengan nanoclay adalah terhad dan telah mengurangkan pengambilan kelembapan dan ubah bentuk dimensi apabila direndam dalam air. Manakala silika airgel digabungkan komposit menunjukkan tahap penyerapan air dan ketebalan yang lebih tinggi berbanding dengan komposit tanpa tambahan. Kajian ini menyimpulkan bahawa penambahan pengisi yang dikaji dengan ketara dapat meningkatkan sifat-sifat komposit diperkuat gentian enau menjadikannya lebih berguna untuk pelbagai aplikasi yang lebih luas.

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I certify that a Thesis Examination Committee has met on 3 September 2018 to conduct the final examination of Rao Muhammad Shahroze Ali on his thesis entitled "Effect of Nanoclay and Silica Aerogel on Mechanical, Thermal and Physical Properties of Sugar Palm Fibre-Reinforced Unsaturated Polyester Composites" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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This is to confirm that:

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LIST OF ABBREVIATIONS

Al	Aluminium
ASTM	American Society for Testing and Materials
AT	Alkaline treatment
Ca	Calcium
CNT	Carbon nanotube
DMA	Dynamic mechanical analysis
FM	Flexural modulus
FRP	Fibre reinforced plastics
FS	Flexural strength
GPa	Giga Pascal
GFRC	Glass Fibre Reinforced Composites
HIPS	High impact polystyrene
ISO	International Organization for Standardization
K	Potassium
MMT	Montmorillonite
MPa	Mega Pascal
Na	Sodium
NaOH	Sodium hydroxide
NC	Nanoclay content
OMMT	Organo montmorillonite
UPE	Polyethylene
Phc	Per hundred compounds
PP	Polypropylene
SAC	Silica aerogel content
SEM	Scanning electron microscopy
SF	Sugar palm frond
SM	Storage modulus
SPB	Sugar palm bunch
SPF	Sugar palm fibre
SPT	Sugar palm trunk
ST	Sea water treatment
Tan δ	Tan delta
TEOS	Tetraethyl orthosilicate
Tg	Glass transition temperature
TGA	Thermogravimetric analysis
TKS	Thickness swelling

TM	Tensile modulus
TPU	Thermoplastic polyurethane
TS	Tensile strength
UPE	Unsaturated polyester
WA	Water absorption
wt. %	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

With such rapid progress in research and technology, there is a constant increase in the need to satisfy the material requirements of such advancements. Composite materials are an engineering necessity to cater to these needs. A composite is formed by the combination of two or more different kinds of materials and provides a wide range of properties to suit a specific requirement better. A polymer composite usually consists of two components, namely polymeric matrix/binder and reinforcements/fibres. Both of these parts contain separate individual properties and result in an entirely different combined set of features. Usually, a composite includes a higher quantity of matrix in comparison to the fibres. The matrix helps to bind the fibres together and protects them from the environment as well. The reinforcements aid to enhance the material properties of the matrix since they are typically stronger, more robust than the binder (Razak & Kalam, 2012). In general, any isotropic material can be used as a reinforcement (Krevelen et al., 2009).

Rising awareness about the environmental impacts of using conventional composites derived from non-renewable resources has shifted the focus to search and produce composites using raw materials acquired from the renewable sources. This awareness has led to an increase in the use of natural fibres as reinforcements in polymer composites to reduce the usage of its non-biodegradable equivalent. Various wasted biodegradable materials such as wood chips, plant fibres, newspapers, etc., are now being used as reinforcements. The use of these materials also helps to reduce the need for raw materials and also contributes to solving the waste management problems (Saba et al., 2016a). Natural fibres offer a wide range of advantages including low density, less wearing from tools, acoustic insulation, lower cost and biodegradability (Haameem et al., 2016).

Properties of the polymers can be improved by using additives to obtain the desired application-specific properties. Nano-fillers, which are advanced additives, are also used effectively as nano-additives for further refining the properties of the polymers and the composites. For the past two decades, nanoscience has been in the limelight amongst researchers throughout the world, to better understand its benefits and applications. A few commercially available nano-fillers include carbon-nanotubes (CNT), layered silicates, Polyoctahedral silsesquioxane and graphite nanoflakes. In general, a nanocomposite is a combination of a polymer and a nano-filler. To classify as nano-sized, a filler must have at least one of its dimensions equal or less than 100 nm. Nanocomposites follow the concept of using nanoparticles for achieving maximum interfacial bonding between the matrix and the reinforcements. They can offer a wide range of functional properties because of their large surface area and aspect ratio. Nano-fillers are known to improve the mechanical, thermal and dimensional stability of the composites.

Hybrid composites are a step forward in the field of composites. In principle, a composite containing two or more reinforcement can be referred to as a hybrid composite (Saba et al., 2016a). Hybrid composites can further improve the balance between price and performance compared to the conventional composites. Hybrid composites, in which at least one of the constituent is nano-sized, are generally known as hybrid nanocomposites. Various researchers investigated the production of hybrid nanocomposites and found that the addition of a nano-component can significantly enhance the overall properties of a natural fibre reinforced polymeric composite.

1.2 Problem Statement

Although there are some advantages of using natural fibres, there are some disadvantages linked to it as well, such as weak fibre-matrix bonding characteristics and high water absorption tendency of the natural fibres, which can affect the mechanical and thermal properties of the natural fibre based composites. However, doing some modifications in the reinforcement or the matrix can significantly reduce the degree of these drawbacks. This will make these composite more competitive to synthetic fibre reinforced composites. To employ this modification principle, a number of studies were performed to explore the potential of sugar palm fibres in composites. In comparison, sugar palm fibres (SPF) were observed to have competitive mechanical and physical properties to other natural fibres such as palmyrah fibres, kenaf fibres and coconut fibre. However, there is a need for improvement within SPF composites to achieve better performance that is close to some high-end natural fibre and synthetic fibre reinforced composites. The studies focusing on modification behaviours usually modify the fibre to make it more compatible with the resin. These processes, however, can be tedious at times and may require extensive efforts to achieve the final product. Investigating a methodology that requires lesser procedural steps in comparison to the existing research for obtaining similar or better improvement in the properties of SPF based composites is the driving point for this work.

It is well known that fibre surface treatments, hybridization and adding fillers/additives are measures to improve the properties of natural fibre reinforced composites. Among these techniques, hybridization and fibre surface treatments such as alkali, acetylation and seawater treatments were explored by previous researchers on SPF based composites. The main focus of the existing literature, on the enhancement of sugar palm fibres reinforced composites, has been on modifying the physical characteristics of sugar palm fibres with various fibre treatments. However, no studies were found on the principle of altering the matrix to make it more compatible with the fibres. More specifically, studies on the effect of introducing fillers into the SPF reinforced composites remain unexplored.

Addition of fillers in the matrix is aimed to enhance the compatibility of the matrix with the fibres so that a better balance of stress transferability, physical and thermal properties can be achieved between the matrix and the reinforcements. Fillers were chosen over other techniques because some of the fillers are less expensive and can improve the features of a composite by controlling viscosity and reduce mould shrinkage, making it a cost-effective approach and user-friendly technique. Adding a minimal quantity of these filler has shown to improve the properties of other natural fibre based composites such as composites comprising of kenaf, sisal, jute and more fibres. This study will focus on exploring the enhancement of properties with the addition of fillers namely nanoclay and silica aerogel in sugar palm fibre composite. Addition of these fillers in excess may lead to a decline in the performance of the composites. So, it is necessary to identify the optimum weight % required to obtain enhanced performance and study its influence on the microstructure of the SPF composites.

1.3 Research Objectives

This study aims to experimentally identify the changes in the properties of sugar palm fibre reinforced unsaturated polyester (SPF/UPE) composite with adding various concentrations of nanoclay and silica aerogel as additives. There are three specific objectives of this study, which are listed as follows:

(a) To determine the effect of adding different concentrations of nanoclay and silica aerogel on the mechanical properties of SPF/UPE composite.

(b) To examine the influence of adding different concentrations of nanoclay and silica aerogel on the thermal properties of SPF/UPE composite.

(c) To identify the impact of adding different concentrations of nanoclay and silica aerogel on the physical properties of SPF/UPE composite.

1.4 Scope of Study

This study was conducted to obtain the enhanced properties of natural fibre reinforced polymer composites, so the use of natural fibres, more specifically sugar palm fibres, can be maximised in composites, leading to an overall reduced usage of synthetic raw materials in the same scope. This study will focus on enhancing the mechanical, thermal and water absorption properties of sugar palm fibres reinforced composites with the use of nanoclay and silica aerogel only. This study is limited to improving the composites for use in tertiary applications where the strength is not a critical factor such as furniture, false ceiling, roofing, etc.

In this study, only one kind of sugar palm fibres was utilised; they were obtained from trees which were at flowering stage. In general, the sugar palm trees with the age of six years and above reach the stage of flowering and are considered as matured trees. This kind of tree provides the best quality of fibres, and hence, these fibres were used in this study. As this study is intended to do the characterisation of composites for use in tertiary structures, unsaturated polyester was employed as the matrix for the composites as it provides the best balance of properties and cost in comparison to other thermoset resins. The effect of adding different kinds of fillers was investigated, to maximise the usability of sugar palm fibres as reinforcements in the unsaturated polyester matrix. The enhancements achieved in this study will facilitate the utilisation of sugar palm fibres on a broader range of application, making productive use of otherwise wasted sugar palm fibres.

Fibres in the form of a naturally woven structure were prepared as reinforcement in the composite. The unsaturated polyester resin was used as the base matrix in the composite, and methyl ethyl ketone peroxide was used as a curing agent to cure the composite. Two kinds of additives, namely nanoclay and silica aerogel, were used in this study at various concentrations and mixed in the resin. The control specimen was prepared without addition of any additives in the matrix while investigated composites were made with various weight percentages of nanoclay and silica aerogel infused in unsaturated polyester resin. The effect of the addition of these additives on mechanical properties, such as tensile, flexural and impact properties, were determined by performing the respective tests as per ASTM standards. The surface morphology of the impact fractured specimen was analysed using SEM. The thermal behaviour of the same composites was studied using dynamic mechanical analysis and thermogravimetric analysis. The composites were also subjected to water immersion of up to 13 weeks, and the water absorption and thickness swelling behaviour were recorded and analysed.

1.5 Structure of the Thesis

This thesis is divided into five chapters. In the first chapter, a general introduction of the overall research conducted in this thesis is described. It includes the background of the study, problem statement, the scope of investigation, objectives and thesis layout. Chapter 2 presents a literature review relevant to the research conducted, focusing on polymers, natural fibres and additives. The third chapter describes the methodology used to carry out this study, including the materials used, fabrication and characterisation processes. In chapter 4 the results obtained are presented along with analysis and discussion. Chapter 5 provides the conclusion of this study along with future work recommendations.

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