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CHARACTERIZATION AND DEVELOPMENT OF SUGAR PALM-FILLED PHENOLIC COMPOSITES AS FRICTION MATERIALS

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By

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Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfillment of the Requirements for the Degree of Doctor of Philosophy

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Faculty : Engineering

Sugar palm fiber (SPF) is one of the prospective fibers that can be used to reinforce polymer composites. This study aimed to characterize SPF and evaluate the physical, mechanical, thermal, morphological, and tribological properties of the sugar palm filled phenolic (SPF/PF) composites as friction materials. The work was divided into four stages to achieve the specified objectives. The first stage focused on the characterization of the thermal, physicochemical, and morphological properties of untreated and treated SPF fibers. The fibers were treated with sea water for 30 days, and with 0.5 M alkaline solution (NaOH) for 4 days. The results showed that the thermal stability of the untreated fibers was slightly higher than the treated ones due to the high percentage of silica (SiO_2) content in the untreated fibers. It was also observed that the fiber surface became clean and smother after treatments and thus better fiber-matrix adhesion was achieved. The second stage examined the physical (Rockwell hardness, water/oil absorption, density, and void content), mechanical (compressive, impact, and flexural), morphological, and thermal (thermogravimetric and dynamic mechanical analysis) properties of SPF/PF composites. Sugar palm fibers in particle size of about ≤ 150 μm and phenolic resin were used to fabricate the composites by the hot press technique, and with different SPF filler loadings of 0, 10, 20, 30, and 40 % by volume. The results showed that, as the SPF filler increases Rockwell hardness decreased, while the water/oil absorption and density increased. The mechanical properties of the composites were also improved, while the thermal stability decreases. Overall, the results showed that the 30 vol. % SPF/PF composites dominated the best physical and mechanical properties, thus it was used for further investigation in the third and fourth stages of this work. The influence of sea water and alkaline SPF fiber treatments on the properties of the phenolic composite was carried out. Both treatments helped to enhance fiber-matrix bonding and consequently improved the physical and mechanical properties of the treated fiber composites. The untreated fiber composites were found to be slightly more thermally stable than the treated ones. In the fourth stage, the tribology behavior of SPF/PF (30 vol. %) was compared with the neat phenolic composites. The results showed that incorporating

SPF in phenolic composites decreases the specific wear rate and the coefficient of friction by 64.1 % and 22.6 %, respectively. Furthermore, the tribology behavior of the untreated and treated fiber composites based on the optimum fiber loading was conducted under room and elevated (250 °C) temperatures. The process parameters such as treatment, load and sliding speed were optimized by using DOE (Factorial technique). The treated fiber composites showed better wear behavior compared to the untreated composites. However, the volume losses of all the composites at elevated temperatures were found to be more than those at room temperatures due to the high sliding friction force. Interestingly, the result revealed that SPF can be used as viable reinforcement material in phenolic composites at room and elevated temperatures. In conclusion, sugar palm fiber can be used as an alternative natural fiber for friction materials such as brake pad composites.

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PENCIRIAN DAN PEMBANGUNAN KOMPOSIT-FENOL TERISI GENTIAN IJUK SEBAGAI BAHAN GESERAN

Oleh

BUSHRA RASHID MOHAMMED

April 2017

Pengerusi : Profesor Madya Zulkiflle Leman, PhD
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Gentian ijuk (SPF) adalah salah satu daripada gentian yang boleh digunakan untuk mengukuhkan komposit polimer. Kajian ini bertujuan untuk mencirikan SPF dan menilai sifat-sifat fizikal, mekanikal, terma, morfologi, dan tribologi komposit fenol berdasarkan gentian ijuk (SPF/PF) sebagai bahan geseran. Kerja ini telah dibahagikan kepada empat peringkat untuk mencapai objektif yang ditentukan. Peringkat pertama memberi tumpuan kepada pencirian sifat-sifat haba, fizikokimia, dan morfologi gentian SPF yang tidak dirawat dan yang dirawat. Gentian telah dirawat dengan air laut selama 30 hari, dan dengan larutan alkali 0.5 M selama 4 hari. Hasil kajian menunjukkan bahawa kestabilan terma gentian yang tidak dirawat adalah lebih tinggi sedikit daripada yang dirawat kerana peratusan tinggi kandungan silika (SiO_2) di dalam gentian yang tidak dirawat itu. Juga diperhatikan bahawa permukaan gentian menjadi bersih dan kasar selepas rawatan dan dengan itu lekatan gentian-matriks yang lebih baik telah dicapai. Peringkat kedua memeriksa ciri-ciri fizikal (kekerasan Rockwell, penyerapan air/minyak, ketumpatan dan kandungan kekosongan), mekanikal (mampatan, impak, dan lenturan), morfologi, dan terma (Termogravimetri dan analisis mekanikal dinamik) komposit SPF/PF tersebut. Gentian ijuk dalam saiz zarah kira-kira ≤ 150 µm dan suatu resin fenol digunakan untuk membikin komposit dengan teknik penekan panas, dan dengan beban pengisi yang berbeza sebanyak 0, 10, 20, 30, dan 40% mengikut isipadu. Hasil kajian menunjukkan bahawa, sebagai SPF pengisi kenaikan Rockwell kekerasan menurun, manakala penyerapan air/minyak dan ketumpatan meningkat. Sifat-sifat mekanikal bagi komposit juga telah bertambah baik, manakala kestabilan haba berkurangan. Secara keseluruhannya komposit 30% isipadu SPF/PF menguasai sifat-sifat fizikal dan mekanikal yang lebih baik, dan dengan itu ia telah digunakan untuk siasatan lanjut di peringkat ketiga dan keempat kerja ini. Pengaruh air laut dan rawatan alkali gentian SPF ke atas sifat-sifat komposit telah dijalankan. Kedua-dua rawatan membantu meningkatkan ikatan gentian-matriks dan seterusnya meningkatkan sifat-sifat fizikal dan mekanikal komposit gentian dirawat. Komposit gentian tidak dirawat didapati lebih sedikit kestabilan termanya daripada yang dirawat. Pada peringkat

keempat, tingkah laku tribologi SPF/PF (30% isipadu) telah dibandingkan dengan komposit fenol yang tidak terisi. Hasil kajian menunjukkan bahawa menggabungkan SPF di dalam komposit fenol mengurangkan kadar haus tertentu dan pekali geseran dengan 64.1% dan 22.6% masing-masing. Tambahan pula, tingkah laku tribologi komposit gentian yang tidak dirawat dan yang dirawat berdasarkan beban gentian optimum telah dijalankan di bawah suhu bilik dan suhu tinggi (250 °C). Parameter proses seperti rawatan, beban dan kelajuan gelongsor telah dioptimumkan dengan menggunakan DOE (teknik Faktoran). Komposit gentian dirawat menunjukkan tingkah laku haus yang lebih baik berbanding komposit yang tidak dirawat. Bagaimanapun, kehilangan isipadu semua komposit pada suhu tinggi didapati lebih daripada yang berada di suhu bilik kerana daya geseran gelongsor yang tinggi. Yang menariknya, keputusan mendedahkan bahawa SPF boleh digunakan sebagai tetulang berdaya maju di dalam komposit fenol di suhu bilik dan suhu tinggi. Kesimpulannya, gentian ijuk boleh digunakan sebagai gentian semula jadi alternatif untuk bahan geseran seperti komposit pad brek.

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I certify that a Thesis Examination Committee has met on 20 April 2017 to conduct the final examination of Bushra Rashid Mohammed on her thesis entitled "Characterization and Development of Sugar Palm-Filled Phenolic Composites as Friction Materials" 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 Doctor of Philosophy.

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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
ASTM	American society of testing and materials
AT	Alkali-treated sugar palm fiber reinforced phenolic composites
C=O	Carbonyl group
C-H	Carbo-hydrogen
C-O	Carbon-oxygen
COF	Coefficient of friction
DOE	Design of experiment
DMA	Dynamic mechanical analysis
DTG	Derivative thermogravimetric analysis
EDX	Energy-dispersive X-ray spectroscopy
FT-IR	Fourier transform infrared
PF	Phenolic formaldehyde
POD	Pin on disc apparatus
Ra	Surface roughness measurement
SEM	Scanning electronic microscope
Si	Silicon
SiO_2	Silica (Silicon dioxide)
SPF	Sugar palm fiber
SPF/PF	Sugar palm fiber reinforced phenolic composites
ST	Sea water-treated sugar palm fiber reinforced phenolic composites
SWR	Specific wear rate
UT	Untreated sugar palm fiber reinforced phenolic composites
VL	Volume loss

CHAPTER 1

INTRODUCTION

1.1 Overview

The issues of global warming and environmental pollution concerned human community, and being addressed by many researchers. Natural fiber reinforced polymer composites, or green composites, could help to overcome the problems. Green composites have gained great attention from researchers due to the awareness towards global environmental concerns (Palanikumar *et al.*, 2016). Natural fibers are environmentally friendly and sustainable materials, which can replace synthetic fibers in the composites industry. In addition, dual benefits can be gained from using natural fibers in the renewable energy field. First, by reducing the usage of synthetic and toxic fibers and replacing them with biodegradable and safer fibers; and second, by contributing to renewable resources. Furthermore, natural fibers have lower cost (US$ 220-1000/ton) and energy to produce (4 GJ/ton) than carbon and glass (cost: US$ 12500/ton and US$ 1200-1800/ton) and energy to produce (30 GJ/ton and 130 GJ/ton), respectively (Shalwan and Yousif, 2013). Several types of natural fibers have been used as reinforcements in polymer composites, such as kenaf, oil palm fiber, jute, roselle, bamboo, hemp, sisal, banana, and pineapple.

Recently, the application of green composites has covered a wide range of industries, (Al-Oqla *et al.*, 2015; Palanikumar *et al.*, 2016). Interest is warranted by the benefits of these natural fibers in comparison to synthetic fibers. Natural fibers are abundantly available at a very low cost and have low environmental impact, and good specific properties (high stiffness and strength per unit mass). Further benefits include low density, superior wear properties, and less harmful health effects (Shalwan and Yousif, 2013). On the flip side, these fibers exhibit certain drawbacks such as poor compatibility with the matrices, high moisture absorption tendency, and low thermal stability. Such shortcomings limit the applications of natural fibers (Isma'ila *et al.*, 2016; Razali *et al.*, 2015). The fiber-matrix adhesion considerably affect performance of green composites (Jawaid and Abdul Khalil, 2011). Thus, fiber surface treatment is highly recommended (Nadlene *et al.*, 2016; Rajkumar *et al.*, 2016).

During the past decade, natural fiber reinforced polymer composites have an increased interest due to the environmental awareness of consumers, as realistic alternative agent to replace or reduce synthetic fiber in many sectors. A number of significant industries such as packaging, construction, and automotive industries have witnessed massive attention in the progress of new green composites. Although, these extensive studies have reported on the monotonic properties; tensile, compressive, flexural, and impact, a noticeable lack of studies on the tribology performance of natural fiber reinforced polymer composites. Many studies have been conducted on synthetic fiber reinforced thermoset and thermoplastic polymer composites. In contrast, less attention was paid to the influence of natural fiber on the tribology behavior of polymer composites, since only few attempts have been reported (Shalwan and Yousif, 2013).

Many industrial parts are exposed to tribological loading under operating conditions such as brake pads, brake linings, and brake couplings. Thus, understanding the tribological behavior of the green composites should be considered as mechanical properties as well (Shalwan and Yousif, 2013). Reinforcing the neat polymer with natural fibers could significantly improve the tribo-performance of the composites (Yousif and El-Tayeb, 2008). However, some researchers reported that the tribological performance of polymer composites-based natural fiber is not essentially on performance but it highly relies on many parameters such as polymer characterization, fiber-matrix adhesion, wear test conditions, and wear operating parameters (Omrani *et al.*, 2016). These aspects of natural based polymer composites are not covered in details and need further studies.

Sugar palm fiber (SFP) is a natural fiber extracted from *Arenga pinnata* trees that were usually grown in South Asia (Ishak, *et al*., 2013d). This fiber seems to have properties of other natural fibers, but the detail properties are not generally known yet. Also, studies of sugar palm fiber composites have been usually focused on their mechanical properties (Sanyang *et al.*, 2016). Nevertheless, no work has been found on the tribology behavior of sugar palm fiber reinforced polymer composites in the literature.

1.2 Problem Statements

Over the last few years, an amazing increase has been observed in the use of natural fibers in the replacement of synthetic fibers when producing various materials. This increase has been pushed further by the worldwide concern for environmental issues along with the use of depleting resources and, as a result, the search for materials that are eco-friendly. More particularly, this concern has led to an increase in new and stronger policies on the environment, which have forced various industries, such as automotive, packaging, and construction, to search for alternative reinforcements for the traditionally used composite materials (Sahari *et al.*, 2012b; Yusriah *et al.*, 2014).

In friction material composites field, according to the regulations against hazardous ingredients in the United States and Europe, several ingredients such as asbestos, copper, and lead have been banned from the use as friction fiber enhanced polymeric composites in brake coupling, brake lining, and brake pads due to their harmful effect on the environment and humans (Elakhame *et al.*, 2014; Menezes *et al.*, 2012). For example, in 1986, the Environmental Protection Agency (EPA) proposed a ban on asbestos that required all new vehicles to have non-asbestos brakes by September 1993, and the aftermarket would have had until 1996 to convert to non-asbestos composites. This is due to an evidence by a medical research that asbestos fibers would lodge in the lungs causing adverse respiratory conditions (Blau, 2001). Also, California State approved the SENATE BILL SB 346 which forbids motor vehicle brake materials that contain more than 5 and 0.5 wt % copper by January 1, 2021, and January 1, 2025, respectively (Lee and Filip, 2013). Increased environmental awareness and consciousness throughout the world has developed an increasing interest in natural fibers and its application as alternative materials.

Nowadays, the growing interest in adopting natural fibers such as flax, coir, palm kernel shell, kenaf, oil palm fiber, jute, roselle, bamboo, and etc. as reinforcement for polymeric composites is increased due to their useful and eco-friendly properties. They are non-toxic, low cost, biodegradable, light weight, renewable, high specific strength, non-abrasively and combustible (Al-Oqla *et al.*, 2016). In addition, such fibers have high specific properties such as stiffness, impact resistance, flexibility, and modulus. Other properties include less skin and respiratory irritation and enhanced energy recovery. The biodegradability of natural fibers can contribute to a healthy ecosystem while their low cost and high performance fulfil the economic interest of industry (Menezes *et al.*, 2012). On the other hand, agro waste products are emerging as new and inexpensive materials in the friction materials development with commercially viable and environmentally acceptable (Mutlu, 2009).

Although there are many advantages to using natural fibers as reinforcements in polymer composites, there are some limitations. The high moisture absorption tendency, poor fiber-matrix interfacial bonding, and low thermal stability are the main problems of natural fibers when they are used to reinforce polymer composites. The performance of the composites depends highly on the fiber-matrix adhesion. Thus, a fiber surface treatment is one of the most effective methods to enhance the fiber-matrix adhesion and overcome this problem (Thakur and Singha, 2015), and should be considered prior to composite fabrication (AlMaadeed *et al.*, 2013; Rajkumar *et al.*, 2016).

Sugar palm fiber (SPF) is mainly found in Malaysia and Indonesia. It is a potential alternative reinforcement to replace conventional synthetic fibers (Ishak, *et al.*, 2013d). Sugar palm fiber has comparable advantages that are similar to other natural fibers. It has a high durability and good resistance to sea water (Isma'ila *et al.*, 2016). Also, using sugar palm fiber as reinforcement material in polymer composites can contribute significantly to the income of farmers. Phenolic resin (PF) normally used in friction composites as a binder because of its good properties. It has a high rigidity, good dimensional stability, and excellent heat resistance (Surojo *et al.*, 2014). Many studies have been reported on the reinforced SPFs in various polymers such as epoxy, unsaturated polyester, high impact polystyrene, etc., composites (Ishak *et al.*, 2013d). On the flip side, no work has been found in the literature regarding SPF fibers as reinforced material in phenolic composites.

The tribology literature is full of records on the tribological characteristics of synthetic fiber reinforced polymer composites. In contrast, very limited studies on the tribo-potential of polymeric composites using natural fiber reinforcement. The positive conclusion of the studies indicates reinforcing natural fiber can improve the wear performance of polymer composites (Omrani *et al.*, 2016; Shalwan and Yousif, 2013). However, there is a lack of understanding the wear mechanism of natural fiber in polymer composites under various process parameters due to the limited background information regarding using natural fiber in the friction composites.

1.3 Research Objectives

The general aim of this research was to evaluate the behavior of sugar palm fibers embedded in phenolic composite. The specific objectives were set out as follows:

1. To characterize the effect of treatments on the physical, chemical, morphological, and thermal properties of the sugar palm fiber.
2. To determine the effect of sugar palm fiber loading on the physical, mechanical, morphological and thermal properties of phenolic composites.
3. To evaluate the influence of treatments on the physical, mechanical, morphological, and thermal properties of the optimum fiber loading of sugar palm fiber embedded in phenolic composites.
4. To investigate the tribological behaviour of the optimum fiber loading of the untreated and treated fiber composites under optimized wear process parameters at ambient and elevated temperatures.

1.4 Scope of Study

In this study, the properties of SPF that were naturally and chemically treated were evaluated. The novelty of embedded SPF filler in phenolic composites was explored in order to contribute to the existing knowledge, on SPFs application, and in the field of natural fiber composites. Thus, the behaviors of SPF/PF composites in terms of physical, mechanical, morphological, and thermal properties was considered. Furthermore, the tribo-performance of the untreated and treated fiber composite were considered under different wear parameters such as the applied load and the sliding speed at room and elevated temperatures.

This study focused on using sugar palm fibers and phenolic resin as primary candidates. Sugar palm fiber was selected due to its good features. It has comparable properties over other natural fibers which are the high durability, the high resistance to sea water, good mechanical properties and absorbed less moisture. Since, the phenolic polymer has good thermal stability and could withstand at high temperature, thus it is commonly used in the friction materials as a binder. The sugar palm fibers were used in filler form (about ≤ 150 µm) as a filler embedded in phenolic polymer composites and the composites fabricated by a hot press machine. Sea water and alkaline treatments were employed to treat the sugar palm fiber. The fibers soak in sea water for 30 days, and in 0.5% solution of sodium hydroxide for 4 hrs. The methodology of this research is an experimental investigation, and the research was divided into four phases:

The first phase of the research is fiber characterizations. The surface-treated SPFs were subjected to physical, chemical, and thermal stability analyses using Fourier transform infrared and energy dispersive X-ray spectroscopy as well as thermogravimetric analysis (TGA), respectively, whereas scanning electronic microscopy (SEM) was used to examine the cross-section and surface modification of the fiber.

SPF/PF composites were prepared using five different loadings which are 0, 10, 20, 30, and 40 % by volume. The SPFs and phenolic powders were mixed using a mechanical string. Then, the mixture poured into a mold and hot pressed at a temperature of 160 °C and pressure of 20 tons for 20 minutes and post cured. The composites were tested for their physical (water and oil absorption, moisture content, hardness, density and voids content), mechanical (flexural, impact, and compressive strength), morphological and thermal (TGA and DMA) properties. The highest yields in the properties of SPF loading/phenolic composites was found to be 30 % and it will be used to produce composites samples for the next phases.

The third phase focuses on the influence of sea water and alkaline treatments on the physical, mechanical, morphological, and thermal properties, which mentioned above, of the optimum fiber loading composites.

The resulted optimum properties composites were tested under tribology parameters at room and elevated (250 °C) temperatures. The wear test conduct using a pin on disc apparatus. Untreated, alkaline, and sea water fiber treated based phenolic composites were designed as UT, AT, and ST composites, respectively. Factorial technique as a design of experiment (DOE) along with the analyses of variance (ANOVA) were used to design and evaluate the wear and friction results. The influenced factors included treatment (UT, ST, and AT), applied load (30, 50, and 70) N, and sliding speed (2.6, 3.9, and 5.2) m/s at 5000 m sliding distance. Scanning electron microscopy (SEM) analysis was used to examine the morphology of the worn surfaces.

1.5 Thesis Outline

This research consists of five chapters including

Chapter 1: presents a brief background of the field of green composites with focusing on the noticeable lack of tribological behavior and highlight the research problems. Also, describing the objectives of research, and finally defines the boundaries of the work.

Chapter 2: contains reviews of the available literature on natural fibers reinforced composites with focusing on sugar palm composites. The tribology behavior as well as mechanical properties of polymer composites is also reviewed. Also, DOE and ANOVA analyses will be discussed in this chapter.

Chapter 3: this chapter shows the material specifications, composites tests details, equipment's and standards followed. Finally, the adopted methodology to attain the research objectives will be explained in details.

Chapter 4: the discussion on the results and findings of the study are presented.

Chapter 5: conclusions on the finding of the research are drawn. Finally, the recommendations for future research are suggested in this chapter.

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