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EFFECTS OF KENAF FIBER BLEACHING AND WASTE COOKING OIL ON PROPERTIES OF PLA/KENAF BIOCOMPOSITE

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EFFECTS OF KENAF FIBER BLEACHING AND WASTE COOKING OIL ON PROPERTIES OF PLA/KENAF BIOCOMPOSITE

By

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EFFECTS OF KENAF FIBER BLEACHING AND WASTE COOKING OIL ON PROPERTIES OF PLA/KENAF BIOCOMPOSITE

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May 2015

Chairman : Nor Azowa binti Ibrahim, PhD
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Biocomposite made from poly(lactic acid) (PLA) and kenaf fiber offers a totally biodegradable and cost effective material. Owing to poor fiber-matrix adhesion and unattractive colour appearance, kenaf fiber was treated by bleaching method. The bleaching treatment was conducted in a solution of hydrogen peroxide at pH 11, 80 °C for 60 minutes. In this study, the biocomposites were prepared via melt blending technique using Haake Polydrive.

Due to the bleaching treatment, the amorphous components such as hemicellulose and lignin were partially removed from the bleached kenaf fiber, which resulting good colour appearance of fiber, improvement in thermal stability and increase of crystallinity. The biocomposites that produced from the combination of bleached kenaf fiber and PLA were considerably brighter than PLA/unbleached kenaf fiber biocomposites. However, the elongation at break of PLA/bleached kenaf fiber biocomposite was lower than PLA, indicating that the kenaf fiber bleaching treatment did not improve the flexibility of biocomposite. Therefore, waste cooking oil (WCO) was tested as a plasticizer for the biocomposites in order to alter this drawback.

The simultaneous effects of kenaf fiber bleaching and WCO on PLA/kenaf fiber biocomposites were investigated with respect to mechanical properties, thermal properties, morphology, biodegradability and water absorption. As compared to PLA/unbleached kenaf fiber biocomposites, good physical interactions between bleached kenaf fiber and plasticized matrix resulted in the increase of elongation at break, tensile strength, flexural strength, impact strength and thermal stability. Because of the plasticization effect, the addition of WCO reduced the tensile modulus, flexural modulus and glass transition temperature. The observation via scanning electron microscope (SEM) on the fractured tensile surface showed close adhesion between bleached kenaf fiber and plasticized matrix. Consequently, it was found that the water absorption and decomposition of biocomposites in soil decreased because the close gap at fiber-matrix interfaces reduced the penetration of water and microorganisms into the biocomposites.

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

KESAN PELUNTURAN SERAT KENAF DAN SISA MINYAK MASAK KE ATAS SIFAT- SIFAT PLA/KENAF BIOKOMPOSIT

Oleh

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Biokomposit diperbuat daripada poli(asid laktik) (PLA) dan serat kenaf menawarkan bahan yang terbiodegradasi sepenuhnya dan kos efektif. Oleh kerana lekatan serat-matriks yang lemah dan penampilan warna yang tidak menarik, serat kenaf telah dirawat dengan kaedah pelunturan. Rawatan pelunturan telah dijalankan dalam larutan hidrogen peroksida pada pH 11, 80 °C selama 60 minit. Dalam kajian ini, biokomposit disediakan melalui teknik leburan pengadunan menggunakan Haake Polydrive.

Disebabkan rawatan pelunturan, komponen amorfus seperti hemiselulosa dan lignin sebahagiannya telah dikeluarkan daripada serat kenaf terluntur, yang mana menyebabkan penampilan warna serat yang baik, peningkatan kestabilan haba dan peningkatan penghabluran. Biokomposit yang dihasilkan daripada kombinasi serat kenaf terluntur dan PLA adalah jauh lebih cerah dari PLA/serat kenaf tidak terluntur biokomposit. Walaubagaimanapun, pemanjangan pada takat putus PLA/serat kenaf terluntur biokomposit adalah lebih rendah dari PLA, menunjukkan bahawa rawatan pelunturan serat kenaf tidak menambah baik fleksibiliti biokomposit. Oleh itu, sisa minyak masak (WCO) telah diuji sebagai pemplastik kepada biokomposit dalam usaha untuk mengubah kelemahan ini.

Kesan serentak rawatan pelunturan serat kenaf dan WCO ke atas PLA/serat kenaf biokomposit telah disiasat berkenaan dengan sifat mekanik, sifat terma, morfologi, biodegradasi dan penyerapan air. Berbanding dengan PLA/serat kenaf tidak terluntur biokomposit, interaksi fizikal yang baik antara serat kenaf terluntur dan matriks yang diplastikkan menyebabkan peningkatan pemanjangan pada takat putus, kekuatan tegangan, kekuatan lenturan, kekuatan impak dan kestabilan haba. Disebabkan kesan pemplastikkan, penambahan WCO pada biokomposit menyebabkan pengurangan modulus tegangan, modulus lenturan dan suhu peralihan kaca. Pemerhatian melalui mikroskop imbasan elektron (SEM) pada permukaan patah tegangan menunjukkan lekatan yang rapat antara serat kenaf terluntur dan matriks yang diplastikkan. Akibatnya, didapati bahawa penyerapan air dan penguraian biokomposit di dalam tanah menurun kerana jurang yang dekat antara permukaan serat-matriks mengurangkan penembusan air dan mikroorganisma ke dalam biokomposit.

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I certify that a Thesis Examination Committee has met on 15 May 2015 to conduct the final examination of Nur Inani binti Abdul Razak on her thesis entitled "Effects of Kenaf Fiber Bleaching and Waste Cooking Oil on Properties of PLA/Kenaf Biocomposite" 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. Committee recommends that the student be awarded the degree of Master of Science.

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TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

ASTM	American Standard for Testing and Materials
BC	PLA/bleached kenaf fiber biocomposite
BWC	PLA/bleached kenaf fiber/3 wt% WCO biocomposite
CH_4	Methane
CO_2	Carbon dioxide
D	Diffusion coefficient
DBP	Dibutylphthalate
DIHP	di-iso-heptylphthalate
DIPP	di-iso-pentylphthalate
DMA	Dynamic mechanical analysis
DOA	Dioctyladipate
DOP	Dioctylphthalate
DTG	Derivative thermogravimetric
EPO	Epoxidized palm oil
ESO	Epoxidized soy bean oil
FTIR	Fourier transforms infrared
HDPE	High density polyethylene
H_2O	Water
H_2O_2	Hydrogen peroxide
ISO	International Standards of Organisation
IUPAC	International Union of Pure and Applied Chemistry
LDPE	Low density polyethylene
MCC	Microcrystalline cellulose
M_m	Maximum water absorption
M_w	Molecular weight
N_2	Nitrogen
NaOH	Sodium hydroxide
O_2	Oxygen
PBS	Poly(butylene succinate)
PBSA	Poly(butylene succinate-co-adipate)
PBAT	Poly(butylene adipate-co-terephthalate)
PCL	Poly(caprolactone)
PE	Polyethylene
PEA	Poly(esteramides)
PET	Poly(ethylene terephthalate)
PHA	Poly(hydroxyalkanoate)
PHB	Poly(hydroxybutyrate)
PHBV	Poly(hydroxybutyrate co-hydroxyvalerate
PLA	Poly(lactic acid)
PP	Polypropylene
PS	Polystyrene
SEM	Scanning electron microscope
TAPPI	Technical Association of the Pulp and Paper Industry
TGA	Thermogravimetric analysis
T_g	Glass transition temperature
T_{max}	Maximum degradation temperature
UBC	PLA/unbleached kenaf fiber biocomposite

UBWC	PLA/unbleached kenaf fiber/3 wt% WCO biocomposite
WCO	Waste cooking oil
Wt%	Percentage of weight
XRD	X-ray diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of study

The utilization of plastics has become a vital part in the commodities and industrial categories. Most of the plastics manufactured today are from synthetic and petroleum based polymers. Polyethylene (PE), polypropylene (PP), poly(ethylene terephthalate) (PET) and polystyrene (PS) are the examples of petrochemical derived polymers which found in large quantities all over the world (Siracusa *et al*., 2008). In fact, the production of the synthetic polymers has reached approximately 140 million tonnes per year (Shah *et al*., 2008). Huge production of these polymers offers the limitless number of applications such as food container, trash bag, electronic insulator and many other things. The synthetic polymers are popular as engineering materials because of their chemical and biological inertness. On the downside, this characteristic is a liability when it comes to waste disposal as the polymers are stable and resist to degradation. Therefore, the disposal of plastic materials can lead to serious waste management in landfill. It is afraid that the increasing amount of plastic wastes being generated would contribute to the filling of all available landfill capacity. Furthermore, the reckless disposal of non-biodegradable plastic materials to the surrounding can cause environmental pollution. Thus, biodegradable material is perceived as means to eliminate some of the landfill wastes and minimize the pollution issue.

The awareness on environmental pollution, non-biodegradability and depletion of petroleum resources have shifted the paradigm towards natural based polymers. Poly(lactic acid) (PLA) is a biopolymer which has good mechanical properties (Fambri *et al*., 1997), biodegradable and renewable (Wee *et al*., 2006). Nevertheless, the application of PLA is limited because the market price of this polymer is relatively more expensive than commodity polymers (Chang *et al*., 2003). The problem can be solved if PLA is mixed with inexpensive material like natural fiber to produce cost effective biocomposite. Recently, kenaf (*Hibiscus cannabinus*, Malvaceae) has been highlighted as filler or reinforcing component for biocomposite because the price of kenaf fiber is just around \$0.70 to \$0.80 per kilogram (Tahir *et al*., 2011) as compared to more pricy glass fiber (synthetic fiber) that usually costs \$1.30 to \$2.00 per kilogram (Joshi *et al*., 2004). Kenaf is quickly grown under a wide range of climate including Malaysia (Aji *et al*., 2009), hence, the supply of this fiber is easily available. Additionally, the incorporation of kenaf fiber into biocomposite can stimulate faster degradation in soil due to its high moisture affinity, which can assist the natural assimilation of microorganism into the biocomposite (Kumar *et al*., 2005). Because of these reasons, PLA/kenaf fiber biocomposite can be a promising solution for the sustainability issues and environmental challenges that are posed by the synthetic polymers.

1.2 Problem statement

The combination of kenaf fiber and PLA to produce a fully biodegradable and low cost biocomposite seems to be an interesting idea. However, the biocomposite made from PLA and kenaf fiber usually have dark colour appearance. This would reduce the commercial value of this biocomposite. Besides, weak interfacial adhesion between fiber and matrix is another challenge in producing good properties of biocomposite. The complex bonding of lignin, hemicellulose and cellulose in the plant fiber as well as the presence of impurities that adhere on the fiber's surface can impede the interactions between cellulose fiber and polymer matrix. The effective interactions between fiber and polymer matrix actually can be achieved if lignin, hemicellulose and other non-cellulosic components are removed from plant fiber (Islam *et al*., 2010). Amorphous components like lignin and hemicellulose cause the plant fiber to have high tendency to absorb moisture. Although water uptake would accelerate the process of biodegradation but in the negative perspective, the absorption of water leads to fiber-matrix debonding at interface, hence, decreasing the mechanical properties of biocomposite (Azwa *et al*., 2013). In view of this thought, some of the amorphous parts should be removed to reduce the penetration of water into the biocomposite.

In order to improve the colour appearance of biocomposite, kenaf fiber should be bleached. As a bleaching agent, hydrogen peroxide (H_2O_2) can be used to decolourize the kenaf fiber. Hence, better colour appearance of biocomposite can be attained when PLA is compounded with brighter colour of kenaf fiber. Furthermore, this bleaching treatment involves elimination of lignin, hemicellulose and surface's impurities during the process (Jonoobi *et al.,* 2010). Therefore, the improvement in fiber-matrix interaction as well as the increase in hydrophobicity of biocomposite could be expected because some of lignin, hemicellulose and impurities are removed from kenaf fiber. Interestingly, both modifications can be done in only one treatment procedure.

PLA is brittle in nature (Zhang *et al*., 2008). The incorporation of natural fiber into PLA commonly increases the brittleness even more because the addition of fiber gives more rigidity to the polymer matrix (Chun and Husseinsyah, 2013). In other words, chains mobility of PLA becomes more restricted in the presence of fiber and consequently prevents the flexibility of biocomposites. This drawback should not be compromised as it would limit the practical uses of the biocomposite.

Flexibility can be altered by adding plasticizer in the biocomposite system. In conjunction with the idea to produce fully biodegradable and cheap biocomposite, it is more preferable if the plasticizer used is originated from natural resources. For more added values, it is more interesting if the plasticizer used for biocomposite can be obtained from waste product. In European countries, the production of waste cooking oil (WCO) is around 700, 000 to 1, 000, 000 tonnes every year (Chhetri *et al.,* 2008). Improper management and disposal of WCO into the water system can cause water pollution. Hence, the potential use of WCO should be explored and expanded in order to transform this waste into useful material.

1.3 Scope of study

In this research, biocomposites made from poly(lactic acid) (PLA) and kenaf fiber had been studied because of their low cost, sustainability and biodegradability. Due to unattractive colour appearance and poor fiber-matrix adhesion, kenaf fiber was bleached using hydrogen peroxide at different pH, temperature and time. For comparison purpose, the photographs of all bleached fibers were captured and the samples were also analyzed for kappa number. In further analysis, the characteristics of bleached kenaf fiber which treated at pH 11, 80 ºC for 60 minutes was compared with unbleached kenaf fiber using Fourier transform infrared (FTIR), thermogravimetric analysis (TGA) and X-ray diffraction (XRD). The surface morphologies of both fibers were examined via scanning electron microscope (SEM).

The biocomposites were prepared by compounding bleached kenaf fiber with PLA via melt blending technique. The biocomposite samples were further compressed into thin sheet using hot-press machine. For comparison purpose, the biocomposites from unbleached kenaf fiber and PLA were also prepared. In order to improve the flexibility of biocomposites, waste cooking oil (WCO) was tested as a plasticizer. The assessments on the mechanical properties were evaluated with respect to tensile, flexural and impact properties. The interfacial adhesion between fiber and matrix were observed via SEM and the possible chemical interactions between bleached kenaf fiber and plasticized PLA matrix were studied using FTIR. Furthermore, the investigation on thermal stability of the biocomposites was performed using TGA whereas the study on thermo-mechanical properties was accomplished using dynamic mechanical analysis (DMA). Besides, the investigation on the responses of biocomposites to soil degradation and water absorption were also studied.

1.4 Research objectives

The principal objectives beyond this research were to:

1. alter the physical colour appearance of kenaf fiber using hydrogen peroxide bleaching treatment and compare the characteristics of bleached kenaf fiber with unbleached kenaf fiber.

2. determine the influence of fiber bleaching treatment on the mechanical properties of PLA/kenaf fiber biocomposites.

3. investigate the simultaneous effects of fiber bleaching treatment and waste cooking oil on the mechanical properties, morphology, thermal properties, water absorption and biodegradability of PLA/kenaf fiber biocomposites.

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