UNIVERSITI PUTRA MALAYSIA

FABRICATION AND CHARACTERIZATION OF HYBRID OIL PALM /KENAF CORE FIBER-REINFORCED POLY(LACTIC ACID) BIOCOMPOSITES

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By

ABUBAKAR BIRNIN-YAURI UMAR

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

August 2017

DEDICATION

To Amina Ibrahim (my mother), and her granddaughter, who she named after her *i.e.,* Amina Abubakar Birnin-Yauri (my daughter).

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the degree of Doctor of Philosophy

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Chairman : Associate Professor Nor Azowa Ibrahim, PhD
Faculty : Science

Conventional fiberboards uses wood fiber and carcinogenic urea formaldehyde, and hence poses economic and environmental problems such as deforestation, desertification, reduced biodiversity and emission of toxic formaldehyde. A potentially promising alternative involves the use of natural fibers thermoplastic composites. This study demonstrated the development of hybrid oil palm-kenaf core fiber reinforced poly(lactic acid) biocomposites for use as a clean and sustainable option to the wood fiberboard. The kenaf core fiber, as secondary fiber was incorporated into empty fruit bunch fiber, or oil palm mesocarp fiber, melt-blended with PLA and hot-pressed into their corresponding hybrid biocomposites, aimed at achieving synergism. The test results showed the best hybridization performances at 5 wt.% kenaf core fiber into 55 wt.% empty fruit bunch fiber or oil palm mesocarp fiber, and 40 wt.% PLA matrix, corresponding to the 60:40 total fiber to PLA loadings, respectively. Though, the difference in the mechanical and physical properties of the hybrid biocomposites is not large, also they revealed morphological defects and high densities, resulting from high contents of hemicellulose and impurities in the natural fibers. Borax modification of the natural fibers, with a water-washing procedures enhances the material performances of the hybrid biocomposites. The borax treatment caused considerable increase in cellulose compositions, minimal removal of lignin, and significant elimination of hemicellulose and waxy substances, as confirmed through chemical analysis, bulk density, Fourier transform infrared spectroscopy, X-ray diffraction analysis, thermogravimetric analysis and scanning electron microscopy of the natural fibers. Optimal improvements in the mechanical and physical properties of hybrid biocomposites were observed at 5 wt.% borax concentration. However, the borax-treated hybrid biocomposites revealed few morphological cracks, recorded relatively higher densities, and statistical insignificance of some mechanical properties, which were attributed to brittleness of the PLA. The use of maleic anhydride-modified PLA greatly aided to overcome these anomalies. The borax-treated hybrid fiber

reinforced maleic anhydride-modified PLA biocomposites provided the best results. Best hybrids showed impressive performances in other fiberboard-related properties, though, they recorded densities within the range of high density fiberboard. This work revealed that the natural fiber hybridization could offer possible synergism, complement material properties of kenaf core fiber, and provides sustainable application of the oil palm fibers in fabrication of natural fiber-based biocomposites.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah.

PEMBANGUNAN DAN PENCIRIAN HIBRID KELAPA SAWIT/TERAS GENTIAN KENAF DIPERKUATKAN POLI (ASID LAKTIK) BIOKOMPOSIT

Oleh

ABUBAKAR BIRNIN-YAURI UMAR

Ogos 2017

Pengerusi : Profesor Madya Nor Azowa Ibrahim, PhD
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Papan gentian konvensional menggunakan gentian kayu dan formaldehid urea yang bersifat karsinogenik dimana ia menimbulkan masalah ekonomi serta alam sekitar seperti penebangan hutan, ketandusan tanah, ketidakseimbangan biodiversiti dan pelepasan toksik formaldehid. Alternatif lain bagi menggantikan gentian konvensional adalah dengan menggunakan komposit termoplastik gentian semulajadi. Kajian ini menunjukan perkembangan hibrid kelapa sawit- gentian teras kenaf polik (asid laktik) biokomposit berpotensi untuk digunakan sebagai papan gentian kayu yang bersih dan mampan. Gentian teras kenaf sebagai serat sekunder, telah dimasukan ke dalam serat tandan buah kosong, atau gentian mesokarpa kelapa sawit, yang dicairkan dan dicampur dengan PLA, dipanaskan dan ditekan menjadi biokomposit hibrid yang sepadan, bertujuan untuk mencapai sinergi. Keputusan ujian menunjukkan prestasi hibridisasi terbaik pada 5 wt.% gentian teras kenaf kepada 55 wt.% gentian tandan kosong atau gentian mesokarp kelapa sawit, dan 40 wt.% PLA matriks, bersamaan dengan nisbah masing-masing 60:40 kepada komposisi gentian dan muatan PLA. Walau bagaimanapun, perbezaan sifat-sifat mekanik dan fizikal biokomposit hibrid tidak ketara, mendedahkan kecacatan morfologi dan kepadatan tinggi, yang disebabkan oleh kandungan hemiselulosa dan kekotoran yang tinggi dalam serat semulajadi. Pengubahsuaian Borax gentian semula jadi dengan prosedur basuhan air meningkatkan prestasi bahan biokomposit hibrid. Rawatan boraks menyebabkan peningkatan besar dalam komposisi selulosa, penyingkiran minimum lignin, pembuangan hemiselulosa yang signifikan dan juga bahan-bahan berlilin, seperti yang disahkan melalui analisis kimia, ketumpatan pukal, jelmaan FTIR, analisis pembelauan sinar-X, analisis Termogravimetri dan imbasan mikroskop elektron. Peningkatan optimum dalam sifat mekanik dan fizikal biokomposit hibrid telah diperhatikan pada 5 wt.% kepekatan borax. Walau bagaimanapun, biokomposit hibrid boraks dirawat mendedahkan beberapa kecacatan morfologi, mencatatkan kepadatan yang lebih tinggi, menghasilkan keputusan statistik yang tidak signifikan bagi

beberapa ciri-ciri mekanikal, yang dikaitkan dengan kerapuhan PLA. Penggunaan PLA dimodifakasi dengan maleik anhidrida sangat membantu untuk mengatasi anomali ini. Serat hibrid yang dirawat borax yang diperkuat dengan biokomposit PLA yang diubahsuai maleik anhidrida memberikan hasil terbaik. Hibrid terbaik menunjukkan persembahan yang mengagumkan dalam sifat berkaitan papan gentian lain, walaupun, mereka mencatatkan kepadatan dalam julat papan serat tinggi. Kerja ini mendedahkan bahawa penghibridan serat semula jadi boleh menawarkan potensi sinergi yang baik serta melengkapi sifat bahan gentian teras kenaf dalam menyediakan aplikasi mampan gentian kelapa sawit untuk fabrikasi biokomposit berasaskan gentian semula jadi.

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I certify that a Thesis Examination Committee has met on 7 August 2017 to conduct the final examination of Abubakar Birnin-Yauri Umar on his thesis entitled "Fabrication and Characterization of Hybrid Oil Palm /Kenaf Core Fiber-Reinforced Poly(Lactic Acid) Biocomposites" 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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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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I hereby confirm that:

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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
BX	Borax
BX(EFBF-KCF)-PLA	Borax-treated empty fruit bunch fiber-kenaf core fiber –poly(lactic acid) hybrid biocomposites
BX(OPMF-KCF)-PLA	Borax-treated oil palm mesocarp fiber –kenaf core fiber –poly(lactic acid) hybrid biocomposites
BX(EFBF-KCF)-mPLA	Borax-treated empty fruit bunch fiber-kenaf core fiber –maleic anhydride modified-poly(lactic acid) hybrid biocomposites
BX(OPMF-KCF)-mPLA	Borax-treated oil palm mesocarp fiber –kenaf core fiber – maleic anhydride modified-poly(lactic acid) hybrid biocomposites
DMA	Dynamic mechanical Analysis
DTG	Derivative of thermogravimetry
EFBF	Empty fruit bunch fiber
EFBF-PLA	Non-hybrid empty fruit bunch fiber-poly(lactic acid) biocomposite
EFBF-KCF-PLA	Untreated empty fruit bunch fiber-kenaf fiber-poly(lactic acid) hybrid biocomposite
FTIR	Fourier transform infrared spectroscopy
KCF	Kenaf core fiber
KCF-PLA	Non-hybrid kenaf core fiber-poly(lactic acid) biocomposite
MDF	Medium density fiberboard
OPMF	Oil palm mesocarp fiber
OPMF-PLA	Non-hybrid oil palm mesocarp fiber-poly(lactic acid) biocomposite
OPMF-KCF-PLA	Untreated oil palm mesocarp fiber-kenaf fiber-poly(lactic acid) hybrid biocomposite

PLA	Poly(lactic acid)
SEM	Scanning electron microscopy
TGA	Thermogravimetric analysis

CHAPTER 1

INTRODUCTION

1.1 Background of the study

Polymer composite industries were largely dominated by petroleum-based and synthetic polymer materials, and synthetic fiber reinforcements for decades. The industries use inorganic materials (*e.g.* glass and carbon) as reinforcements with petroleum-based or biopolymer matrices. However, the traditional fiber reinforcements had exhibited a reasonable number of environmental, health and economic concerns (Abdul Khalil *et al.* 2012; Onishi *et al*. 2010; Bentley, 2002). The traditional fibers are associated with the serious ecological and health hazards to the employees working in the polymer composite industries (Jawaid *et al.* 2015a). The fiberglass dust particulates can contaminate air, thereby causes irritation to the eyes and skin, as well as pulmonary fibrosis and cancer (Ishak *et al.* 2010).

There are currently growing interest in the utilization of cleaner option by using natural fibers such as oil palm, kenaf, banana, pineapple, hemp, bamboo, sisal *etc.* These natural fibers, together with other numerous agro-waste fibers, have shown impressive performances in polymer composite over the conventional fiber reinforcements (Faruk *et al.* 2012). These beneficial performances include renewability (Mukherjee & Kao, 2011), natural abundance (Eichhorn *et al.* 2001), biocompatibility (Kim *et al.* 2006), eco-friendliness (Leão *et al.* 2008), requirement for low energy during processing (Jawaid *et al.* 2015b), low density profile (Hashim *et al.* 2015), attractive specific properties (Li, 2004), good thermal stability and insulating ability (Khazaeian *et al.* 2015), low wear and tear during fiber purification, extraction and modification (Nayak *et al.* 2009), relative less abrasiveness and sustainable tendencies *etc.* (Abdul Khalil *et al.* 2010; Khalil et al., 2012).

The natural fibers differ in reinforcing abilities compared to the synthetic fibers. For instance, the oil palm fibers are cellulose-rich (Sreekala *et al.* 1997), and more hygroscopic than the synthetic fibers (*e.g.* glass and carbon).Thus, the combination of the oil palm fibers with the synthetic fibers could enhance the hydrophobicity of the composites (Thwe & Liao, 2003). Conversely, the synthetic fibers are stiffer, and tougher than oil palm fibers and will provide an improved mechanical performance to the hybrid composites, whereas the oil palm fibers complement the inflexibility of the synthetic fibers.

Similarly, natural fibers varies in their chemical compositions such as cellulose, hemicellulose, lignin *etc*. These variations could hamper their utilization as a single reinforcement into the polymer matrix. Thus, natural fiber-natural fiber hybridization could complement material properties of hybrid fiber component and enables achieving synergism in material performances, and provides sustainable application of the fiber raw materials (Abdul Khalil *et al.* 2012). Oil palm fibers have demonstrated amazing hybridization potentials, upon incorporation as secondary fibers, in order to supplement the rubberwood in the production of alternative medium density fiberboard (MDF) in Malaysia (Abdul Khalil *et al.* 2010b).

The Malaysia fiberboard industries have increasing interests to develop a completely pure oil palm fiber-based fiberboard (Suleiman *et al*. 2011). The conventional fiberboard uses urea formaldehyde (UF) which is carcinogenic (He *et al.* 2012; Kim & Kim, 2005; Nasir *et al.* 2013). Thermoplastic polymers such as poly(lactic acid) (PLA) possess impressive material properties to be employed as greener option (Chieng *et al.* 2013). The search for another natural fiber, which could be compatible with the oil palm fibers in a hybrid biocomposites, may be a rewarding solution towards averting over-dependence on the oil palm fibers. Kenaf cultivation and its fiber production are abundant in Malaysia. The plant is rich in both bast and core fibers (Abdul Khalil *et al.* 2010).

The kenaf core fiber is light-weighted and rich in lignin, though its high composition of hemicellulose and low composition of cellulose are its major drawbacks for composites fabrication. Therefore, it will be impressive to mix the cellulose-rich oil palm fibers and lignin-rich kenaf core fiber to form a hybrid, thereby, complementing the fiber properties lacking in both oil palm fibers and kenaf core fiber. Also, the hybridization could enables the sustainability of the oil palm fibers considering the growth variation between the two fiber sources. The oil palm tree can take up to 2.5 to 4 years (Bergert, 2000; Wahid *et al.* 2005), while the kenaf plants take only 150-60 days to attained maturity (Aji *et al.* 2009; Ridzwan & Ishak, 2007).

Hybrid natural fiber biocomposites exhibit remarkable improvement in material performances compared to those of their non-hybrid fiber biocomposites (Abdul Khalil *et al.* 2010; Kalaprasad & Thomas, 1995; Nayak *et al.* 2009; Shinoj *et al.* 2011). Different natural fibers (e.g. sisal, kenaf, bamboo *etc.*) were hybridized and used as reinforcements for thermoplastic composites (Deka *et al.* 2013; Shinoj *et al.* 2011). However, little or no attempts are made to develop hybrid oil palm fiber and kenaf core fiber reinforced PLA biocomposites for alternative wood fiberboard application.

Poor interface adhesion arising due to hydrophilic nature of natural fibers and non-polar behavior of the polymer matrix often affect the material performances of biocomposites. (Aji *et al.* 2009; Mukherjee & Kao, 2011; Velmurugan & Manikandan, 2007). Thus, the natural fibers need to be modified, and could be achieved through either physical, chemical, and biological methods (Asumani *et al.* 2012; Ishak *et al.* 2009; Kaddami *et al.* 2006). Chemical modification using sodium hydroxide is popularly accepted, though lacks green credentials. An alternative benign chemical approach uses mixtures of boric acid and borax (Özalp, 2010; Widiarto, 2005). An improved wood fiber to high-density polyethylene polymer matrix interface bond was achieved, and the composites showed fire retardant properties due to the boric acid and borax, which are well known for their fire retardant effects (Donmez Cavdar *et al.* 2015).

Herein, novel hybrid biocomposites are fabricated by incorporation of kenaf core fiber into empty fruit bunch fiber, and kenaf core fiber into oil palm mesocarp fiber reinforced PLA (*i.e.* EFBF-KCF-PLA and OPMF-KCF-PLA). Also, a novel borax treatment of the natural fibers with a water-washing procedure, as well as maleic anhydride modification of PLA was employed, aimed at achieving synergistically improved material properties (such as physical, chemical, mechanical, morphological, thermal, and dynamic mechanical) of the hybrid biocomposites fabricated for use as an alternative to MDF.

To the best of our knowledge, the incorporation of kenaf core fiber into empty fruit bunch fiber-PLA and oil palm mesocarp fiber-PLA, the borax modification of natural fiber with a water-washing procedure, and the maleic anhydride modification of PLA for the reactive compatibilization with the borax-treated hybrid fibers were first introduced in the present study.

1.2 Problem statement

Fiberboard manufacturing industries in Malaysia uses wood fiber as the main raw material for their production. The cutting down of trees for wood fiber has attracted increased concerns from naturalists and environmentalists, considering that it contributes to deforestation, desertification, and reduced biodiversity. Urea formaldehyde is the main binder used in the fiberboard manufacture. The board usage, either as household or industrial article, poses the risk of toxic formaldehyde emission into the air, which may causes carcinogenic problem to human health. Thus, a sustainable and potentially promising option now employs agricultural residues and natural fibers as reinforcements, and thermoplastic polymers as matrices. Oil palm-based natural fibers in Malaysia can be viable option to wood fibers. Yet, the oil palm fibers are unsystematically burnt through open incineration, thereby contributes to environmental pollution such as release of greenhouse gases, airborne particles and other toxic matters.

Natural fiber reinforced thermoplastic composites, having strong interface bonds, could be cleaner alternatives to conventional wood fiberboards. Though, the disparities in the chemical compositions among the natural fibers used to reinforce thermoplastic polymers could have limitations towards achieving strong interface adhesion within the biocomposite materials. Natural fiber-natural fiber hybridization could strengthens the interface bond, and provides synergy in the material properties of the biocomposites, and complement the properties of the hybrid components.

Strong interface bond is difficult to achieve between the hydrophilic natural fibers and hydrophobic polymer matrix. Natural fiber modification using sodium hydroxide enhances the interface adhesion, and has become very popular but environmentally unfriendly. Borax impregnation of natural fibers has green credentials compared to alkalization using sodium hydroxide. Yet, the hygroscopicity of borax in impregnated state hampers the formation of strong natural fiber to polymer interface bond, and affect the material performance of the biocomposite materials. The thermoplastic poly(lactic acid) (PLA) is brittle, and could restricts the formation of strong interface bond with the natural fibers. Maleic anhydride modification of PLA may improves its bonding qualities with the natural fibers.

1.3 Aim and objectives

1.3.1 Aim

To develop borax-treated hybrid fiber reinforced maleic anhydride-modified PLA biocomposites *i.e.,* BX(EFBF-KCF)-mPLA and BX(OPMF-KCF)-mPLA, having promising mechanical and physical properties to be employed as alternatives to wood fiberboard.

1.3.2 Objectives

The specific objectives of this study were:

(i) To develop and characterize the non-hybrid fiber reinforced PLA biocomposites *i.e.,* empty fruit bunch fiber-PLA, oil palm mesocarp-PLA, and kenaf core fiber-PLA biocomposites respectively
(ii) To develop and characterize the untreated hybrid fiber reinforced unmodified PLA biocomposites *i.e.,* EFBF-KCF-PLA, and OPMF-KCF-PLA, respectively.
(iii) To develop and characterize the borax-treated hybrid fiber reinforced unmodified PLA biocomposites *i.e.*, BX(EFBF-KCF)-PLA and BX(OPMF-KCF)-PLA, respectively.
(iv) To develop and characterize borax-treated hybrid fiber reinforced maleic anhydride-modified PLA biocomposites *i.e.,* BX(EFBF-KCF)-mPLA and BX(OPMF-KCF)-mPLA, respectively.
(v) To test the best hybrid biocomposites for other application performance *i.e.*, horizontal burning, screw withdrawal, contact angle, chemical resistance, and environmental aging.

Therefore, the main hypothesis of the present research can be stated as “Hybrid biocomposites fabricated from BX(EFBF-KCF)-mPLA and BX(OPMF-KCF)-mPLA can have promising mechanical and physical properties to be employed as alternative to conventional MDF”.

The scope of the current study only considered the EFBF, OPMF, KCF, and PLA. It also only considered varying weight percent to obtain best non-hybrid fiber-to-PLA and hybrid fiber-to-PLA loadings. All other parameters (such as temperature, pressure, time, fiber size *etc.*) were not varied. Similarly, only borax treatment of fibers with a water-washing, as well as maleic anhydride modification of the PLA were considered. Furthermore, only concentration was varied to obtain best borax modifier, and during maleic anhydride modification of PLA, other parameters were not varied such as temperature, pressure, time, fiber size, molecular weight of PLA *etc.* The material properties of the hybrid biocomposites considered in this research includes only mechanical (*i.e.* tensile, flexural and impact), physical (*i.e.* dimensional stability and density), morphological (*i.e.* SEM), structural (*i.e.* XRD), thermal (*i.e.* TGA and DMA), and MDF application tests *i.e.,* horizontal burning, environmental aging, chemical resistance, screw withdrawal and contact angle.

1.4 Significance of the study

The significant contributions of this research to knowledge is that hybridization of the oil palm fibers with kenaf core fiber into PLA matrix could provide synergism in material performance, complement the material properties of kenaf core fiber, and provides sustainable application of oil palm fibers in biocomposites fabrication. Also, the borax modification of natural fibers could enhance their bonding qualities with the thermoplastic PLA. And the combined approaches of borax treatment of the natural fiber and maleic anhydride modification of PLA could provide synergistically improved interface adhesion and better material properties than the separate modification methods.

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