



UNIVERSITI PUTRA MALAYSIA

***DEVELOPMENT OF BIOCOMPOSITES FROM POLY(LACTIC ACID) AND
OIL PALM EMPTY FRUIT BUNCH FIBER***

MARWAH BINTI RAYUNG

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EMPTY FRUIT BUNCH FIBER**

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**MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA**

2014



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AND OIL PALM EMPTY FRUIT BUNCH FIBER**

By

MARWAH BINTI RAYUNG

**Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia,
in Fulfilment of the Requirements for the Degree of Master of Science**

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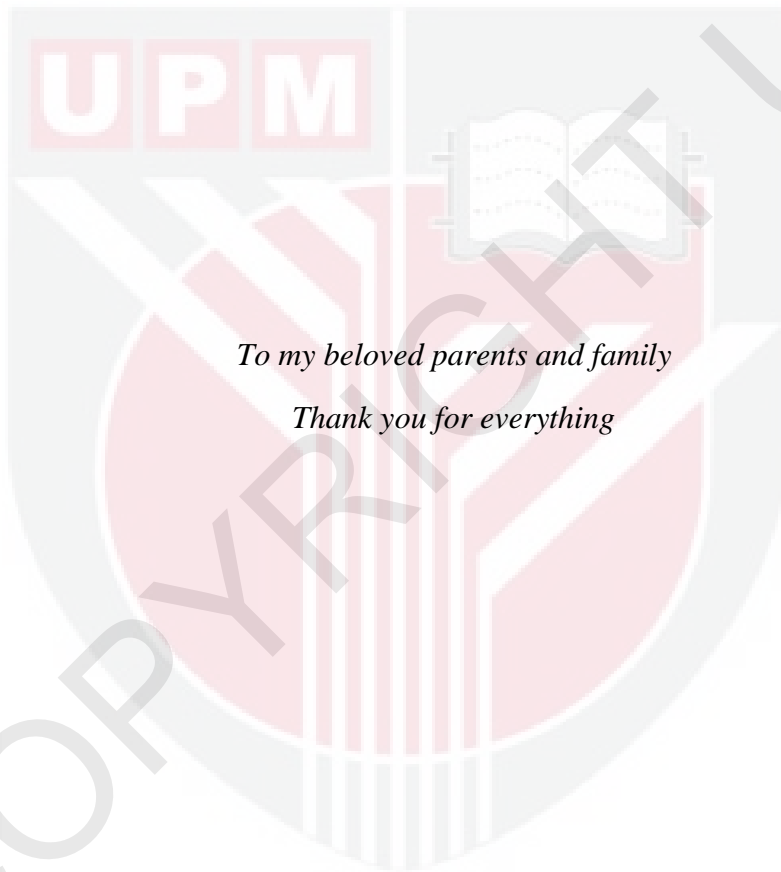
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DEDICATION



To my beloved parents and family

Thank you for everything

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment
of the requirement for the degree of Master of Science

**DEVELOPMENT OF BIOCOMPOSITES FROM POLY(LACTIC ACID)
AND OIL PALM EMPTY FRUIT BUNCH FIBER**

By

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October 2014

Chairman : Nor Azowa Binti Ibrahim, PhD

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The primary objective of this research was to develop fully biodegradable composite material based on poly(lactic acid) (PLA) and oil palm empty fruit bunch (OPEFB) fiber. However, the incompatibility between PLA and OPEFB fibers lead to poor fiber-matrix adhesion resulted in subpar composites performance. In addition, a typical hybridization of natural fiber/polymer matrix produced dark brown colour of product, a factor that limits its applications. Therefore, efforts had been made to treat the fibers by means of alkaline bleaching by using hydrogen peroxide to remove lignin, hemicellulose and surface impurities. The removal of these components is important to enhance the fiber/matrix interlocking system simultaneously increase the brightness of the composites. From the experimental studies, the optimum condition for bleaching process was obtained at pH 11, 70 °C for 90 minutes. Basically, the bleaching process produced brighter colour fiber with rougher surface as can be observed from Scanning Electron Microscopy (SEM). The Infrared (IR) spectra confirmed the removal of lignin and hemicellulose, while X-Ray Diffraction (XRD) result showed a change in fiber crystallinity from 36.8% to 60.6% after bleaching process due to the removal of these amorphous components.

The biocomposites were prepared by mixing PLA with the treated and untreated fiber at different ratio by using internal mixer at temperature 160 °C and 50 rpm. A bright colour biocomposites was successfully produced by using bleached fiber and the addition of colorant improved the appearance. The mechanical properties for bleached fiber biocomposites showed an improvement indicating better fiber-matrix adhesion which is proved by SEM micrographs. Thermal analysis study showed the composites have lower thermal stability compare to neat PLA itself. Water absorption study demonstrated the bleached fiber biocomposite was more water resistance compare to the untreated fiber. At 50% fiber loading, water uptake for bleached fiber biocomposites was recorded at 14.3% whereas 18.7% for untreated

fiber biocomposites. Biodegradable study revealed with the addition of fiber into PLA increased the biodegradation rate of the biocomposites. As a matter of fact, the degradation increased because the fiber is more susceptible to microbial attacked, therefore the addition of fiber helps to accelerate the degradation of biocomposites. Besides, treatment of fiber increased the degradation from 6% to 9% after three months burial time. Conclusively, these results supported the role of PLA/OPEFB as environmental friendly composite and showed a great potential as an alternative to the non-biodegradable plastic.



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

PEMBANGUNAN BIOKOMPOSIT DARIPADA POLI(LAKTIK ASID) DAN SERAT TANDAN BUAH KELAPA SAWIT KOSONG

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Objektif utama kajian ini adalah untuk membangunkan bahan komposit terbiodegradasi sepenuhnya berasaskan poli(laktik asid) (PLA) dan serat tandan buah kelapa sawit kosong (OPEFB). Walau bagaimanapun, ketidakserasian antara PLA dan serat OPEFB membawa kepada masalah lekatan serat/matriks menghasilkan prestasi komposit yang tidak memuaskan. Di samping itu, penghibridan serat/matriks semula jadi selalunya menghasilkan produk yang berwarna coklat gelap yang menghadkan aplikasinya. Oleh itu, kajian telah dilakukan untuk merawat gentian melalui pelunturan alkali dengan menggunakan hidrogen peroksida untuk membuang lignin, hemiselulosa dan benda asing di permukaan serat. Penyingkiran komponen ini adalah penting untuk meningkatkan sistem lekatan serat/matriks pada masa yang sama meningkatkan kecerahan komposit. Berdasarkan eksperimen yang dijalankan, keadaan optimum untuk proses pelunturan adalah pada pH 11, 70 °C dalam masa 90 minit. Proses rawatan telah menghasilkan serat berwarna cerah dengan permukaan kasar seperti yang dapat diperhatikan melalui mikrograf pengimbas elektron mikroskop (SEM). Spektra infra merah (IR) mengesahkan penyingkiran lignin dan hemiselulosa manakala keputusan pembelauan sinar-X (XRD) menunjukkan perubahan dalam penghabluran daripada 36.8% kepada 60.6% selepas proses pelunturan disebabkan penyingkiran komponen amorfus.

Bio-komposit telah disediakan dengan mencampurkan PLA dengan serat yang dirawat dan tidak dirawat pada nisbah yang berbeza dengan menggunakan pengadun dalaman pada suhu 160 °C dan 50 rpm. Komposit berwarna cerah telah berjaya dihasilkan menggunakan serat terluntur dan penambahan pewarna telah memperbaiki rupa fizikal komposit. Sifat-sifat mekanikal bagi komposit serat terluntur telah bertambah baik dengan ketara menunjukkan lekatan serat/matriks yang dibuktikan oleh mikrograf SEM. Kajian analisis terma menunjukkan biokomposit mempunyai

kestabilan terma yang lebih rendah berbanding dengan PLA sendiri. Kajian penyerapan air menunjukkan serat biokomposit terluntur lebih tahan air berbanding dengan serat yang tidak dirawat. Pada 50% kandungan serat, penyerapan air bagi biokomposit daripada serat terluntur adalah sebanyak 14.3% manakala 18.7% untuk biokomposit daripada serat yang tidak dirawat. Kajian biodegradasi menunjukkan penambahan serat ke dalam PLA meningkatkan kadar biodegradasi bagi komposit. Realitinya, proses degradasi meningkat kerana gentian serat lebih mudah terdedah kepada serangan mikroorganisma, oleh itu penambahan serat membantu mempercepatkan degradasi biokomposit. Di samping itu, rawatan serat meningkatkan kadar degradasi daripada 6% kepada 9% selepas tempoh tiga bulan kajian dijalankan. Kesimpulannya, hasil kajian ini menyokong peranan PLA/OPEFB sebagai komposit mesra alam sekitar dan menunjukkan potensi yang besar sebagai alternatif kepada plastik yang tidak mesra alam.



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APPROVAL

I certify that a Thesis Examination Committee has met on 21st October 2014 to conduct the final examination of Marwah Binti Rayung on her Thesis entitled “Development of Biocomposites from Poly(Lactic Acid) and Oil Palm Empty Fruit Bunch Fiber” 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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LIST OF ABBREVIATIONS

ASTM	American Society for Testing of Materials
BF	Bleached fiber
DMA	Dynamic Mechanical Analysis
FTIR	Fourier Transform Infrared
HDPE	High density poly ethylene
ISO	International Standards Organization
LDPE	Low density poly ethylene
MB	Masterbatch
MPa	Mega Pascal
OPEFB	Oil Palm Empty Fruit Bunch
PBS	Poly(butylene succinate)
PCL	Polycaprolactone
PET	Polyethylene terephthalate
PGA	Poly(glycolide acid)
PHA	Poly(hydroxyalkanoate)
PHB	Poly(hydroxybutyrate)
PLA	Poly(lactic acid)
PLA/BF	Poly(lactic acid)/bleached fiber
PLA/BF/MB	Poly(lactic acid) /bleached fiber/masterbatch
PLA/UTF	Poly(lactic acid)/untreated fiber
PP	Poly propylene
PS	Poly styrene
PVC	Poly vinyl chloride
PVP	Poly(vinyl pyrrolidone)
SEM	Scanning Electron Microscopy
SHS	Superheated steam
T _g	Glass transition temperature
TGA	Thermogravimetric Analysis
UTF	Untreated fiber
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 General Background

Plastics have become an essential part of the modern lifestyle. Plastics are used extensively in various fields such as packaging, clothing, building and construction, automobiles, medical devices, electrical appliances, furniture and household goods and so forth. A wide range of products are made of plastics replacing the traditional materials because of their better physical properties, notably strength and toughness, lightness and barrier properties. In fact, production of plastics is also energy efficient and required low operational cost (Scott, 2002). It is safe to say that through plastics, everyday life were made easier and convenient.

The growing demands of plastics every year, shows the dependency of human being towards this materials. In one aspect this trend will lead to economy expansion while in another point of view, the growing bring along negative impact. It is a fact that most of the conventional plastics widely used today are derived from petroleum resources (Kim *et al.*, 2006), for example low and high density poly(ethylene) (LDPE, HDPE), poly(propylene) (PP), poly(styrene) (PS) and poly(vinyl chloride) (PVC). One of the constrain here is the natural resources cannot be sustain forever and subjected to price fluctuation and must eventually be depleted in the future. Since the source is non-renewable, the future of these materials seems unsure and cannot be predicted.

In a more severe scenario, plastics become a huge threat to human being, environment and the ecosystem. With the increasing production of plastic, the amount of waste generated from this industry also increase. The problems arise when these wastes cannot be degraded naturally; in fact they took thousand years to decompose. Since most of these wastes resist to biological degradation, they usually disposed-off using traditional methods such as land-fill and incineration techniques. However, in a long term the disposal methods are not viable option because land fill takes up valuable spaces, while incineration gives off toxic gaseous and affected human health and the environment.

A lot of effort had been done to find the proper plastic waste management system. Recycling is one of the techniques suggested to tackle the problem. Recycling is the process of collecting and processing waste material and turning them into new product. The implementation of this method will reduce the amount of waste sent to landfill and incinerators and help to conserve natural resources. However, recycling techniques is not economically wise since the recovered material has a low intrinsic value and the fact that the original polymers are easily produced cheaply (Scott and Wiles, 2002).

The concern on preservation of natural resources, economic and ecological drawbacks of petroleum-based polymer stimulate interest in biodegradable polymer based on renewable resources. Bio-based polymers derive from agricultural stock are a piece of sustainability picture and they offer environmentally benefits. These polymers have the capability to degrade naturally into organic substances without releasing any toxic component (Wahit *et al.*, 2012). With many interesting features, bio-based polymers have a great potential to be used as an alternative and/or replacement to the existing conventional polymer in many area.

Research and development of biodegradable polymers have been widely conducted in past decades and a number of new bio-based polymers were synthesized. Poly(hydroxybutyrate) (PHB) was the first fully biodegradable polymer produced, derived from microbial fermentation of sugar. The discovery of this material has triggered researches to find other bio-based polymer (Scott, 2002). There are number of bio-based polymer that has been introduced commercially and available in the market nowadays such as poly(glycolide acid) (PGA), poly(lactic acid) (PLA), poly(hydroxyalkanoate) (PHA), polycaprolactone (PCL), poly(butylene succinate) (PBS) and so on. Among all, PLA attracts enormous attention because of its versatility, biocompatibility and biodegradability characteristic.

Poly(lactic acid) (PLA) is a type of linear aliphatic thermoplastic polyester derived from renewable agricultural raw materials (Yu *et al.*, 2009). PLA owns good stiffness, strength and transparency and shows comparable properties to the other petroleum based polymer. The unique characteristics of PLA which can be process similarly to polyolefin make it an attractive option as an alternative to the conventional polymer. There are many instances where PLA has been used in industrial application such as packaging, pharmaceutical and medical usage (Chee *et al.*, 2013). The commercial market for PLA based product has a bright future ahead as consumer demand for environmental friendly products increases.

1.2 Problem Statements

The utilization of bio-based polymer in place of petroleum based polymer gives positive effect in the sense of reduction on environmental impact and offsetting petroleum consumption. One of the major challenges to replace the conventional polymer is the high cost production of bio-based polymer. Another obstacle to deal with bio-based polymer is the fact that some of these materials exhibit limitation in their properties which cause them less preferable to be used as commodity plastic. A lot of studies had been conducted in order to reduce the cost and enhance the properties of the materials such as introducing suitable filler or reinforcement materials and compatibilizer into the system. Modification and enhancement of these polymers is a crucial step to be done to meet the expectation of developing eco-friendly materials that function in variety of commercial applications.

In the case of PLA, it has relatively high cost (RM25/kg) compare to conventional polymer (Mathew *et al.*, 2005). One way to solve this problem is through the incorporation of natural filler, which not only reduces cost but will also improve product performance. Researchers have been focusing on the prospective of natural

fiber as filler. Natural fiber offers both economic and ecological advantages over commercial synthetic fiber, as they are renewable resources, cheap, widely available and non-abrasive. The combination of both bio-based polymer and natural fiber will produced fully biodegradable composites.

Oil Palm Empty Fruit Bunch (OPEFB) fiber has a great potential to be used as filler in polymer materials. OPEFB fiber is a waste product generated from oil palm industry, available abundantly in Malaysia and its surrounding South East Asia countries (Hassan *et al.*, 2010). The incorporation of OPEFB fiber into PLA will maximize the use of oil palm and turn it from waste to value added product. In particular, OPEFB fiber attracts interest because of its environmental friendly featured, abundance in nature, low density and most importantly low operational cost (Abdul Khalil *et al.*, 2008).

Though when combining PLA with OPEFB fiber produced composites materials that are fully degradable, lightweight and cheaper, the combination presents several limitations. For instance, the incompatible character between hydrophilic OPEFB and hydrophobic PLA matrix resulted to poor interfacial adhesion of the two components (Ibrahim *et al.*, 2009). In addition, a typical mixing of native fiber without treatment into PLA produces a dark brown colour of product, one of the factors that limits its application because the range of color that can be obtained is limited and makes it hard to be commercialized. Therefore, the matrix and/or fiber need modification for enhanced performance.

The properties of the composites can be improved by employing two approaches; treatment of fibers and the use of an appropriate additive (Avella *et al.*, 2009). Generally, modification of fiber can be classified into physical and chemical treatment. Physical methods such as super-heated steam, plasma and corona treatment change structural and surface properties of fiber. Chemical route modification of fiber like silane treatment, alkaline treatment, acetylation, maleated coupling and enzymatic treatment usually aimed to improve the fiber-matrix interaction (Faruk *et al.*, 2012). All of these methods only able to solve the incompatibility issues but did not contribute to the enhancement on the appearance of the composites.

Alkaline bleaching treatment by using hydrogen peroxide (H_2O_2) is another alternative that can be used for fiber treatment. This method has been extensively used in the paper and textile industries (Rosa *et al.*, 2009) but quite new in the polymeric composites field. Theoretically, as a bleaching agent, H_2O_2 has the capability to decolorize the fiber by removing chromophoric group in lignin that responsible for the color. In addition, hemicellulose which is the most hydrophilic group in the fiber and other surface impurities will be discarded during the process. The bleaching treatment will produce better physical appearance of the composites and simultaneously improves the fiber-matrix interaction. Interestingly, the treatment will also enables further modification of the composites with color additive to make it more attractive and acceptable. In this study, the effect of hydrogen peroxide treatment on the appearance and performance of the composites was investigated.

1.3 Research Objectives

This study was conducted to synthesize fully biodegradable composites based on PLA and OPEFB fiber. Therefore, three main objectives were carried out, stated as follow:

1. To modify the OPEFB fiber by chemical treatment and study the effect of treatment on the properties of fiber.
2. To prepare PLA/OPEFB fiber composites and evaluate the effect of fiber loading, fiber treatment and color additive on the properties of composites.
3. To characterize the composites in terms of physical, mechanical, thermal stability and morphological properties.

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