

**MECHANICAL, THERMAL AND
MORPHOLOGICAL PROPERTIES OF
POLY(LACTIC ACID)/SUGARCANE BAGASSE
FIBER COMPOSITES**

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UNIVERSITI SAINS MALAYSIA

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**MECHANICAL, THERMAL AND MORPHOLOGICAL
PROPERTIES OF POLY(LACTIC ACID)/SUGARCANE BAGASSE
FIBER COMPOSITES**

by

KHOO RONG ZE

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LIST OF ABBREVIATIONS

DMA	dynamic mechanical analysis
DSC	differential scanning calorimetry
ESO	epoxidised soybean oil
EVA	poly(ethylene-co-vinyl acetate)
FE-SEM	field emission secondary electron microscope
NaOH	sodium hydroxide
PCL	polycaprolactone
PEA	polyesteramide
PHA	polyhydroxyalkanoate
PHB	poly(3-hydroxybutyrate)
phr	part per hundred
PLA	poly(lactic acid)
PLLA	poly(L-lactide)
PP	polypropylene
PVC	polyvinyl chloride
SCB	sugarcane bagasse
SCBF	sugarcane bagasse fiber
SCBFP	sugarcane bagasse fiber powder
SEM	secondary electron microscope
TGA	thermogravimetry analysis

LIST OF SYMBOLS

E'	Storage modulus
E''	Loss modulus
$\tan \delta$	E''/E' ratio
T_{cc}	Cold crystallization temperature
T_g	Glass transition temperature
T_m	Melting temperature
T_{onset}	Onset decomposition temperature
wt%	Weight percentage
χ_c	Degree of crystallinity

SIFAT MEKANIK, TERMA DAN MORFOLOGI KOMPOSIT POLI(ASID LAKTIK)/GENTIAN HAMPAS TEBU

ABSTRAK

Komposit poli(asid laktik)/gentian hampas tebu (PLA/SCBF) disediakan dengan menggunakan kaedah penyebatian leburan dan diikuti oleh pengacuanan mampatan. Minyak kacang soya terepoksida (ESO) dipilih sebagai pemplastik bagi komposit PLA/SCBF. Demi mengkaji kesan saiz gentian terhadap sifat-sifat komposit PLA, SCBF yang dirawat alkali (~5 mm) telah dihancurkan ke dalam bentuk serbuk dan dinamakan SCBFP (~500 μm). Sehubungan dengan itu, tiga parameter telah diselidik dalam kajian ini, iaitu kandungan SCBF (5-15 wt%), muatan ESO (5-15 phr) dan kesan SCBFP. Ujian hentaman Charpy digunakan untuk mengkaji kesan hentaman terhadap komposit PLA sementara sifat morfologi dikaji dengan menggunakan mikroskop electron pensakanan pancaran medan (FESEM). Sifat terma komposit diperiksa dengan penganalisa mekanik dinamik (DMA), kalorimeter pengimbasan pembezaan (DSC) dan penganalisis thermogravimetrik (TGA). Kekuatan hentaman PLA telah dikurangkan dengan penambahan SCBF. Walau bagaimanapun, kekuatan hentaman komposit PLA/SCBF-10 telah ditingkatkan dengan kehadiran ESO disebabkan kesan pemplastikan. Ujian DMA menunjukkan bahawa penambahan SCBF dapat meningkatkan modulus simpanan (E') PLA. Selain itu, SCBFP boleh mempertingkatkan E' dengan lebih ketara. Keputusan DSC menunjukkan bahawa SCBF dan SCBFP menaikkan darjah penghabluran PLA. ESO telah menurunkan suhu peralihan kaca (T_g) bagi komposit PLA. TGA menunjukkan bahawa kestabilan terma komposit PLA/SCBFP adalah setara dengan PLA/SCBF.

MECHANICAL, THERMAL AND MORPHOLOGICAL PROPERTIES OF POLY(LACTIC ACID)/SUGARCANE BAGASSE FIBER COMPOSITES

ABSTRACT

Poly(lactic acid)/sugarcane bagasse fiber (PLA/SCBF) composites were prepared using melt compounding followed by compression molding. Epoxidized soybean oil (ESO) was selected as plasticizer for the PLA/SCBF composites. In order to study the effect of fiber size on the properties of PLA composites, alkali treated SCBF (5 mm length) was grounded into powder form and designated as SCBFP (~500 μm). Accordingly, three parameters were investigated in this research, i.e., SCBF content (5-15 wt%), ESO loading (5-15 phr) and SCBFP effects. Impact properties of PLA composites were characterized by Charpy impact test while morphological properties were studied using field emission scanning electron microscopy (FESEM). Thermal properties of the composites were examined with dynamic mechanical analysis (DMA), differential scanning calorimetry (DSC) and thermogravimetry analysis (TGA). The impact strength of PLA was reduced by the addition of SCBF. Nevertheless, the impact strength of PLA/SCBF-10 composites was improved significantly in the presence of ESO due to the plasticization effect. DMA results showed that the addition of SCBF increased the storage modulus (E') of PLA, and the effect is more pronounced for the one containing SCBFP. From DSC, results showed that SCBF and SCBFP increased degree of crystallinity of PLA. ESO was found to decrease the glass transition temperature (T_g) of PLA composites. As for TGA, the thermal stabilities of PLA/SCBFP composites are comparable to that of PLA/SCBF.

CHAPTER 1

INTRODUCTION

1.1 Research background

The awareness of preserving Mother Earth is increasing among people from all walks of life. As global population continues to increase time by time, topics like greenhouse gas emissions, toxicity and resource depletions are being emphasized lately. Environmental friendly is one of the major attentions in manufacturing companies when they are introducing products to market. In recent years, we have seen that green products are emerging and slowly play an important part in our daily lives. Green polymers are polymers that were produced using bio-based materials obtained from plant sources (Gilfillan et al., 2012). We can see that they are replacing non-biodegradable polymers that were involved in production of home appliances, electronic devices, household cleaners and even production of vehicle parts continuously.

In the field of polymer science and engineering, many studies are made regarding different biodegradable polymers that could replace petroleum based plastics in all kinds of application (Frone et al., 2013). In order to help reduce environmental burden of the petroleum based polymer materials, development of bio-based composites gained enormous interest in polymer community (Bajpai et al., 2013). Green composites are one of the most attractive topics where composites can be manufactured by adding various natural resources into polymers such as poly(lactic acid) (PLA). The addition of natural fibers into polymer materials not only helps to reduce environmental impact, they could also reduce the manufacturing cost and increase commodity of the product.

Poly(lactic acid) (PLA) is an ecological aliphatic polyester obtained from lactic acid (Ali et al., 2009). PLA, having extremely good mechanical properties, is produced on a large scale from fermentation of corn starch to lactic acid and subsequent polymerization. After being used for a longer period, pure PLA can be degraded into carbon dioxide, water, and methane (Qin et al., 2011).

Since PLA is produced using natural resources, its degradation process only involves simple hydrolysis of ester bond and does not require any assistance of enzymes to activate this reaction (Garlotta, 2001). This contributes to wide application of PLA in various fields like biomedical and packaging applications where the materials can be disposed of easily without harming the environment. However, there are some deficiencies of PLA that can affect its application, such as high production cost, brittleness, low thermal stability, lower water vapour and gas barrier properties (Frone et al., 2013; Shukor et al., 2014).

Consequently, reinforcement of PLA matrix had become the attention of many researchers in order to improve overall properties of PLA. Many efforts had been done to improve the characteristics of PLA in order to compete with low cost and flexible commodity polymers. Various materials had been used to blend with PLA including non-biodegradable polymers and biodegradable materials (Bajpai et al., 2013). A simple way of improving the mechanical, physical and thermal properties of PLA is to add natural fibers, such as banana fiber (Majhi et al., 2010), enzyme modified oil palm fibers (Mamun et al., 2013), sugarcane bagasse residue (Wang et al., 2013) and olive pit powder (Koutsumitopolou et al., 2014).

In general, natural fibers are able to enhance selected mechanical properties (e.g., tensile strength, modulus, impact strength) of a biopolymer (Majhi et al., 2010; Mukherjee & Kao, 2011). Graupner et al. (2009) stated that natural fiber reinforced

PLA had already made its way into market. For instance, biodegradable urns consisting of PLA and flax that were produced by Jakob Winter (Satzung, Germany) by using compression moulding method. Other examples include collaboration research of NEC Corporation and UNITIKA LTD, developing bioplastic composites for mobile phone shells that were made from PLA and 15-20% kenaf fibers. This shows that PLA/natural fiber composites were indeed capable of providing many benefits for our daily activities.

In the field of material science, converting biomass-derived waste into useful and high valued materials is one of the most challenging topics recently (Moubarik et al., 2013). Many natural wastes from plants were used or mixed to prepare different types of material such as biodegradable polymers. One of the examples is the sugarcane bagasse (SCB) that recently gained popularity among material researchers.

Bagasse is a natural residue that remains after sugarcane stalks are crushed to extract their juice. The fact that SCB is often used as an ideal raw material is because it can be easily obtained without much costing since they are wastes. The components of SCB had been investigated and result showed that there are cellulose, hemicellulose, lignin, ash and wax in the bagasse (Loh et al., 2013). These compositions make SCB an ideal filler to be added to biopolymer such as PLA. PLA is considered as 'green' and eco-friendly polymeric material. Thus, hybridization of PLA and SCB is targeted to produce a 'green' composite and will also be able to improve the physical and chemical properties of PLA composites.

1.2 Problem statement

Although the bioplastics market is growing rapidly, the price of PLA is still relatively high. Incorporating natural resources (as well as waste from natural resources) into the matrix of PLA can be an alternative to reduce the cost of production where lesser PLA will be used. In this research, alkali treated sugarcane bagasse fiber (SCBF) and sugarcane bagasse fiber powder (SCBFP – which produced by grinding SCBF into powder form) was added into PLA. The SCBF was selected because of its high availability. Besides reducing the production cost of PLA composites, using SCBF as filler material can also help to solve sugarcane bagasse waste problem. In other words, converting waste into value added product.

Brittleness is one of the reason that limits the application of PLA (Bajpai et al., 2013). Although PLA/SCBF composite is a good candidate for the development of ‘green’ composites, the brittleness and thermal stability of the PLA composites need to be concerned. Toughening of PLA composites is necessary because impact strength of PLA will be reduced when SCBF is added into PLA matrix. Similar results were obtained by Nuthong et al. (2013) where addition of bamboo fiber, vetiver grass fiber and coconut fiber reduced the impact strength of PLA. Thus, toughening of PLA/SCBF composites is an important task, in order to widen the application of these green composites.

1.3 Research objectives

- (a). To determine the effects of sugarcane bagasse fiber (SCBF) loading on the mechanical, thermal and morphological properties of PLA.
- (b). To compare the effects of SCBF and sugarcane bagasse fiber powder (SCBFP) on the impact and thermal properties of PLA.
- (c). To improve the impact strength and thermal properties of PLA/SCBF composites by the addition of epoxidized soybean oil (ESO).

1.4 Research scopes

In this study, sugarcane bagasse fiber (SCBF) was first treated with alkali. Then the SCBF was grounded into powder form (SCBFP). The PLA/SCBF (and PLA/SCBFP) composites were prepared using internal mixer followed by compression molding. The SCBF loading was increased in the range of 5, 10 and 15 wt%. The effects of SCBF and SCBFP on the impact strength and thermal properties of PLA are studied. In order to increase the impact strength of PLA/SCBF composites, epoxidized soybean oil (ESO; at 5, 10, and 15%) is introduced. ESO has long been used as plasticizer for polyvinyl chloride (PVC) compounds and chlorinated rubbers (Ali et al., 2008). Thus, it is expected that the ESO could also be used to improve the impact properties of PLA/SCBF composites. The impact strength of the PLA composites is studied using Charpy impact tester. The thermal properties of the specimens are investigated using differential scanning calorimeter (DSC), thermogravimetric analyzer (TGA) and dynamic mechanical thermal analyzer (DMA). The morphological properties of the PLA composites are examined by field-emission scanning electron microscopy (FESEM).

1.5 Thesis structure

Chapter 1 introduces the development of PLA green composites. The problem statement, scope of study and structure of thesis are included in this chapter.

Chapter 2 discusses the literature review of this study. The influence of different parameters (example: types of fiber, fiber loading and length) were being studied and discussed based on research done by other researchers.

Chapter 3 provides the information about the material and method used in this study. The general description of the sample characterization methods such as impact test, morphology assessment thermal analysis are given in this chapter.

Chapter 4 focuses on the experimental result and discussion. Further elaborations on the effects of SCBF and SCBFP on the impact and thermal properties of PLA, as well as the influence of ESO on the properties of PLA/SCBF (and PLA/SCBFP) composites were provided in this chapter.

Chapter 5 summarizes the significant findings in this study. Suggestions for future studies were also recommended.

CHAPTER 2

LITERATURE REVIEW

2.1 Green composites

The usage of natural fibres in thermoplastics and thermosets is now becoming a growing trend among composite manufacturers (Faruk et al., 2012). The term ‘green composite’ means composites fabricated by natural fibers or crops, which are eco-friendly (John & Thomas, 2008). Besides environmental friendly, the other advantages of green composites include the abundant availability of natural fiber, low cost and higher bio-degradability. In general, they can decompose on its own without using additional energy or resources.

A review done by Dicker et al. (2014) summarises the major properties and also certain potential applications of green composites. In the review, they concluded that green composites have good mechanical properties (e.g., good strength and stiffness), environmental friendly (renewable, biodegradable, low embodied energy, non-toxicity), low cost, and biocompatibility. However, some of the disadvantages of green composites need to be mentioned, e.g., high water absorption, low durability, and low toughness. Hence, there are still room for research and development on the properties of green composites so that they can be put into good use in various industries and applications.

2.2 Biodegradable polymers

Since environmental preservation became an important issue, biodegradable materials are starting to gain popularity ranging from consumers to researchers. Biodegradable technologies have then skyrocketed in just a few years and many materials with biodegradable properties had been produced. Such materials include biodegradable

polymers that were derived from renewable resources. There are four families of biodegradable polymers, where the first three families are produced from biomass. As for the fourth family, they are obtained from fossil origin (see Figure 2.1). First family consists of agro-polymers (e.g. polysaccharides) derived from biomass by fractionation. For the second and third families, they are polyesters, respectively produced by fermentation from biomass or from genetically modified plants (e.g. polyhydroxyalkanoate (PHA)) and by synthesis from monomers obtained from biomass (e.g. polylactic acid (PLA)). Lastly, the fourth family are polyesters, fully manufactured by the petrochemical process (e.g. polycaprolactone (PCL); polyesteramide (PEA); aliphatic or aromatic copolyesters) (John & Thomas, 2008).

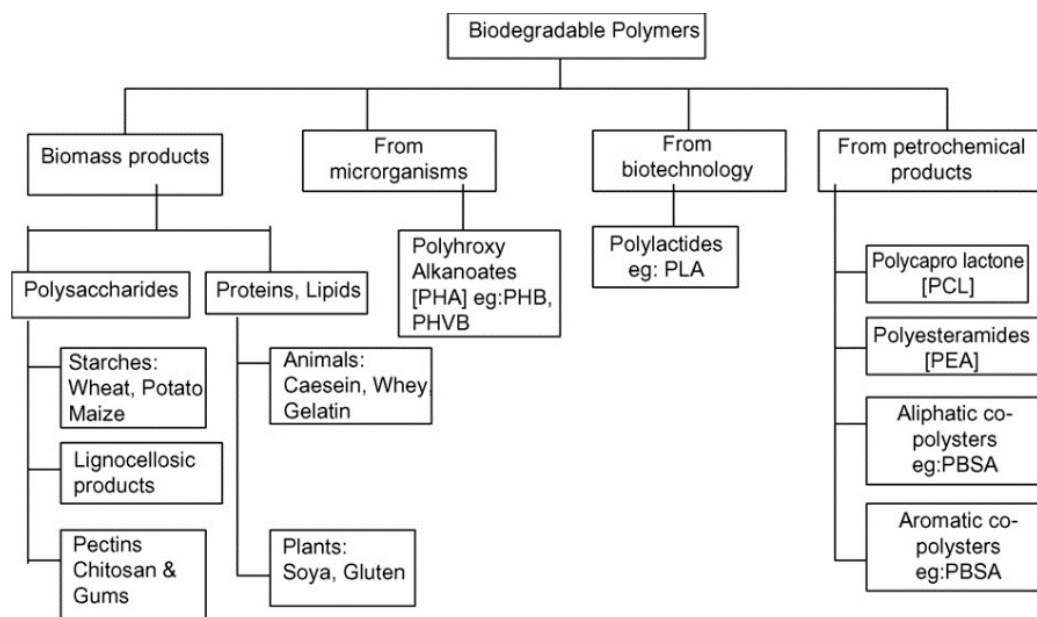


Figure 2.1: Classification of biodegradable polymers (John & Thomas, 2008).

Many of these biodegradable polymers can be found and obtained commercially. With a large range of promising properties, biodegradable polymers are capable of competing with non-biodegradable polymers in various industrial fields such as packaging and biomedical devices. This leads to a vast market and opportunity

for polymer manufacturers to explore the advantage and benefits of biodegradable polymers. As seen in Figure 2.2, the global market of biodegradable polymers in 2010 was 771 million pounds. This amount is expected to increase significantly to nearly 932 million pounds in 2011 and will then further increase to 2.5 billion pounds in 2016. These data shows the usage of biodegradable polymers had already been accepted and applied in various industries, at a compound annual growth rate (CAGR) of 22% for the 5-year period. At these rate, future of biodegradable materials especially polymers shows great potential to be further developed and applied all over the world.

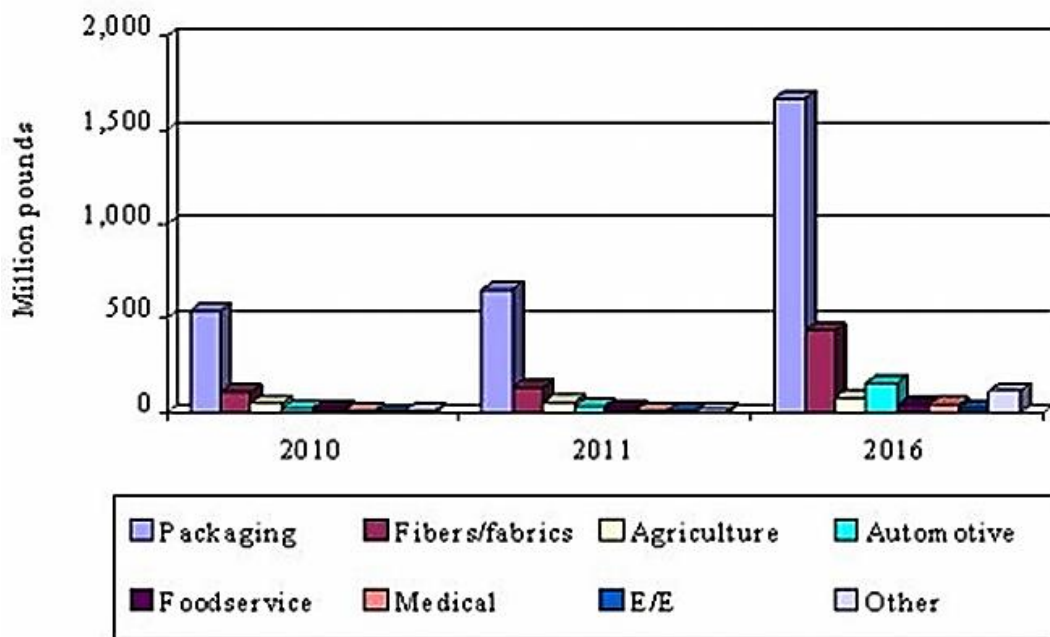


Figure 2.2: Global biodegradable polymer market from year 2010 to 2016 (Ruchika, 2012).