



DEGRADATION BEHAVIOUR OF PINEAPPLE LEAF FIBRE REINFORCED POLY LACTIC ACID UNDER STIMULATED CONDITIONS

This report is submitted in accordance with requirement of the University Teknikal
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by

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee are as follow:

.....
(Dr. Zaleha binti Mustafa)

ABSTRAK

Matlamat utama kerja ini adalah untuk mengkaji tingkah laku degradasi bagi asid laktik poli yang diperkuatkan dengan serat daun nanas di bawah keadaan yang memberangsangkan. Degradasi melalui hidrolisis dan dalam tanah telah digunakan untuk menilai kekuatan tegangan, sifat terma dan analisis morfologi bagi komposit PLA/PALF. Keputusan yang diperolehi menunjukkan bahawa kekuatan tegangan paling tinggi diperolehi oleh 70 wt. % PALF iaitu 209.32 MPa berbanding dengan PLA tulen yang hanya 33.56 MPa. Kekuatan tegangan muktamad turut menurun pada 40 wt. % dan 70 wt. % serat daun nanas bermula dari 143.20MPa ke 69.71MPa dan 209.32 MPa ke 99.64MPa selepas degradasi. Pada masa yang sama, PLA tulen telah gagal untuk menjalankan ujian tegangan disebabkan ia terlalu rapuh selepas didedahkan dengan degradasi hidrolisis. Dalam aspek kelakuan terma, 70 wt. % serat daun nanas mencipta nilai yang paling tinggi iaitu $T_g=66.3^{\circ}\text{C}$, $T_m=158.8^{\circ}\text{C}$ dan crystallinity dengan 17.4% berbanding dengan PLA tulen iaitu $T_g=64.8^{\circ}\text{C}$, $T_m=152.9^{\circ}\text{C}$ dan crystallinity dengan 11.8%. Selepas degradasi, 70 wt. % serat daun nanas berkurang kepada $T_g=65.5^{\circ}\text{C}$, $T_m=153.8^{\circ}\text{C}$ dan 8.9% crystallinity namun 40 wt. % serat daun nanas menurun secara drastik kepada $T_g=32.7^{\circ}\text{C}$, $T_m=137.8^{\circ}\text{C}$ dan 2.6% crystallinity. Bagi degradasi dalam tanah, 70 wt. % serat daun nanas melalui degradasi yang paling pantas diikuti dengan 40 wt. % serat daun nanas dan akhirnya PLA tulen. Kenyataan ini boleh dibuktikan dengan penurunan berat pada 70 wt. % serat daun nanas (5.39%), 40 wt. % serat daun nanas (3.41%) dan PLA tulen (0.51%) pada hari ke-90.

ABSTRACT

The aim of this work was to study the degradation behaviour of pineapple leaf fibre (PALF) reinforced poly lactic acid (PLA) under stimulated conditions. Hydrolytic degradation and soil degradation were used to evaluate tensile strength, thermal properties and morphological analysis of PLA/PALF composites. The result showed that tensile strength is highest at 70 wt. % fibre loading which is 209.32 MPa compared to neat PLA with only 33.56MPa. The ultimate tensile strength were reduced at 40 wt. % and 70 wt. % of PALF which were from 143.20MPa to 69.71MPa and 209.32 MPa to 99.64MPa while the neat PLA was too brittle for testing after hydrolytic degradation. In differential scanning calorimetry (DSC) testing, 70 wt. % PALF composite achieved the highest thermal properties with $T_g=66.3^{\circ}\text{C}$, $T_m=158.8^{\circ}\text{C}$ and 17.4% of crystallinity compared to neat PLA with $T_g=64.8^{\circ}\text{C}$, $T_m=152.9^{\circ}\text{C}$ and 11.8% of crystallinity. After hydrolytic degradation, thermal properties of 70 wt. % of PALF reduced slightly with $T_g=65.5^{\circ}\text{C}$, $T_m=153.8^{\circ}\text{C}$ and 8.9% of crystallinity whereas there is drastically reduction in neat PLA which were $T_g=32.7^{\circ}\text{C}$, $T_m=137.8^{\circ}\text{C}$ and 2.6% of crystallinity. This indicated that PLA and PALF were influenced by hydrolytic process. For the soil degradation, 70 wt. % PALF composite undergoing rapid degradation followed by 40 wt. % PALF and neat PLA. This can be proven by highest weight loss achieved by 70 wt. % PALF (5.39%), 40 wt. % PALF (3.41%) and neat PLA (0.51%) at the end of 90 days.

DEDICATION

I would like to dedicate my appreciation to

My beloved parents,
Honourable supervisor,
Supportive friends.

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LIST OF ABBREVIATION AND NOMENCLATURES

PLA	-	Poly Lactic Acid
PALF	-	Pineapple Leaf Fibre
PMC	-	Polymer Matrix Composite
DSC	-	Differential Scanning Calorimetry
SEM	-	Scanning Electron Microscopy
PP	-	Polypropylene
PE	-	Polyethylene

LIST OF SYMBOLS

Wt. %	-	Weight percentage
°C	-	Celsius
cm ³	-	Centimetre cube
mm	-	Millimetre
MPa	-	Mega Pascal
GPa	-	Giga Pascal

CHAPTER 1

INTRODUCTION

1.1 Background Study

In the past few decades, petroleum-based polymer had dominant the market shares and mostly used by the industries for various applications. Petroleum-based polymer is manufactured from many petrochemicals monomers like ethylene, styrene, vinyl chloride and so on. Petroleum is a non-renewable resources so that the unlimited extraction of petroleum lead to the depletion of petroleum resources. Moreover when the petroleum products such as gasoline are burned for energy, it significantly cause to the happening of the air pollution. According to Leao et al. (2010), only 2% of the 300 million tons of plastic production is originated from plant compared to petroleum or natural gas. Thus, biodegradable polymer seems like a perfect solution to solve these environmental problems.

Among the biodegradable polymers, poly lactic acid (PLA) is the most suitable replacement for petroleum-based polymer. Tawakkal et al. (2014) claimed that PLA is an aliphatic polyester that can be produced either through the carbohydrate fermentation or chemical synthesis of lactic acid monomer. The lactic acid monomer is derived from renewable resources such as corn starch or sugar cane while most of the existing polymers are made from petrochemicals. The production of PLA definitely consumed less energy than the petroleum-based polymer. In addition, PLA is able to degrade into lactic acid and carbon dioxide with the help of biological organisms. Zee (2005) stated that degradation of PLA in

natural environment is affected by various factors such as chemical, biological, temperature, humidity, PH, oxygen and so on. In addition, the advantage of PLA as the thermoplastic is able to recycle back into monomer via thermal depolymerisation or hydrolysis. When purified, the monomer can be applied in the manufacturing of virgin PLA with maintained of its original properties. However due to its low mechanical and thermal properties, it need to reinforce with natural fibres to enhance its performance.

There are many types of natural fibres such as kenaf, flax, jute, coir, hemps, sisal, banana and pineapple leaves which are directly extracted from the agricultural products. Asim et al. (2015) mentioned that pineapple plant is the third important agricultural product in Malaysia after the banana and citrus. Pineapple leaf fibre (PALF) with its low cost, continuous supply and non-toxic properties is very suitable to replace the synthetic fibre in making the polymer composites. This is due to the synthetic fibre is manufactured from the petroleum-based resources and it consumed high energy in making the polymer composites. PALF contained approximately 70-82% cellulose, 5-12% lignin and approximately 1.1% ash. Although the density of PALF is almost same with other natural fibres but its tensile strength and stiffness is very high. PALF with their excellent mechanical behaviour and environmental sustainability successfully gained the interest of researchers to use them as the reinforcement at various applications for example mobile phones, automotive and biomedical industries and so on.

In this study, long PALF was used as the reinforced elements in PLA matrix as to produce a fully degradable composite with improved properties.

1.2 Problem Statement

Generally, the current polymer matrix composite (PMC) is made up of petroleum-based polymer. This conventional petroleum-based polymer cause to the depletion of petroleum resources since it is non-renewable. Moreover, the poor management of agricultural wastes is happened due to the peoples only extracted the pineapple plant for its fruits purpose, leaving its leaf as agricultural wastes. Therefore, there are two alternative methods used to manage the wastes either through disposed in the land or burning in the field. For the disposal of agricultural wastes, it contributed to the landfill shortage. However, for the burning of petroleum for energy and burning the agricultural wastes subsequently cause the air pollution.

Poly lactic acid (PLA) as the bio-polymer is an ideal replacement for petroleum based polymer due to its bio-degradable and environmental friendly properties. However, PLA is brittle and low thermal properties which limiting its application. As to overcome these problems, it need to reinforce with PALF in order to enhance its mechanical and thermal properties. In addition, its degradability can be altered by reinforcing the PALF inside its matrices.

For the reinforcement of PALF in PLA matrix, there is a major drawback which is the hydrophilic nature of PALF is difficult to incorporate with the hydrophobic polymer matrices. Therefore, it is overcome by using alkaline treatment in order to remove the pectin from fibre surface. In addition, it is very difficult to produce high volume fibre content using the conventional method due to not wetting and delamination resulting from insufficient flow of the matrix during the fabrication. Thus, this problem can improved by adopting the pre-pregging technique as to coat the fibre with PLA solution prior to hot compression moulding.

1.3 Objectives

The objectives of this research are:

- i. To investigate the effect of fibre loading on the hydrolytic degradation of PALF-PLA composite.
- ii. To evaluate the effect of the hydrolytic degradation on the thermal characteristic of PALF-PLA composite.
- iii. To characterize the soil degradation behaviour of PALF-PLA composite.

1.4 Scope

The scope of this study was to investigate the degradation behaviour of PLA with various fibre loading (0wt. %, 40wt. % and 70wt. %). Initially, the surface of PALF was modified by using alkaline treatment. For the fabrication of composites, pre-pegging and hot compression moulding were used. The mechanical properties of PLA/PALF bio-composite is characterized using Tensile Test (ASTM D638) and SEM (fracture surface). For the influence of reinforcement loading on the thermal behaviour of PALF-PLA composite, DSC testing was used where the thermal attributes such as T_g, T_m and crystallinity will be measured. Last but not least, degradability of the composite will be evaluated through the hydrolytic and soil degradation.

CHAPTER 2

LITERATURE REVIEW

A literature review on previous research work which is related to this study is presented in this chapter.

2.1 Bio Composites

Bajpai et al. (2014) described that matrix, reinforcement and interphase are the important constituents of bio-composites. A good matrix must be easy to deform when the load is applied, transferred the load onto the fibres and distributed the stress concentration uniformly. The matrix materials can be natural-based polymer or petroleum-based polymer whereas the reinforcement was the component added to improve the mechanical properties of the matrix. The contiguous region (interphase between matrix and fibre) was formed due to different of constituents existed on the matrix system. The properties of the composite was mainly affected by this contiguous region.

Figure 2.1 shows the classification of bio-composites (Mohanty et al., 2005). Poly lactic acid (PLA) used as the matrix whereas natural fibre as the reinforcement to improve mechanical properties of the bio-composites.

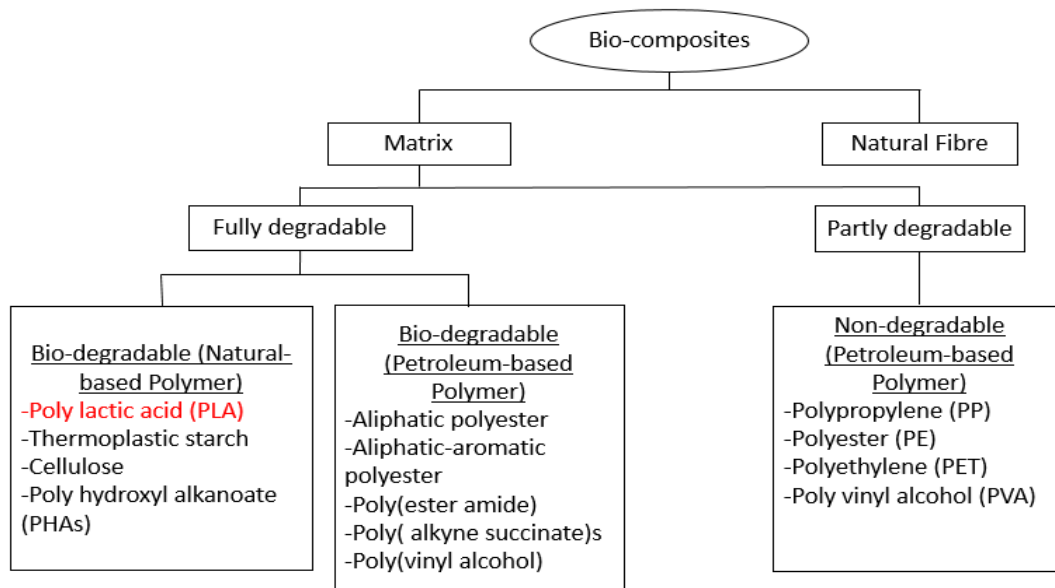


Figure 2.1: Classification of bio-composites (Mohanty et al., 2005).

2.2 Natural Fibre

Natural fibres can be categorized into two groups which are animal fibres and plant fibres. For the plant fibres under the non-wood type, it can be divided into five varieties: straw, bast, leaf, seed/fruits and grass. Fuqua et al. (2012) described that the internal structure and constituents of a plant will influence the physical properties of the fibres. Plant fibres belong to lignocellulosic structures with the constituents like cellulose, hemicelluloses, and lignin, with other small units such as pectin, wax, protein, tannins, ash, and inorganic salts. The constituents are different depend on the source of fibre, growing conditions, age of plant and digestion processes. In general, the cellulose content is responsible for the properties and performance of fibres. However, the growth of non-cellulosic components may decrease the modulus and strength of fibres.

Figure 2.2 shows the classification of natural fibres (Mohanty et al., 2005). Pineapple leaf fibre (PALF) is a non-wood type of plant fibre which can extracted from its leaf.

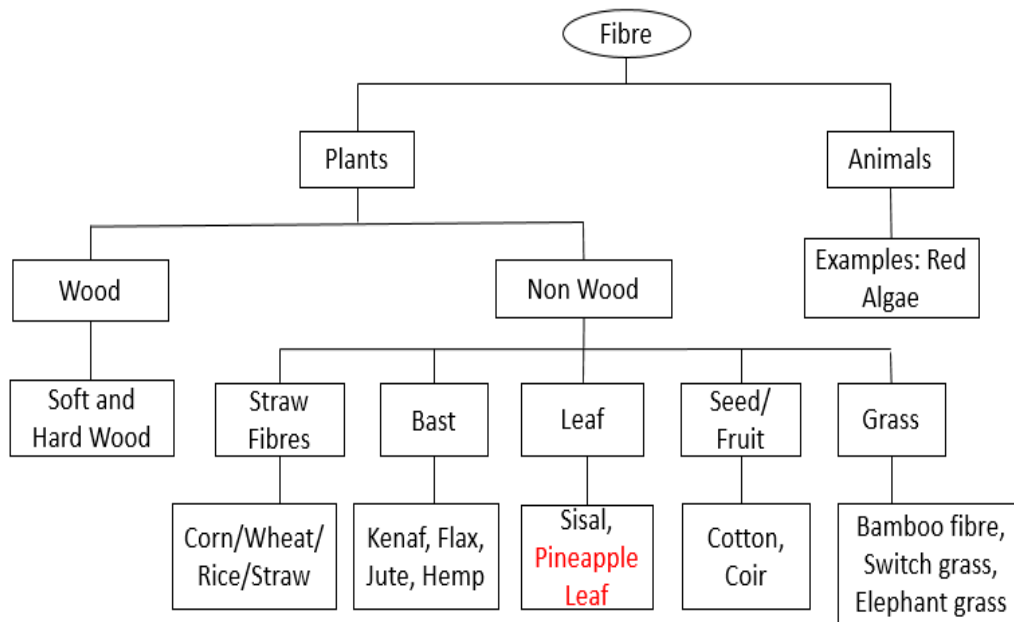


Figure 2.2: Classification of natural fibres (Mohanty et al., 2005).

2.3 Pineapple Leaf Fibre (PALF)

According to Leao et al. (2015), pineapple is an herbaceous perennial monocotyledonous plant and belongs to Bromeliaceae family. It is genera of *Ananas* and *Pseudoananas*; species of *Ananas comosus* (L). The cultivation of pineapples are mainly for its fruits purpose (need approximately 6 to 9 months to bear fruits) leaving the leaves as agricultural wastes. After producing the fruits, its development continues through its base, growing for the following years and producing a new fruit. Moreover, the pineapple plant consists of short and thick stem, in which its leaves grow in narrow, rigid, and axillary roots. The mature pineapple can achieved a height from 1 to 1.2 meters and a width of 0.80 to 1.50 meters.

Repon et al. (2017) mentioned that PALF exhibits with the excellent mechanical properties among all of the natural fibres. Typically PALF consists of 10-82% cellulose, 5-12% lignin and 1.1% ash. The excellent mechanical behaviour are originated from its high cellulose content and low micro-fibrillar angle. Figure 2.3 (a) shows the detail view of