



**PREPARATION AND CHARACTERIZATION OF
LINEAR LOW DENSITY POLYETHYLENE
LLDPE/THERMOPLASTIC STARCH (TPS)/BANANA
FIBER COMPOSITES**

by

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LIST OF ABBREVIATIONS

| | |
|-------|------------------------------------|
| LLDPE | linear low density polyethylene |
| PE | Polyethylene |
| TPS | Thermoplastic starch |
| BF | Banana fiber |
| SEM | Scanning electron microscope |
| DSC | Differential scanning calorimetric |
| TGA | Thermogravimetric analysis |

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Penyediaan dan Pencirian Komposit Polietilena Berketumpatan Rendah Linear/ Kanji Thermoplastik/ Serabut pisang

ABSTRAK

Adunan polietilena linear berketumpatan rendah (LLDPE) dengan kanji termoplastik (TPS) terisi serabut pisang (BF) telah dikaji. Dua jenis adunan disediakan; matriks LLDPE/TPS dengan nisbah adunan yang berbeza, dan komposit LLDPE/TPS (80/20) yang terisi 5-30 wt% serabut pisang. Perubahan morfologi yang diperhatikan melalui mikroskop imbasan elektron (SEM) menunjukkan partikel-partikel TPS tersebar secara sekata di dalam matriks LLDPE. Dalam sistem lain, BF didapati tertanam di dalam fasa TPS, menunjukkan interaksi yang baik di antara fasa BF dan TPS. Pemerhatian ini sejajar dengan nilai modulus Young yang mana meningkat dengan kandungan BF. Peningkatan nilai modulus Young juga disebabkan oleh kesukaran pergerakan rantaian LLDPE/TPS dengan kehadiran BF. Selain itu, kesan kandungan TPS dan BF ke atas sifat-sifat terma matriks LLDPE juga dikaji. Pengukuran dari kalorimeter imbasan pembezaan (DSC) dan analisis termogravimetrik (TGA) membuktikan keberkesanan TPS dan BF dalam meningkatkan tahap degradasi adunan. Komposit LLDPE/TPS/BF menunjukkan kestabilan terma yang lebih baik berbanding adunan LLDPE/TPS, disebabkan oleh kesukaran pergerakan rantaian LLDPE. Kemasukkan BF ke dalam adunan LLDPE/TPS juga didapati menghalang pergerakan rantaian yang meningkatkan kestabilan terma dan memperbaiki kekuatan adunan.

Preparation and Characterization of Linear Low Density Polyethylene LLDPE/Thermoplastic Starch (TPS)/Banana Fiber Composites

ABSTRACT

The blending of linear low density polyethylene (LLDPE) with thermoplastic starch (TPS) and filled with banana fiber (BF) were investigated. Two types of systems were prepared; the LLDPE/TPS matrix with different blend ratio and (LLDPE/TP) (80/20) composites filled with 5 – 30 wt% of BF. Morphological changes using scanning electron microscope (SEM) were observed and it showed that TPS particles are homogeneously dispersed in LLDPE matrix. On the other hand, BF was found to be well embedded in TPS phase, showing the good interaction between BF and TPS phases. This observation shows an agreement with the Young's modulus value which is increased with the increment of BF contents. The increment in Young's modulus value was also attributed to the difficulties in LLDPE/TPS chains movement with the presence of BF. Besides that, the effect of TPS and BF contents on thermal characteristics of LLDPE matrix was also investigated. The measurements from differential scanning calorimetric (DSC) and thermo-gravimetric analysis (TGA), proved the effectiveness of TPS and BF in improving the blend degradation. The LLDPE/TPS/BF composites showed better thermal stability than the LLDPE/TPS blend, which is reflected to the LLDPE chains movement restriction. The incorporation of BF into the LLDPE/TPS blends was found to restrict the chains movement and resulting in more thermally stable and improving the blends toughness.

CHAPTER 1

INTRODUCTION

1.1 Overview

Polymer composite may be defined as materials made up of two or more components and consisting of two or more phases. Such materials must be heterogeneous at least on a microscopic scale (Feng et al, 2008; Yuan et al, 2010). A polymer composite consists of fibers or fillers embedded in or bonded to a matrix with distinct interfaces between the two constituent phases. The matrix must keep fibers or fillers in a desired location or orientation, separating fillers and fibers from each other to avoid mutual abrasion during periodic straining of the composites.

The matrix acts as a load transfer medium in the composites. Since the matrix is generally more ductile than fibers and fillers, it is the source of composite toughness (Reis et al, 2008; Saeoui et al, 2010). The matrix also serves to protect the fibers and fillers from environmental damage before, during and after composite processing.

Linear low density polyethylene (LLDPE) has interesting mechanical properties which provide good tensile properties, dimensional stability and high thermal and chemical resistance. Incorporation of thermoplastic starch (TPS) as disperse phase in LLDPE matrix is due to the high demand in green technology approach. Currently there are many intensive study on the biodegradable plastic based on the natural resources due to the many environmental downsides caused by conventional plastics such as LLDPE, starting from the production of plastics until the problem of waste disposal (Reis et al, 2008; George and Carol, 2008).

Among the concerns for the environmental issues, blending synthetic polymer with a cheap natural biopolymer such as starch, provide new attention to manufacture biodegradability products (Feng et al, 2008; Yuan et al, 2010). Blending polysaccharide with LLDPE polymer could also be the promising alternative to the commonly used conventional filler reinforce LLDPE. The combination of polysaccharide, i.e cellulose and starch with synthetic polymer to produce biodegradable newer materials that are competitive with synthetic polymer reinforced with conventional filler such as glass fibre, glass bead, carbon black, silica, etc, is gaining attention over the last decade.

The advantages of natural filler from the renewable plant source over the conventional fillers are as follows: lightweight, low cost, abundantly available, ease for separation, acceptable mechanical strength and biodegradable (Shin et al, 2010; Brindha et al, 2012). Environmental friendly materials have the potential to be the new materials in the future and could be the partial solution to the many environmental problems (Yuan et al, 2010; Sirisinha et al, 2010). Most of the synthetic polymers are incompatible at molecular level with TPS and leads to poor interfacial adhesion strength. Due to strong intermolecular hydrogen bonding, TPS tend to agglomerate and do not easily disperse in LLDPE matrix. These problems have made the properties of the blends to reduce significantly with increasing TPS content. In order to overcome incompatibility of these blends, the area of study was concentrated on adding banana fiber that can be compensated the deterioration caused by the incorporation of TPS. Therefore, this study performed novel preparation and characterization of linear low density polyethylene LLDPE/thermoplastic starch (TPS)/banana fiber composites for partial biodegradable composite production.

1.2 Biodegradable Materials

A biodegradable material has shown the ability to break down easily by biological means and disappear into the environment without any harm (Vroman and Tighzert, 2009). Putting biodegradable material to polymer blends will help to break up the polymer into small fragments when exposed to microorganisms (Vroman and Tighzert, 2009).

Cellulose and starch are example of many polymers found in nature. Cellulose is made of repeat units of the monomer glucose. This is the same glucose which body metabolizes in order to live, but can't digest it in the form of cellulose. Because cellulose is built out of a sugar monomer, it is called a polysaccharide (Yuan et al, 2010)

Starch is a linear polymer made up of repeating glucose groups linked by glucosidic linkages in the 1-4 carbon positions. The length of the starch chains will vary with plant source but in general the average length is between 500 and 2 000 glucose units. There are two major components in starch namely: amylose and amylopectin. The alpha linkage of amylose starch allows it to be flexible and digestible. Starch-based biodegradable plastics may have starch contents ranging from 10% to greater than 90%. Starch based polymers can be based on crops such as corn (maize), wheat or potatoes. Starch content needs to exceed 60% before significant material breakdown occurs. As the starch content is increased, the polymer composites become more biodegradable and leave less recalcitrant residues. Often, starch-based polymers are blended with high-performance polymers (e.g. aliphatic polyesters and polyvinyl alcohols) to achieve the necessary performance properties for different applications.

Biodegradation of starch based polymers is a result of enzymatic attack at the glucosidal linkages between the sugar groups leading to a reduction in chain length and the splitting off of sugar units (monosaccharide's, disaccharides and oligosaccharides) that are readily utilized in biochemical pathways. At lower starch contents (less than 40%) the starch particles act as weak links in the plastic matrix and are sites for biological attack. This allows the polymer matrix to disintegrate into small fragments, but not for the entire polymer structure to actually bio-degradable.

There are several categories of biodegradable starch-based polymers including:

- Thermoplastic starch products;
- Starch synthetic aliphatic polyester blends;
- Starch PBS/PBSA polyester blends; and
- Starch PVOH Blends.

1.2.1 Blending of Polyethylene with Starch

The blending of polyethylene starch blends is possible to improve the blend properties and characteristics such as yield stress, modulus, impact strength, and biodegradability by choosing an appropriate combination of blends. For typical semi-biodegradable polymer blends, generally it is composed of the combination between polymer matrices, such as from thermoplastic and/or elastomer and another component as biodegradable agent (Sadiku et al, 2014).

Based on the prerequisite characteristics, common semi-biodegradable materials can be classified as bio-composites, where biodegradable agent act as a filler or secondary blends components when biodegradable agent exist in plasticized form. In this section, the detailed descriptions of blend's component especially related to the polyethylene and starch is presented. Polymer blends have played significant role in

the last few decades in providing high performance alternative to pure polymer. The term 'polymer blends' may be defined as a physical mixture of two polymers or two copolymers, giving rise to materials with a range of properties, which could not be delivered by any of the constituents. Polymer blending provides a simple and inexpensive process to combine properties of blend's components and has the advantages of obtaining them in one product. In addition, the properties of blends can be adjusted by simply changing the blends composition (Margaria et al, 2008;Sadiku et al, 2014).

1.3 Problem Statement

Linear low density polyethylene (LLDPE) and starch based product have been received promising attention as partial replacement for petroleum based product. Moreover, most of the plastic products are made from petroleum based plastics which are not biodegradable or discompose naturally. Therefore disposing of these materials will attracts major attention regarding the sustainability issue and demanding continuous developments of producing new biodegradable or semi-biodegradable end products.

However, contrary to their biodegradable behavior, the mechanical properties of starch based blends are quite poor with increasing starch content. This is attributed to the poor interfacial adhesion due to the incompatibility of hydrophilic starch with hydrophobic synthetic polymer (Shin et al, 2010).

Cellulose reinforced composites have shown interesting properties and generally, natural fiber has shown a growth of due to the economic and environmental advantages and the attractive specific properties. Therefore, applying natural fibers in biodegradable material shows great interest since it provides a possible way to produce composites that are degradable at very low cost.

Although natural fibers are abundantly available at low cost, the composite made up from the natural fibers based are not very strong enough to meet the market requirement.

1.4 Objectives

- (1) To identify the best mixing proportion between LLDPE and TPS with acceptable mechanical properties.
- (2) To examine the effect of different banana fiber blends proportion on the mechanical and thermal properties of LLDPE/TPS blends.

1.5 Scope of Study

- (1) Effort will be made to identify the mixing proportion of LLDPE and TPS for better improvement of mechanical, physical and optical properties of LLDPE / TPS / BF composites.
- (2) The properties of different mixture proportion of the blend will be tested for surface morphology, mechanical properties, and thermal properties via scanning electron microscope (SEM), fourier transform infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA) and differential scanning calorimetry (DSC) respectively.
- (3) Justification on optimum condition for the production of the best blend with capable of yielding good mechanical and physical properties will be conducted and be tested using scanning electron microscope (SEM), fourier transform infrared spectroscopy (FTIR), thermal gravimetric analysis (TGA) and differential scanning calorimetric (DSC).

1.6 Thesis Organization

The thesis background which consists of problem statements, objectives and research scope are presented in Chapter 1. Chapter 2 explored the literature on the linear low density polyethylene (LLDPE), thermoplastic starch (TPS) and banana fiber composites. These included some theoretical aspects of molecular structures, beginning with the overviews and then examining some of the significant factors that combine to determine the ultimate various mixing proportions. Chapter 3 discusses the methods and efforts used in undertaking the blends preparation, measurement and characterizations approaches. Chapter 4 discusses the results of characterization of the composites and present the results of numerous measurements intended to help in understanding the properties of materials blends. Chapter 5 offers conclusions and recommendations based on the work presented in the preceding chapters.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter presents the literature reviewed on the current work related to starch and cellulose based polymer blends. The current research is aimed at producing new degradable materials properties which is comparable to their conventional plastic counterparts and coupled with the added advantage of biodegradability.

2.2 Polymer Blends

Polymer blends have played significant role in the last few decades in providing high performance alternative to pure polymer. The term 'polymer blends' may be defined as a physical mixture of two polymers or two copolymers, giving rise to materials with a range of properties, which could not be delivered by any of the constituents (Reis et al, 2008). Polymer blending provides a simple and inexpensive process to combine properties of blend's components and has the advantages of obtaining them in one product. In addition, the properties of blends can be adjusted by simply changing the blends composition (Rohaniet al, 2010).

The development of polymer blends, composites and laminates is a very active area of science and technology; of great economic importance not only for the plastics industry but also for many other industries where the use of such products is becoming increasingly more common (Sadiku et al, 2014). Most pairs of polymers are immiscible with each other. Even worse, in fact that they also have less compatibility and would be required in order to obtain the desired level of properties and performance from their blends, compatibilizers are often used as additives to improve

the compatibility of immiscible polymers and thus improve the morphology and resulting properties of the blend. It is often challenging to disperse fillers effectively in the matrix polymer of a composite, or to adhere layers of polymers to each other or to other substrates (such as glass or metals) in laminates (Gad et al, 2010). Continued progress in the development of compatibilization technologies is, hence, crucial in enabling the polymer industry to reap the full benefits of such approaches to obtaining materials with optimum performance and cost characteristics.

The improvement of polymer blends and composites qualities is the great economic importance for the plastics industry and for other industries the use of such products is becoming increasingly. Advanced polymer modification techniques have grown in importance during the last two decades as the "point of diminishing returns" has been approached in improving the performance price balance by altering just the chemical structures of polymers.

The most important polymer modification techniques are blending dissimilar polymers, preparing composites where a matrix polymer is modified by fillers. The objective is to seek synergies between the components so that one can attain better performance without increasing cost or maintain acceptable performance at lower cost.

Polymeric are polymers that can be used (normally in small percentages) as additives to help assemble dissimilar components into polymer blends, composites and laminates with improved properties. These more attractive properties generally result from phase separation on a finer scale (microscale or even better nanoscale, instead of macroscale) along with stronger interconnections between phase domains. Impact modification (toughening) is one major benefit that can often be attained by using polymeric compatibilizers. It can be inferred from the anticipated continued growth of

markets for polymer-based heterophasic products that polymeric compatibilization technologies will also continue to grow in importance (Feng et al, 2008; Derrien et al, 2009).

2.2.1 Linear Low Density Polyethylene (LLDPE)

Polyolefin are polymer built from high molecular weight hydrocarbons. Polyolefins include linear low density polyethylene, low density polyethylene, high density polyethylene, polypropylene copolymer, polypropylene, and polymethyl pentene. These polymers are the only plastics that have a lower specific gravity than water. When ethylene is polymerized the result is relatively straight polymer chains. From the main chain they can branch out. There are different kinds of polyethylene from the varying degree of branching in their molecular structure. The figure 2.1 to 2.3 showed the brief descriptions on how the polyethylene differs from each other. Low density polyethylene (LDPE) has the most excessive branching. This causes the low density to have a less compact molecular structure which is what makes it less dense. It has a density of 0.910-0.925 g/cm³. LLDPE based materials are the ideal choice for applications which require significant material flexibility, in addition to strength and durability, combined with outstanding resistance to low temperatures and ultraviolet exposure.



Figure 2.1: Illustrative structure representative of LDPE (Durmus et al, 2007).

HDPE (high density polyethylene) has minimal branching of its' polymer chains and give rigid properties and less permeable when compared to LDPE. It has a density of 0.941-0.965 g/cm³ as shown in Table 2.1.



Figure 2.2: Illustrative structure representative of HDPE (Durmus et al, 2007).

LLDPE (linear low density polyethylene) has a significant numbers of short branches. Because it has shorter and more branches its' chains are able to slide against each other upon elongation without becoming entangled, like LPDE. This gives LLDPE higher tensile strength and higher impact and puncture resistance than the LDPE. It has a density of 0.91-0.94 g/cm³.



Figure 2.3: Illustrative the structure representative of LLDPE (Durmus et al, 2007).

Table 2.1: The mechanical and thermal properties of the LLDPE (Feng et al, 2008).

| LLDPE Material Properties | Properties | Units |
|-----------------------------------|---------------|-------------------------------|
| Melt Index | 8-1.2 | g/10min |
| Density | 0.93 | g/cm ³ |
| Mold Shrinkage | 0.020 - 0.022 | in/in |
| Tensile Strength | 1900-4000 | psi |
| Tensile Modulus | 25-.5 Gpa | % |
| Ultimate Tensile Elongation | 850 | psi |
| Elastic Modulus in flex | 160000 | Shore D |
| Hardness | D48-D56 | kJ/m ² |
| IZOD notched or un-notched | 1.86 | |
| Deflection Temperature | 45 | °C |
| Dielectric Strength | 2500 | V/mil |
| UL Listing | HB | |
| Thermal Conductivity | -2.55 | BTU*in/hr*ft ² *°F |
| Heat Capacity | 0.52 | BTU/lb*°F |
| Coefficient of Friction | 0.55 | % |
| Crystallinity | N/A | °C |
| Conditions Favoring Crystallinity | 133 | °C |
| Crystalline Melt Temp | 120-160 | °C |
| Glass Transition Temp | 119 | °C |
| Service Temp range | 160-232 | °C |
| Degradation Temp | 250 | °C |
| Viscosity at TM | - | |

2.3 Starch

Starch is the second most abundant natural polymer, after cellulose. It has a low cost and can be easily extracted from a large variety of plants. Starch is fully biodegradable, like all natural polymers, can be used in many applications for the materials or filler. Starch-containing plastics can be used to make capsules, coatings, garbage bags, flower-pots and many disposable objects. After use, such objects can be disposed of by biodegradation. Both starch and cellulose consist of glucose molecules linked together to form long chains. Starch is the main form of energy storage of plants; cellulose is the main component of wood. Starch is extracted in form of granules that have different shape and size depending on its origin.

Though it is highly hydrophilic, starch is not soluble in water. Differently from cellulose, starch can be found in industrial applications as a thermoplastic polymer. When processed with proper treatments it turns to a true thermoplastic that can be further processed with traditional techniques. The main drawback of starch is its high sensitivity to moisture that renders it not suitable for many applications. Combination of starch with traditional, synthetic plastics allowed overcoming this problem, leading to new materials with properties similar to the main plastics that fulfil the market demand. Such "mixed" plastics may contain as much as 50% of starch.

It can easily be processed and during processing, amylose and amylopectin degradation occurs and this phenomenon is obviously dependent on the thermal and mechanical energy brought to the system. Numerous studies were conducted to determine the degradation mechanism and to understand the contribution of each parameter. Since starch is a hydrophilic material, water is the best plasticizer.

Nevertheless, the water content and thus the plasticized starch properties are strongly dependent on the storage conditions (temperature and atmosphere relative humidity). This drawback is partially solved with the use of less volatile plasticizers, which present lower plasticization efficiency. These compounds bearing hydroxyl groups, can interact with the starch chains through hydrogen bonds. Glycerol is the most common plasticizer.

2.3.1 Chemical Structure of Starch

Starch is a very important and widely distributed natural product, occurring in the leaves of green plants, seeds, fruits, stems, roots, and tubers. It serves as the chemical storage form of the energy of the sun and is the primary source of energy for the organisms on the earth. Starch is composed of two kinds of polysaccharides, amylose and amylopectin, exclusively composed of d-glucose residues with α -(1 \rightarrow 4) linkages in a linear amylose and α -(1 \rightarrow 4) linkages and \sim 5% α -(1 \rightarrow 6) branch linkages in amylopectin, both combined in a water-insoluble granule that is partially crystalline and whose size, shape, and morphology are dependent on its biological source. Chemically, starch is composed of two different molecules, amylose and amylopectin. In amylose, the glucose molecules are linked in a fashion; however, the tetrahedral chemistry of carbon (and the bond angles that result from this chemistry) gives amylose an overall spiral shape as shown Figure 2.4

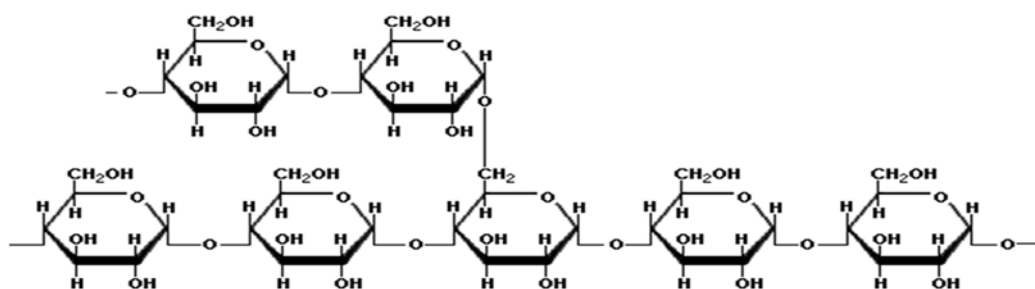


Figure 2.4: Structure of a starch (amylopectin) (Serge Perez and Eric Bertoft ,2010).