

FABRICATION AND CHARACTERIZATION OF BIODEGRADABLE  
COMPOSITE FILM FROM BANANA STEM

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requirements for the award of the degree of  
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I declare that this thesis entitled “*Fabrication and characterization of biodegradable composite film from banana stem*” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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## **ABSTRACT**

The diverse utilization of packaging films from bio-based compounds has received so much attention lately due to the fact that they are readily biodegradable. Banana stem fiber was subjected to acid hydrolysis and three types of film samples, banana stem fiber-chitosan, cassava starch-chitosan and banana stem fiber-cassava starch-chitosan were fabricated with the addition of PEG400. The film samples were later characterized in terms of their morphological and physical properties through FTIR, TGA, DSC and AFM. Analytical results showed that the three compounds used were almost identical in structure and therefore the miscibility between them was of considerable degree. Results also showed that the thermal stability of the three films was significantly noteworthy to be used as a packaging material. The addition of bio-fibers also affected the thermal and mechanical properties of the film samples. Thus, this study gave a new in-depth look into the usage of biofibers as reinforcing agents of biodegradable films of low thermal and mechanical properties.

## ABSTRAK

Penggunaan filem pembungkusan mudah terbiodegradasi yang diperbuat daripada bahan biologi telah menerima perhatian yang meluas baru-baru ini. Serat batang pisang dihidrolisis melalui asid hidrolisis dan tiga jenis sampel filem dihasilkan iaitu serat batang pisang-chitosan, tepung ubi kayu-chitosan dan serat batang pisang-tepung ubi kayu-chitosan dengan campuran PEG400. Sampel filem tersebut kemudiannya dianalisis morfologi dan kualiti fizikal mereka melalui FTIR, TGA, DSC dan AFM. Keputusan analitikal menunjukkan ketiga-tiga bahan yang digunakan mempunyai struktur yang sangat identikal maka kebolehlarutan di antara ketiga-tiga bahan tersebut adalah agak tinggi. Keputusan juga menunjukkan kestabilan haba ketiga-tiga sampel filem tersebut adalah sesuai dengan penggunaan mereka sebagai filem pembungkusan. Penambahan serat tumbuhan juga memberi impak kepada kualiti haba dan mekanikal sampel-sampel filem tersebut. Oleh yang demikian, kajian ini memberikan satu pendedahan baru kepada penggunaan serat tumbuhan sebagai agen penguat untuk biofilem yang mempunyai kualiti haba dan mekanikal yang rendah.

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**LIST OF ABBREVIATIONS/SYMBOLS**

%	-	percentage
<	-	less than
>	-	more than
°C	-	degree celcius
µm	-	micrometer
10 <sup>-5</sup>	-	0.00001
10 <sup>-7</sup>	-	0.0000001
ABO	-	blood group system
AFM	-	Atomic Force Microscopy
alpha-Gal	-	alpha-Galactosidase A
ATR	-	attenuated total reflectance
cm	-	centimeter
CO <sub>2</sub>	-	carbon dioxide
DA	-	degree of N-acetylation
DD	-	degree of deacetylation
DDA	-	degree of deacetylation
DNA	-	deoxyribonucleic acid
DRR	-	disease resistance response
DSC	-	Differential Scanning Calorimtry
<i>et al.</i>	-	et alii/and others
etc.	-	etcetera
FDA	-	Food and Drug Administration of the USA
FTIR	-	Fourier Transform Infrared
g	-	gram
H <sup>+</sup>	-	hydrogen ion



H <sub>2</sub> O	-	water
H <sub>m</sub>	-	heat of melting
Hz	-	hertz
i.e.	-	<i>id est</i> /that is
IR	-	infrared
J.g <sup>-1</sup>	-	joule per gram/unit for energy
kHz	-	kilohertz
LCD	-	liquid crystal display
M	-	molar
mg	-	milligram
mL	-	milliliter
mL/min	-	milliliter per minute
N/m	-	newton per meter
NCMC	-	N-carboxy-methylchitosan-N,O-sulfate
nm	-	nanometer
O <sub>2</sub>	-	oxygen
O-GlcNAc	-	O-linked N-acetylglucosamine
pH	-	negative logarithm for hydrogen ion concentration
PoP	-	point-on-purchase
PR	-	pathogenesis-related gene
R	-	replicate gene
RH	-	relative humidity
rms	-	root-mean-square
RPM	-	revolution per minute
T <sub>c</sub>	-	conclusion temperature
T <sub>c</sub>	-	conclusion temperature
T <sub>g</sub>	-	glass transition temperature
TGA	-	Thermogravimetric Analysis
T <sub>m</sub>	-	melt transition temperature
T <sub>o</sub>	-	onset temperature
T <sub>o</sub>	-	oxidation temperature
T <sub>onset</sub>	-	onset temperature
T <sub>p</sub>	-	peak temperature

$T_p$	-	peak temperature
v/v	-	volume per volume
w/w	-	weight per weight
$\alpha$	-	Alpha – glycoside link
$\beta$	-	Beta – glycoside link
$\Delta H$	-	enthalpy

## CHAPTER 1

### INTRODUCTION

#### 1.1 Research background

Almost the entire available consumer products have been dispensed through packaging system. This system is greatly utilized to fulfill at least one of the listed functions below (Davis and Song, 2005):

- a) to provide product protection from physical damage, contamination and deterioration;
- b) to give a product the sales appeal;
- c) to ensure that the product identity is easily recognizable;
- d) to give information about the product
- e) to optimize distribution and storage costs;
- f) to provide consumers with the convenience and safety.

Food packaging preserves and protects all types of foods and their raw materials (Tharanathan, 2003) with which their traceability, convenience, and tamper indication are secondary functions recognizably of increasing importance (Marsh and Bugusu, 2007). These protective films and suitable packaging by the food industry have become an ongoing topic of monumental interest because of their packaging potentiality attributed to the ability in increasing the shelf life of many food products (Sorrentino *et al.*, 2007). By means of the correct selection of materials and packaging technologies, it is able to keep the product's quality and freshness during the time required for its commercialization and most importantly, its consumption (Stewart *et al.*, 2002).

In recent years, bio-based, materials such as carbohydrates and proteins have, gradually if not extensively, been tested and experimented to develop biodegradable films which had been proven to have more and more versatile properties (Perez-Mateos *et al.*, 2009). Also, natural fibers present important advantages such as low density, appropriate stiffness and mechanical properties and high disposability and renewability. Moreover, they are recyclable and biodegradable. There has been lot of research on use of natural fibers in reinforcements (Mukhopadhyay *et al.*, 2008). Natural fibres are getting the attention as a reinforcing agent in both thermoplastic and thermosett matrices (Pothan *et al.*, 2006). This has indefinitely set off the diverse utilization of food packaging films made of bio-based materials.

## 1.2 Identification of problems

Global production of packaging materials is estimated at more than 180 million tons per year, spurred by the fact that both growth and demand are increasing annually. Within the plastic packaging market, food packaging is the largest growing sector (Cutter, 2006). For the last 20 years, petrochemical polymers, commonly called “plastics,” have been booming and are by far the most widely used polymers for packaging due in part to their high performance, low cost (Callegarin *et al.*, 1997), availability in large quantities at low cost and favorable functionality characteristics, such as good tensile and tear strength, good barrier properties to oxygen and heat-sealing capabilities (Alves *et al.*, 2006).

Indefinitely, plastics have indeed gained a unique position in food packaging technology for a number of quite different reasons including (Psomidaou *et al.*, 1997) :

- a) higher strength, elongation and barrier properties against waterborne organisms responsible for food spoilage,
- b) lower cost and higher energy effectiveness,
- c) lightness and water resistance.

They are also incredibly durable and inert even in the presence of microorganisms, leading to a sustainable long-term performance (Mali *et al.*, 2002; Arvanitoyannis *et al.*, 1998). Until as recent as today, the largest part of all materials used in the packaging industries is derived from fossil fuels and practically non-biodegradable (Sorrentino *et al.*, 2007; Ban *et al.*, 2006). These traditional packaging materials also encourage the migration of harmful additives (Lopez-Rubio *et al.*, 2006) into food products.

As the amount of plastic waste increases every year, the exact time needed for its biodegradation is unknown (Reis *et al.*, 2008). Approximately 40 million metric tons of such films are consumed annually on a global basis (Ban *et al.*, 2006). The world is also running out of landfill space as degradation of plastics requires a long time and most of them end up overburdening on landfill (Xu *et al.*, 2005).

Waste is not confined only to plastic materials. According to Abdul Khalil *et al.* (2006), Malaysia has a large area of plantation of oil palm (3.87 million hectares), coir (147 thousand hectares), banana (34 thousand hectares), and pineapple (15 thousand hectares). Large quantities of cellulosic and non-cellulosic raw material are generated during harvesting (Abdul Khalil *et al.*, 2006). The explosive expansion of these plantations in Malaysia has generated enormous amounts of plant wastes, creating problems in replanting operations and tremendous environmental concerns.

Packaging materials, especially for food products or produce, like any other short-term storage packaging materials, therefore represent a serious global environmental problem (Kirwan and Strawbridge, 2003) if no concerted actions are adopted to address and prevent it.

### **1.3 Significance of study**

A big effort to extend the shelf life and enhance food quality while reducing packaging waste has encouraged the exploration of new bio-based packaging

materials, such as edible and biodegradable films from renewable resources (Tharanathan, 2003) for the goal of food packaging is to contain food in a cost-effective way that satisfies industry requirements and consumer desires, maintains food safety, and minimizes environmental impact (Marsh and Bugusu, 2007). Since the depletion of oil, societal and environmental pressures continue to prompt efforts to develop renewable, cost-effective, and environmentally friendly materials for the manufacture of a number of products, including these films (Ban *et al.*, 2006).

Hence, at present, one of the major trends in the food packaging field is the development and use of polymeric materials of biodegradable and/or edible nature that decompose naturally causing no environmental problems when discarded as waste and can also be considered an alternative to traditional plastics obtained from petrochemical industry (Muratore *et al.*, 2005). This notable growth of interest in developing packaging materials based on biopolymers has been witnessed as early as the last decade (Mendieta-Taboada *et al.*, 2008).

Biological Degradation	- Fungi, Bacteria, Insects, Termites
Enzymatic Reaction	- Oxidation, Hydrolysis, Reduction
Chemical Reactions	- Oxidation, Hydrolysis, Reduction
Mechanical	- Chewing
Thermal Degradation	- Lightning, Sun, Man
Pyrolysis Reactions	- Dehydration, Hydrolysis, Oxidation
Water Degradation	- Rain, Sea, Ice, Dye
Water Interactions	- Swelling, Shrinking, Freezing, Cracking, Cyclic Wetting and Drying
Weather Degradation	- Ultraviolet radiation, Water, Heat, Wind
Chemical Reactions	- Oxidation, Hydrolysis
Mechanical	- Erosion
Chemical Degradation	- Acids, Bases, Salts, Metals
Chemical Reactions	- Oxidation, Reduction, Dehydration, Hydrolysis
Mechanical Degradation	- Dust, Wind, Hail, Snow, Sand - Stress, Cracks, Fracture, Abrasion

Figure 1.1: Degradation reactions which occur when bio-based resources are exposed to nature (Rowell, 1998).

The search for biologically active compounds from natural sources has taken the center stage in recent years for their low or absent toxicity, their complete biodegradability, their availability from renewable sources, and, their low-cost if

compared with those compounds obtained by total chemical synthesis (Tringali, 2001). Also, the abundance of natural fibres combined with the ease of their processability is an attractive feature (Pothan *et al.*, 2006). The incorporation of these plant fibers which are mostly residues of agriculture and agro-industries, allows a valorization of these wastes and a limitation of environmental damages. It had been demonstrated that natural fibers can reinforce concrete and exhibit the same performance behavior as that of conventional fiber reinforced concrete produced from steel and other inorganic/synthetic fibers (Bilba *et al.*, 2007).

Starch is the commonly used agricultural raw material, since it is a renewable source (Zhai *et al.*, 2004). In the food packaging sector, starch-based material has received great attention owing to its biodegradability, wide availability and low cost (Avella *et al.*, 2005). Starch owes much of its functionality to the two major high-molecular-weight carbohydrate components, amylose and amylopectin, as well as to the physical organization of these macromolecules into the granular structure (Romero-Bastida *et al.*, 2005).

Chitosan is recognized for its antimicrobial activity and film-forming properties (Sebastien *et al.*, 2006) besides its biocide effects (Fernandez *et al.*, 2008). In addition, chitosan also possesses useful properties such as biodegradability, biocompatibility (Sashiwa *et al.*, 2003), and non-toxicity leading to extensively use over a wide range of applications (Bangyekan *et al.*, 2006).

The scope of films made with starch combined with other polysaccharides was widened to include chitosan for several reasons. First, chitosan is a biopolymer, obtained by N-deacetylation of chitin, which is the second most abundant polysaccharide on the earth after cellulose (Bangyekan *et al.*, 2006). It is commercially available from a stable renewable source, that is, shellfish waste (shrimp and crab shells) of the sea-food industry. Second, chitosan forms good films and membranes (Vandamme *et al.*, 2002). Since the use of synthetic polymers is dependent on the use of crude oil, nature has been touted as another possible resource for structural polymers (Jansson and Thuvander, 2004).

## 1.4 Objectives

The objectives of this study are:

- a) To fabricate different types of biodegradable composite films from banana stem fiber.
- b) To characterize different types of biodegradable composite films from banana stem fiber.

## 1.5 Scopes of study

The scopes of this study are:

- a) Film preparation:
  - i. Banana stem fiber-chitosan film
  - ii. Cassava starch-chitosan film
  - iii. Banana stem fiber-cassava starch-chitosan film
- b) Film characterization:
  - i. Morphological properties using AFM (Atomic Force Microscopy)
  - ii. Physical properties tests using FTIR (Fourier Transform Infrared) spectroscopy, TGA (Thermal Gravimetric Analysis), and DSC (Differential Scanning Calorimetry)