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Fabrication and Characterization of Sawdust Composite Biodegradable Film

M.Z. Norashikin and M.Z. Ibrahim

Abstract**—**This report shows the performance of composite biodegradable film from chitosan, starch and sawdust fiber. The main objectives of this research are to fabricate and characterize composite biodegradable film in terms of morphology and physical properties. The film was prepared by casting method. Sawdust fiber was used as reinforcing agent and starch as polymer matrix in the casting solution. The morphology of the film was characterized using atomic force microscope (AFM). The result showed that the film has smooth structure. Chemical composition of the film was investigated using Fourier transform infrared (FTIR) where the result revealed present of starch in the film. The thermal properties were characterized using thermal gravimetric analyzer (TGA) and differential scanning calorimetric (DSC) where the results showed that the film has small difference in melting and degradation temperature.

Keywords**—**Sawdust, composite, film, biodegradable.

I. Introduction

In food industry, it is important to extend the storage and protect quality of food. Currently, food technology research concerns on preservation and protection of all types of foods and their raw materials from oxidation and microbial spoilage. Artificially synthesized additives or preservatives normally have been added to food directly or indirectly to improve the stability of food. These additives can cause bad effect to the health once consumed.

Petrochemical based plastics have been increasingly used as packaging materials because of their availability in large quantities at low cost and have good tensile and tear strength, good barrier properties to oxygen and aroma compounds and high heat stability [1]. In contrast, they have very low water vapor transmission rate and most importantly they are totally non-degradable and therefore lead to environmental pollution which pose serious ecological problems. Hence, their use in any form or shape has to be restricted and may be even gradually abandoned to circumvent problems concerning waste disposal.

The use of plastics in cooking and food storage can carry health risks especially when hormone disrupting chemicals from some plastics leach into foods and beverages. Plastic manufacturing and incineration creates air and water pollution and exposes workers to toxic chemicals. When plastic waste end up in the ocean, it cause death to the marine wildlife like turtles and lion seas. Plastic wastes also cause water pollution.

There is a paradigm shift imposed by the growing of environmental awareness looking for packaging films which are biodegradable and therefore compatible with the environment. In a sense, biodegradability is not only a functional requirement but also an important environmental attribute. Thus, the concept of biodegradability enjoys both user-friendly and eco-friendly attributes and raw materials are essentially derived from either replinishable agricultural waste or marine food processing industry wastes and therefore it capitalizes on natural resource conservation with an underpinning on environmentally friendly and safe atmosphere [2].

Designing biodegradable packaging alternatives and ensuring that they end up in an appropriate disposal system can be used to protect the environment [3]. An additional advantage of biodegradable packaging materials is that on biodegradation or disintegration and compositing because they may act as fertilizer and soil conditioners, facilitating better yield of the crops. Though it is a bit expensive, biodegradable packaging is tomorrow's need for packaging especially for a few values added food products. Also, there is an urgent need for a method of extending food storage time without using a preservative.

In order to overcome the foregoing problems, research for developing antibacterial packaging film is being actively conducted. It is general that the antibacterial packaging film provides antibacterial property to the packaging material according to types of added antibacterial substances and preparations methods. Particularly, the antibacterial effect, maintenance and properties of the packaging material can vary depending on interaction between the used antibacterial substances and high molecules, which is the main component of the film construction [4].

Chitosan is a β-1,4-linked polymer of glucosamine (2-amino-2-deoxy-β-D-glucose) and lesser amounts of *N*-acetylglucosamine. It is formed by the deacetylation of chitin (poly-*N*-acetylglucosamine), an abundant byproduct of the crab and shrimp processing industries [5].

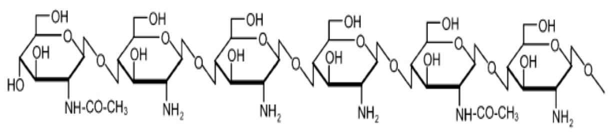

Fig. 1 Chemical Structure of Chitosan

Norashikin binti Mat Zain is with Universiti Malaysia Pahang .Malaysia. e-mail:shikin@ump.edu.my

According to Se'bastian *et al.* (2006), chitosan is non-toxic, biodegradable and biocompatible polymer [6]. Chitosan widely existing in the nature and has antibacterial effect, heavy metal adsorption effect, antioxidation effect and film formability. Also, it is a functional substance showing various functions that a food packaging film needs, such as stabilization of blood pressure, enhancement of immunity, ability of adsorption and excretion of fat and cholesterol, water retention ability and anti-tumor effect and thus, is suitable as a material for a food packaging film [7]-[9].

Starch is one of the polysaccharides frequently used to develop edible films because it is natural polymer capable of forming a continuous matrix and it is a renewable and abundant resource.

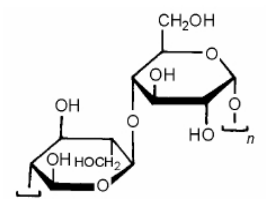

Fig. 2 Chemical Structure of Starch

In particular, cassava starch is a good commercial cash crop and a major source of good quality starch which is cheaper than other starches usually used by the food industry. Edible films formulated with starch and polyethylene glycol has shown good mechanical properties expressed. Starch, major plant storage form of glucose, amylase and amylopectine give stronger, more flexible film. According to the Tharanathan (2001), starch having a thermoplastic property upon the disruption of its molecular structure and enhanced biodegradability by microbial and oxidative degradation [1]. In this study natural materials are used to develop biodegradable film from sawdust.

Fig. 3 Sawdust

II. METHODOLOGY

A. Film Preparation

Cellulose fibers were extracted from sawdust by chemical treatment resulting purified cellulose. Sawdust was soaked in a concentrated sodium hydroxide (NaOH) solution and then after 2 hours it was washed with distilled water. The pre-treated pulp was hydrolyzed by hydrochloric acid (HCl) solution at 80°C for 2 hours and then washed with distilled water repeatedly. The pulp was treated once more with NaOH solution at 80°C for 2 hours. The alkaline treated pulp was washed several times with distilled water until the pH became neutral before being dried at room temperature.

2 gram starch and 100 milliliters distilled water was gelatinized by heating at 90°C. 2 gram chitosan powder was dissolved with 100 milliliters acetic acid and both solution were mixed and stirred until become homogeneous. Then, the solution was added with 2 gram sawdust fiber and 3 milliliters additive. After that the solution was degassed for 24 hours. The solution was poured onto a glass plate and dried at room temperature. The film was carefully removed by peeling from the glass plate.

B. Film Characterization

The film fabricated was characterized by atomic force microscope (AFM), Fourier transform infrared (FTIR), differential scanning calorimeter (DSC), and thermo gravimetric analysis (TGA).

For AFM analysis, surface morphology (2D and 3D topographic images) and roughness analysis of the film in scan area of 2 μm × 2 μm was characterized using atomic force microscope model Shimadzu SPM 9500J2.

Fourier transform infrared spectroscopy (FTIR) identifies chemical bonds in a molecule by producing an infrared absorption spectrum. FTIR spectra was recorded between 400 and 4000 cm^{-1} with a piece of film 2 cm in diameter. Spectral output was recorded in absorbance as a function of wave number.

Thermo gravimetric analysis (TGA) was performed to study the degradation characteristic of sawdust composite film fabricated. Thermal stability of the film was determined using TGA Q500 series Thermo gravimetric analyzer (TA Instruments) with a heating rate of 10 °C/minute in a nitrogen environment. It has a weighing capacity 10 to 20 milligram and final temperature of 600°C.

Differential scanning calorimetry (DSC) identifies the melting temperature of the film. The thermal properties of the film with a weight about 10 to 20 mg was performed by a DSC Q1000 series (TA Instrument) under nitrogen atmosphere, with a flow capacity of 25 milliliter/minute from 20 to 300 °C at a heating rate of 10 °C/minute.

III. Results and Discussion

Sawdust composite biodegradable film fabricated is presented in Fig. 4.

Fig. 4 Sawdust composite biodegradable film

Atomic force microscope is a characterization technique, which presents very high possibilities of application in the field of microscopy observation and characterization of various surfaces [10]. 2D and 3D AFM images of the biodegradable film were shown in Fig. 3 and 4 respectively. The nodules are seen as bright high peaks whereas the pores are seen as dark depressions.

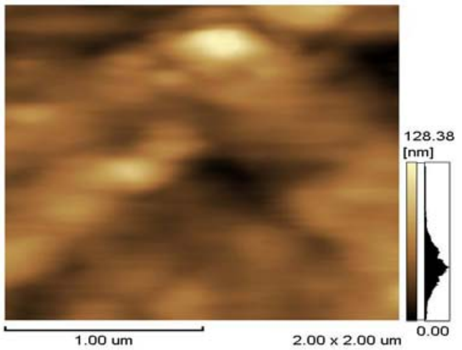

Fig. 5 2D AFM topographic image

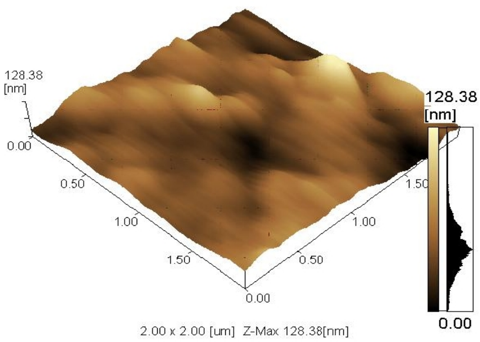

Fig. 6 3D AFM topographic image

From Fig. 5 and 6 were clearly showed surface morphology of biodegradable film fabricated. It is observed that the nodules are not merged and have some peaks. The surface roughness parameter of the biodegradable film fabricated was presented in Table 1. The result showed that the film has rough surface indicating by high roughness value.

Table I The surface roughness of sawdust composite biodegradable film

Roughness Parameter	Value
Ra	12.65
Ry	126.52
Rz	70.93
Rms	16.70

FTIR spectroscopy was used to determine the interactions between the chemical used in the casting solution. Fig. 7 shows the infrared spectra of sawdust composite biodegradable film.

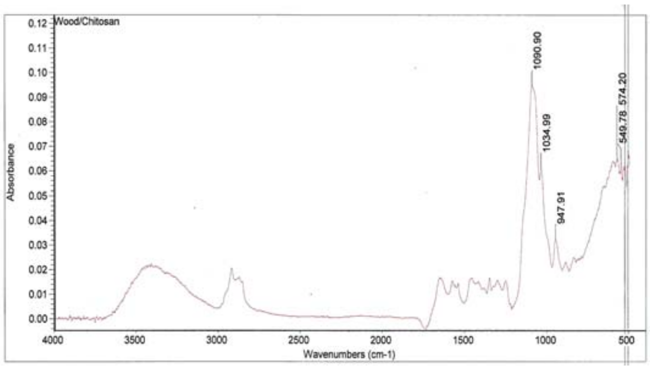

Fig. 7 FTIR spectra of sawdust composite biodegradable film

The broad band area 3500 and 3000 cm^{-1} was the O-H stretching, while the band around 1650 and 1500 cm^{-1} was the N-H bending and C=O stretching presence amide II and amide I respectively. The peaks at 1090.90 cm^{-1} and 1079.76 cm^{-1} suggested the presence of an ether group in the film and 599 to 574 cm^{-1} indicative of aromatic ring. When two components are mixed, the physical blends versus chemical interactions are affected by changes in the characteristic spectra peaks [7].

The thermo gravimetric analysis is used to determine the weight loss of the material as it is heated [11]. The film was run for thermal gravimetric analysis and the result was shown in Fig. 8.

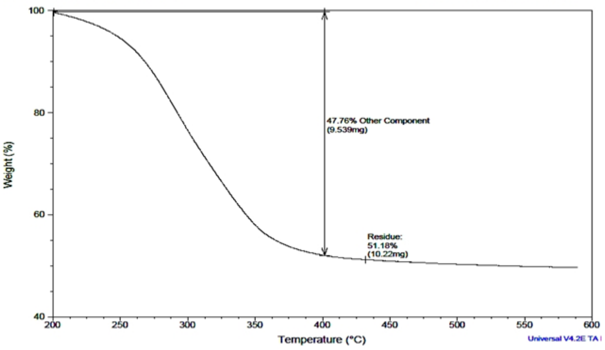

Fig. 8 TGA curve of corn husk composite biodegradable film

Decomposition temperature was obtained from TGA curve. The first weight loss of 1-5 % observed up to 100 °C is due to loss of water from polymer composites. Chitosan shows a discrete weight loss at 286 °C, attributable to the degradation of chitosan chains. Also, there was found that the starch start to degrade at around 275 °C. The addition of starch contributed to the most amount of weight loss. Thus, the amount of residue left behind decrease also with the addition of starch. Starch film well known their ability to bind with water molecules therefore, a tolerable amount of water contains inside a starch. Sawdust composte biodegradable film exhibits thermal stability with residue percent of 47.76 %.

To further understand the structure and interaction between the components, DSC study of the film was performed and the result was shown in Fig. 9.

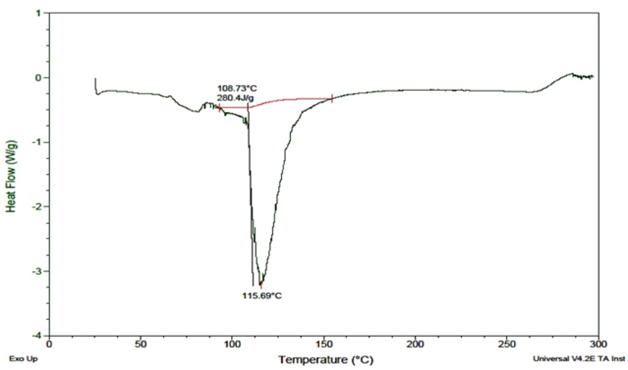

Fig. 9 DSC curve of sawdust composite biodegradable film

Thermogram results showed an initial broad peak at 64.5°C due to removal of absorbed moisture or nonstructural water. The temperature range from 30 °C up to 300 °C was selected to avoid endothermic signals related to the melting of frozen water around 0°C and to limit possible sample degradation.

The observed endotherms are related to the evaporation of water present in the samples that occur over a large temperature interval (100 °C and 150 °C for chitosan and starch respectively). The melting temperature of sawdust composite film is 115.69 °C.

IV. Conclusion

Sawdust composite biodegradable film was prepared by solution mixing. These results from AFM, TGA and DSC analysis exhibit the properties of the sawdust film fabricated. The film fabricated has rough surface, various interaction in the film fabricated, the film has low degradation temperature and melting temperature at 115.69 °C.

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M.Z. Norashikin was born in Perlis, Malaysia on 7th April 1982. She has received bachelor in chemical engineering-bioprocess in 2004 and master in bioprocess engineering in 2006 from Universiti Teknologi Malaysia, Skudai, Johore, Malaysia. She has been work as Lecturer at Universiti Malaysia Pahang (UMP), Kuantan, Pahang, Malaysia for more than 3 years. She engages in supervising undergraduate and postgraduate student research and conducting undergraduate lectures. She also actively involves with administration at faculty and university level where she become committee member for UMP publication unit, Publication and Seminar Coordinators, Editor for Annual Reports, Editor for Buletin and others. She involves in a variety of research activities and become the representative for the university in national and international exhibition through her inventions. She has involve in professional membership where she become a member of International Water Association (IWA), Affiliate of Institution of Chemical Engineer (IchemE) and Graduate Engineer of Board of Engineer Malaysia (BEM).

M.Z. Ibrahim was born in Malacca, Malaysia on 22th July 1982. He received bachelor in electric-mechatronic engineering from Universiti Teknologi Malaysia, Skudai, Johore, Malaysia in 2004 and the master degree for the study of Hybrid Neuro Fuzzy Speaker Recognition System from Universiti Teknologi Malaysia, Skudai, Johore, Malaysia in 2007. From 2004 to 2006, he was with Hewlett-Packard (HP) Malaysia Sdn. Bhd. in Johore Bahru, where he was involved with in-circuit testing and new product introduction design. In 2007, he joined Universiti Malaysia Pahang (UMP) as a Lecturer at Faculty of Electric and Electronics Engineering. His current research interests are Artificial Intelligent System, Image & Signal Processing and Embedded System.