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Seminar Hasil –Hasil Penelitian

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Seminar Hasil – Hasil Penelitian

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Hasil – hasil penelitian yang diseminarkan pada tanggal 29 September 2014 berjumlah 37 makalah yang dikelompokkan menjadi dua yaitu kelompok ilmu-ilmu eksakta dan ilmu-ilmu sosial . Hasil penelelitian ini diharapkan dapat memberikan kontribusi dalam pengembangan ilmu pengetahuan, teknologi, dan seni (IPTEKS) serta mendukung pembangunan nasional.

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Bandar Lampung, Desember 2014
Ketua,

Dr. Eng. Admi Syarif
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DAFTAR ISI

Morphology, Thermal Stability, Functional Group of Wood Flour and Rice Bran as Filler Applied with Poly Lactic Acid Film

Oleh

Edwin Azwar

Abstract

Poly lactic acid (PLA) is a promising biopolymer due to its mechanical and biodegradable properties. Its need to incorporate the filler especially fiber filler from agricultural and forest waste to reduce the cost of PLA. The characterization were done through the interfacial interaction between PLA and fiber filler, the thermal stability, the functional group through hydrogen bondings. SEM image show the fracture surface of the PLA and fibers particles being visible and the rough edges of the voids. Adding fiber filler increased the thermal degradation temperature of the PLA matrix, as a function of the reinforcing content. Hydrolitic degradation occurs, mainly in the outermost polymer layers and not in the inner parts of a matrix.

Keywords : Poly lactic acid, fiber, cellulose, wood flour, rice bran, filler

INTRODUCTION

Poly lactic acid is a thermoplastic that has high strength and modulus and can be manufactured from renewable resources and can be produced from agricultural renewable resources by the combination of fermentation and polymerization. PLA has several advantages, eco-friendly, biocompatibility, processability and has generated great interest as one of the most innovative materials being developed for a wide range of applications. Although poly lactic acid is a relatively stiff polymer characterized by good mechanical strength, it is considered too brittle for many commercial applications. Reinforcing poly lactic acid with fibers offers one possibility to enhance its mechanical and thermal stability[19].

The use of natural fibers, as reinforcements in PLA, gives interesting alternatives for production of low cost and ecologically friendly composites. Biobased composites obtained from PLA, with cellulose fibers possess superior mechanical and thermal properties as a result of reinforcement. Composites of PLA and wood flour were prepared to assess the effects of wood four and rice bran filler on morphological, thermal, and the mechanical properties of the composites.

As the chemical makeup of wood is complex, which consists of cellulose, hemicelluloses, and lignin. Cellulose-based polymer composites are characterized by their low cost, low density, high specific stiffness and strength, biodegradability, and good mechanical properties[1, 3, 4, 11, 14]. However, cellulose fibers are not extensively used in reinforcing thermoplastics, because of their low thermal stability during processing and poor dispersion in the polymer melt[3, 5]. The interfacial adhesion depends on the bonding strength at the interface[7, 15, 22]. Good fiber dispersion have the effect on mechanical and thermal properties, but good dispersion of fibers in a polymeric matrix has been reportedly difficult to achieve[17]. Cellulose functions as the primary structural component within the wood fiber cell walls. Therefore, cellulose is hygroscopic because it is a polar molecule and can easily undergo hydrogen bonding[9]. In thermoplastic wood fiber composites, cellulose is primarily used for reinforcement. The hydroxyl groups on the fiber surface are usually either blocked or modified to be more reactive with thermoplastics. A high proportion of cellulose is believed to be crystalline and is held together by intermolecular hydrogen bonding. Cellulose molecules are completely linear and have a strong tendency to form intra and intermolecular hydrogen bonds. Hydroxyl groups are either located intra-molecular linkages between glucose units in the same molecule or intermolecular linkages between two adjacent molecules. These hydroxyl groups are also responsible for the hygroscopic nature of wood.

Hemicelluloses are predominantly found in the primary and secondary cell walls and represent about 20% of wood[2]. It is a short chain with a degree of polymerization in the low molecular weight polymer and primarily serves as a connecting agent that links or bonds the microfibrils providing additional structural reinforcement to the wood fiber cell wall. Hemicelluloses contain various sugar units, with much shorter chains and by branching of the chain molecules which make it different from cellulose.

Lignin is accounts for 20-30% of wood and widely distributed throughout the cell wall but it is highly deposited in the middle lamella region[5]. Lignin forming a supra molecular structure, known as the wood cell wall because its matrix is associated with cellulose fibrils and hemicelluloses[13]. Lignin, amorphous polymeric material, acts as cement in bonding the cellulose hardwood lignin. Lignin has a low occurrence of hydroxyl groups compared to polysaccharide components(18).

PLA could be degradehydrolytically. Under hydrolytic degradation, PLA is degraded by hydrolysis of the ester bond into monomers. The hydrolytic degradation of these materials in aqueous solution proceeds through random cleavage of the ester bond. This process is controlled by four basic parameters: the rate constant, the amount of absorbed water, the diffusion coefficient of chain fragments within the polymer and the solubility of degradation products.

The overall objectives of the research project are to preparation and investigate interaction between poly lactic acid and fiber filler (rice bran, wood flour) by solution casting technique. On the other hand it need to investigate the effect of fiber filler (rice brand, wood flour) on the structure and property of film with characterization morphology, thermal stability, and mechanical behavior and its hydrodegradation.

EXPERIMENTAL

MATERIALS

Poly lactic acid materials were bought from Hycail Finland, Chloroform (Aldrich). The fiber fillers rice bran and wood flour were obtained from local farmers in Indonesia.

FILMS PREPARATION

Poly lactic acid films were prepared by casting with fiber filler woof flour, rice bran (10-20%. The poly lactic acid and fiber filler are mixed and make into solution by chloroform solvent 75 ml into erlenmeyer. The solution was heated in hot plate HaakeRheocard 600 heated at 45°C, 1 hour. The film forming solution was spread into a petri dish bottom (100x 15 mm) and allowed to air-dry at room temperature overnight.

CHARACTERIZATION

Scanning Electron Microscopy (SEM)

SEM analyses were performed with Hitachi S-4800 is a versatile platform comprising high resolution performance. Film pieces were mounted on bronze stubs using a double-sided tape and then coated with a 2 nm thick carbon layer and 2nm thick gold layer. All samples were examined using an accelerating voltage of 15 kV.

Fourier Transform Infrared Spectrometry (FTIR)

The FTIR spectra of the films were recorded in an IR spectrometer (Perkin Elmer Spectrum 200) in the range 4000-500 cm^{-1}. The spectra obtained were used to determine possible interactions of functional groups between starch with plasticizer and the filler by quantisized molecular resonances that absorb electromagnetic energy selectively from a broadband infrared source. The absorbance is due to resonance caused by vibration-rotation, or rotation of bonds in the studied molecule. A molecule will absorb infrared radiation if it vibrates in such a way that its electric dipole moment changes during vibration.

Thermogravimetric Analysis (TGA)

A thermal weight change analysis instrument, was used in conjunction with a thermal analysis controller. The TG analyzer was employed to measure the amount and rate of change in weight of the material, either as a function of increasing temperature or time, in a controlled atmosphere. Thermogravimetry analyses were carried out by a Mettler TGA851 instrument. The initial weight of each sample was 10 mg aluminium pan. The samples were kept in a *alumina crucible and heated in the furnace, flushed with* N2 gas at the rate of 50 ml/min, from 30 to 450C, at the rate of 10C/min. The percentage weight loss and derivative weight loss were plotted against temperature for all samples.

RESULTS AND DISCUSSION

SCANNING ELECTRON MICROSCOPY (SEM)

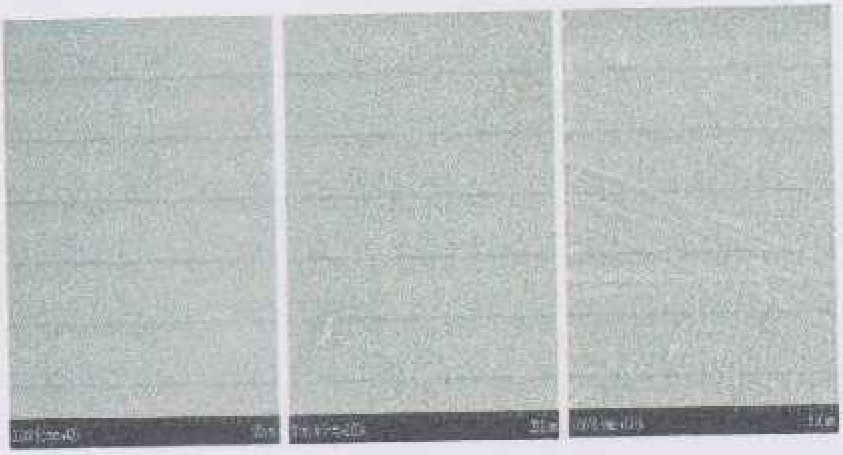

Figure 1.SEM pure poly lactic acid.

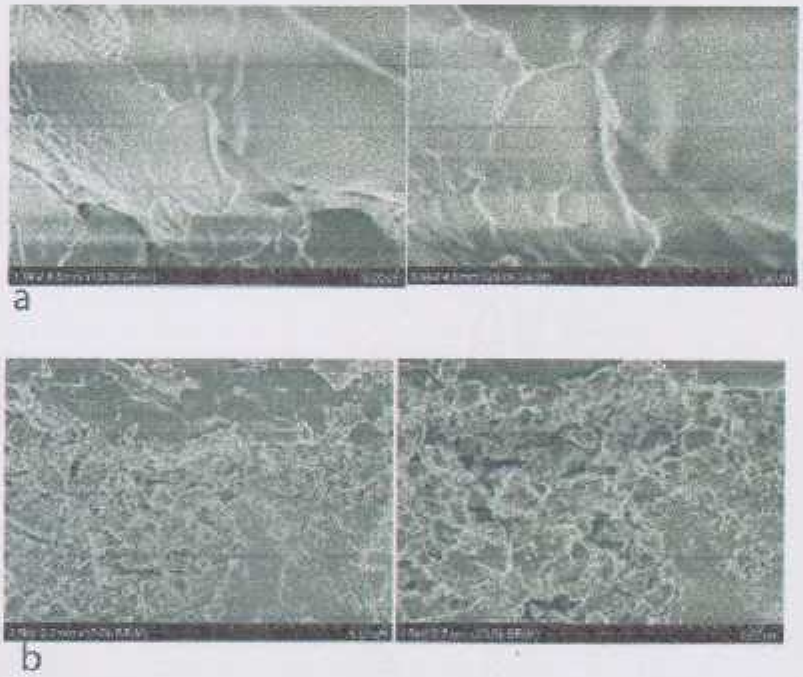

Figure 2. a. SEM poly lactic acid and rice bran,

b. SEM poly lactic acid with wood flour

Agglomerates of wood-flour and rice bran particles in the composites are expected to be susceptible to failure under stress, thereby representing zones of potential stress concentration. Figure 2 shows at the SEM image of the fracture surface of the poly lactic acid and wood flour, rice brand containing 1,5g (30%) indicates a inhomogenity at the fracture surface, with voids, the wood-flour and rice bran particles being visible and the rough edges of the voids being the result of particle pull-out, which shows that gaps exist at the matrix particle interface with no evidence of particle deformation. Some fibers are tightly connected with the matrix. It is probable that the fiber surface has been covered with a thin layer of the matrix, which led to better stress transfer between the matrix and the reinforcing fibers. Due to a high intramolecular bonding among the wood fibers, the dispersion of the wood fibers in the polymer matrix is small and the reinforcing ability of the fiber is reduced remarkably turn to the tendency of untreated wood to form large aggregates. The union of aggregates, although weakly associated through nonbonded physical interactions, leads to an agglomerate. Agglomerates of wood-flour and rice bran particles in the composites are expected to be susceptible to failure under stress, thereby representing zones of potential stress concentration. The mixing and dispersion of fillers in a material involves primarily the incorporation and distribution of filler breakdown into agglomerates, and then into aggregate structures. The better the fibers dispersed in advance in the polymer matrix the better its uniformity.

Fgure1. showed pure PLA film were transparent and smooth. While examination from the aged samples (figure 6), the whitening of the surface of PLA degraded, this could be result from accelerated spherulite formation when chain reorganized.

FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)

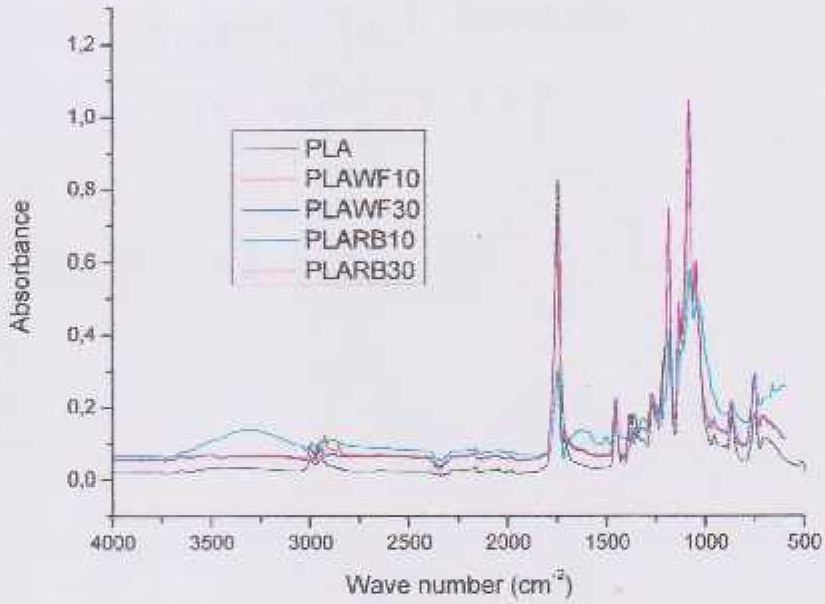

Figure 3.FTIR poly lactic acid with fiber fillers.

Figure 3 show pure poly lactic acid and poly lactic acid with wood flour and rice bran filler, its chemical functional groups were detected several characteristic peaks representing in the FTIR spectra. The strong peak in the spectrum of poly lactic acid at 1,749 and 1746 cm^{-1} is due to C=O stretching of the carbonyl group, while the bending vibration of this group appears at 1267; 1266; and 1265 cm^{-1}.

The peaks at 866; 865 and 752; 750 cm^{-1} represent the amorphous and crystalline phases of poly lactic acid, respectively the intensity of the amorphous and crystalline peaks is nearly the same, which indicates that the poly lactic acid is a semicrystalline material with nearly equal presence of both phases.

The spectral structure at 3286; 3328 cm^{-1} regions were assigned to OH groups in wood flour and rice bran 30%, An interaction between the carbonyl groups of poly lactic acid and hydroxyl groups of wood through hydrogen bonding [14, 15, 17]. While 2882 cm^{-1} CH stretching in wood combination of cellulose, hemicellulose and lignin.

The decreased intensity of the crystalline peak of poly lactic acid at 752; 750 cm^{-1} with respect to the amorphous peak at 866; 865 cm-1 suggests a reduction in the crystallinity of poly lactic acid with the addition of wood flour[6, 8].

THERMALGRAVIMETRIC ANALYSIS (TGA)

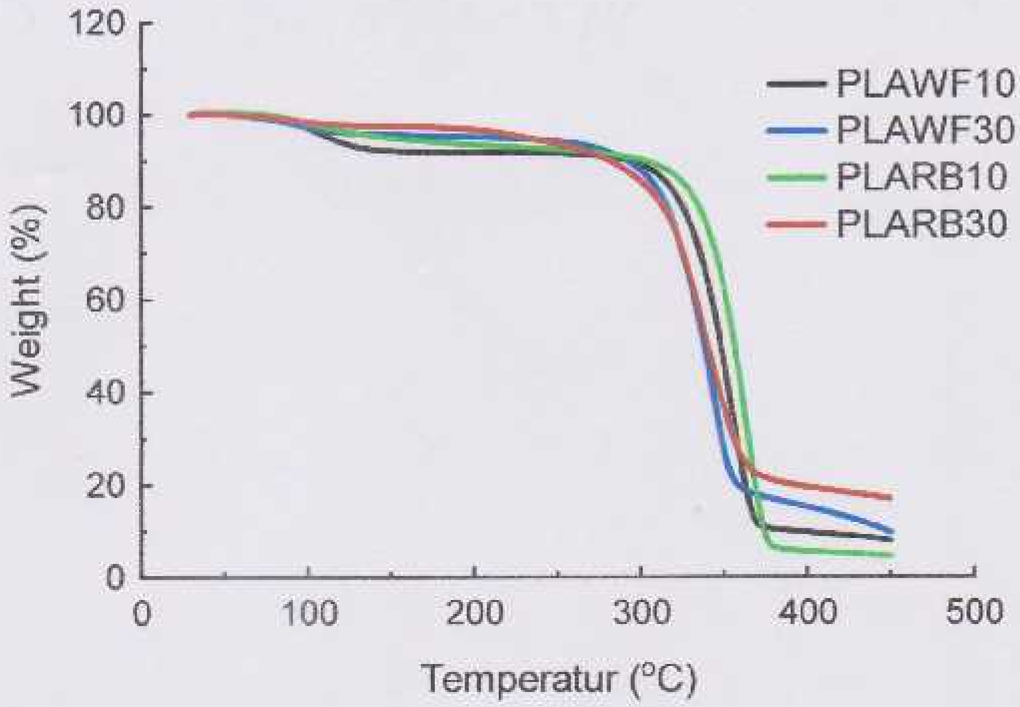

Figure .4TGA Poly lactic acid composite with fiber fillers.

The wood and rice bran fiber fillers are thermally stable up to 338,33°C. Adding both fiber filler in poly lactic acid has increased the thermal degradation temperature of the matrix, as a function of the reinforcing content. It can be seen ash content of rice brand filled in poly lactic acid composites at 1,5g (30%) is the highest 16,99% this due to rice bran have much higher inorganic materials than cellulosic materials. Ash in the rice bran is mainly composed of silica.

The weight loss in cellulose begins at a higher temperature and take place a two-step reaction, with the change in the mechanism occurring at about the transition point. At degradation temperature below the transition point about 300^{0}C the predominant pathway results in reduction in the degree of polymerization, elimination water, and carbonyl, carboxyl, hydroperoxide groups.

CONCLUSIONS

Poly lactic acid and filler showed good mechanical interlocking between poly lactic acid with wood flour and rice bran filler attributed to the surface roughness. Adding wood flour and rice bran as filler in poly lactic acid has increased the thermal degradation temperature of the matrix, as a function of the reinforcing content. The possible interaction between the poly lactic acid with wood flour and rice bran filler showed the amorphous and crystalline phases of poly lactic acid, respectively the intensity of the amorphous and crystalline peaks is nearly the same, *which indicates that the poly lactic acid is a semicrystalline material* with nearly equal presence of both phases.

FUTURE WORK

Further steps in the development poly lactic acid with fiber filler will focus on in the liquefaction of wood four and rice bran. And also continue process the hydroxyl group containing the species in the wood flour and rice bran components that can be used as synthesis of monomeric ester plasticizers.

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