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INTERACTION STARCH COMPOSITE FILM AND ITS THERMAL STABILITY AND FUNCTIONAL GROUP

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Abstract

Starch as biopolymer from nature consist of amylose and amylopectin. Most starch based composites exhibit poor material properties such as tensile strength, stiffness and elongation at break. The addition of plasticizers to starch films increase the mobility of polymer chains, decrease its inherent brittleness by reducing intermolecular forces and its glass transition temperature so improve their flexibility. Beside plasticized the tapioca starch need to be filled by fiber and mineral as reinforcement improve the properties. The thermal stability of tapioca starch could be carried out by TGA analysis to measure the amount and rate of change in weight of the material. Through hydrogen bondings, the possible interaction between the tapioca starch, plasticizer and filler could be seen by FTIR analysis by quantized molecular resonances that absorb electromagnetic energy broadband infrared source obtained from spectra.

Keywords :

Polysaccharides, amylose, amilopectin, glycerol, sorbitol, citric acid, cellulose, kaolin, halloysite, filler.

1. INTRODUCTION

The use of starch such as tapioca starch in recent years has been growing interest for an alternative biodegradable polymer, as renewable plastic source, to reduce the environmental pollution caused by plastic wastes with low cost. Biopolymers like tapioca starch create the basic network structure of the film. However, films prepared from tapioca starch are often too fragile. Thus, they have to be plasticized using low molecular weight substances such as polyols, which decrease interactions between the starch biopolymer chains. The tapioca starch can be formed into plastic film and formation of a rigid structure of open cell, by process network building, swelling and gelatinization.

1.1. STARCH PROPERTY

The loss of crystallinity and irreversible granule swelling happen in gelatinization of starch, when starch is heated in aqueous condition. Mixing is needed to maximize amylase and amylopectin leaching into water, water adsorption is interrupted into granule because it surrounded by concentrated regions of leached amylase^[1].

1.1.1. Amylose

Amylose has a right hand helix linear structure and inside of the helix is lipophilic and consist of only hydrogen atom. It predominantly a linear chain of α (1-4) linked D-glucosyl Units^[2]. There are hydrophilic hydroxyl groups on the outside of amylose chains. Leaching of amylose occurs when starch is heated with water, and it is an order phase transition within the swollen granule^[3]. The total disruption of granule occurs if continued heating.

1.1.2. Amylopectin

Amylopectin is a polymer with a massive weight, with degrees of polymerization of 15-20 glucose units and results in entanglements between amylopectin molecules with very long life times^[2]. It is a highly branched polysaccharide consisting of α (1, 4) linked glucose with α (1, 6) linkages.

1.2. PLASTICIZERS

To increase the mobility of starch polymer chains, plasticizer must be added. By reducing intermolecular forces and glass transition temperature, the plasticizer decreases the inherent brittleness of starch.

At low plasticizer content have not the classical effect of elongation starch film^[4, 5]. With higher plasticizer content, the elongation starts to increase, for glycerol approximately 15% and 27% for sorbitol. As the plasticizer content increase, it can exhibit phase separation and this composition more sensitive to ambient humidity^[6]. If the plasticizer content below 10% the starch interaction is weak and fragile, while 20% there is a progressive increase in the flexibility and in the elongation properties of the films^[7].

It has been proposed that the end hydroxyl groups of the backbone of polyols are the most accessible to bind water and also interact with starch molecules^[8]. Because water sorption affinity of polyols increases it increase in water sorption at water activities around 0.6^[9]. With polyols plasticized the starch films have been shown to adsorb less water than films without plasticizer up to water activities around 0.6^[10, 11]. Starch creates steric hindrance against water adsorption coincidentally with low affinity of polyols to bind water at low water activities and interaction between polyols^[10].

Compared with glycerol, citric acid which act as plasticizer, its carboxyl groups can form stronger hydrogen bonds with the hydroxyl groups on starch molecules so as to promote the ageing resistance ability^[11]. By introducing covalent bonds that supplement intermolecular bonds cross linking of a blend reinforces the intermolecular binding so as to improve the mechanical properties^[12].

1.3. FILLERS

1.3.1 Fiber Filler

The chemical similarities of starch and wood fiber provide a good compatibility. When natural fibers are mixed with thermoplastic starch their mechanical properties are improved^[13, 14]. Properties of a fiber reinforced polymer composite depend on many factors like fiber matrix adhesion, volume fraction of fiber, fiber aspect ratio and orientation of fiber^[15].

The chemical made up of wood is complex. As the fibers characteristics depend on the properties of the individual constituents, the fibril structure and the lamellae matrix, which consists of cellulose, hemicelluloses, and lignin. Cellulose functions as the primary structural component within the wood fiber cell walls. The major wood component, cellulose, can be characterized as a linear and high molecular mass polymer built up of β (1-4)-D-glucopyranosyl units^[16, 17]. It is a long chained, unbranched, condensation polymer of β -D-glucose units, with a degree of polymerization as high as 30,000. Because of its high degree of polymerization and crystallinity, cellulose is responsible for strength in wood^[18]. A high proportion of cellulose is believed to be crystalline and is held together by intermolecular hydrogen bonding^[19, 20]. Cellulose molecules are completely linear and have a strong tendency to form intra and intermolecular hydrogen bonds. Cellulose is hygroscopic because it is a polar molecule and can easily undergo hydrogen bonding^[19]. However, the hydroxyl groups in the crystallites are linked with each other. Hydroxyl groups present in the amorphous region of cellulose are available for chemical reactions^[21].

Hemicelluloses are predominantly found in the primary and secondary cell walls and represent about 20% of wood^[16]. Hemicelluloses contain various sugar units, with much shorter chains and by branching of the chain molecules which make it different from cellulose^[21]. Hemicelluloses are alkaline extractable hetero-polysaccharides that are branched and of lower molecular weight than cellulose^[20].

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^[22]. Lignin forms a supra molecular structure, known as the wood cell wall because its matrix is associated with cellulose fibrils and hemicelluloses^[20]. Lignin has a low occurrence of hydroxyl groups compared to polysaccharide components^[23].

1.3.2. Mineral Filler

The introduction of inorganic fillers to a polymer matrix increases its strength and stiffness and sometimes creates special properties, originating from the synergistic effect between the component materials.

Halloysite is naturally dioctahedral 1 : 1 clay mineral ($\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4 \cdot n\text{H}_2\text{O}$), which has the same chemical composition as kaolinite. Kaolin has been identified as a mineral that shares an analogous microstructure to that of talc.

2. EXPERIMENTAL

2.1. MATERIALS

Tapioca starch was obtained from Ibu Tani, cap anak no.1, Bogor, Indonesia. Glycerol, citric acid and sorbitol was purchased from Aldrich. Rice bran and wood flour from the local farm. The mineral halloysite, kaolin are purchased from Aldrich.

2.2. FILMS PREPARATION

Tapioca starch films were prepared by casting with different plasticizers and different composition: glycerol, sorbitol and citric acid (10-30% of tapioca based starch composition), and blend with fiber filler: wood flour, rice bran (10-20% of tapioca based starch composition) and mineral filler halloysite, kaolin and silica (10-20% of tapioca based starch composition). The 100g tapioca starch, plasticizer and filler are mixed and made into solution by adding deionized water 125ml into erlenmeyer. The solution was heated in hot plate Haake Rheocard 600. Stirring and heating were ended when tapioca starch gelatinized at temperature of 90°C, 1 hour at 500 rpm. The film forming solution was spread into a petri dish bottom (100x 15 mm) and allowed to air-dry at room temperature overnight.

2.3. CHARACTERIZATION

2.3.1. Thermogravimetric Analysis (TGA)

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 450°C, at the rate of 10C/min. The percentage weight loss and derivative weight loss were plotted against temperature for all samples.

2.3.2. Fourier Transform Infrared Spectrometry (FTIR)

The FTIR spectra of the films were recorded in an IR spectrometer (Perkin Elmer Spectrum 200) in the range 4000-650 cm⁻¹. The wavelength of the absorbed light will absorb around 1700 cm⁻¹.

3. RESULTS AND DISCUSSION

3.1. THERMALGRAVIMETRIC ANALYSIS (TGA)

3.1.1. Tapioca Starch blend with Plasticizer and Mineral and Fiber Filler

Table 1. Tapioca starch with plasticizer, mineral and fiber filler

Composition	Onset temperature (°C)	Remaining weight (%)
1. Tapioca starch5, Glycerol3, halloysite1, wood flour1	286,94	27,61
2. Tapioca starch5, glycerol2, citric acid1, kaolin1, rice bran1	288,05	37,71
3. Tapioca starch5, glycerol2, citric acid1, kaolin1, wood flour1	293,73	32,30

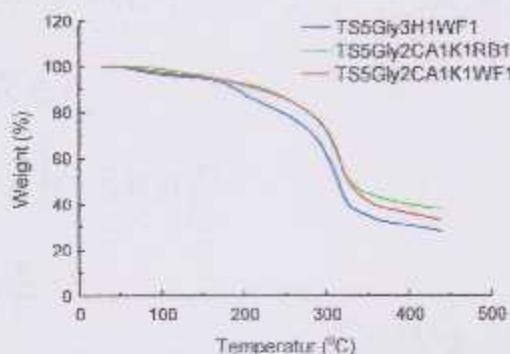


Figure 1. TGA Tapioca starch with plasticizer, mineral and fiber filler

The wood flour and rice bran fillers are thermally stable up to 200°C. Adding both fiber filler has increased the thermal degradation temperature of the tapioca starch matrix, as a function of the reinforcing content. The weight loss of the tapioca starch composites with plasticizer, mineral and fiber filler loading are shown in figures 5. Wood fiber is mainly composed of cellulose, hemicellulose and lignin. Lignin is the most thermal stable than the rest materials. Because of the higher cellulose content of wood flour, the thermal stability of woof flour is a slightly higher than that of rice bran^[20]. It can be seen thermal stability of wood flour filler (tapioca starch5, glycerol2, citric acid1, kaolin1, wood flour1 at 293,73°C), while the rice bran filled (tapioca starch5, glycerol2, citric acid1, kaolin1, rice bran1 at 288,05°C). As the content of lignin and cellulose increases, the thermal stability of composites can be shown as more stable at high temperature. For the tapioca starch composites, the moisture content mainly contributed to mass loss at the onset temperature^[19] this was due to the good adhesion between tapioca starch and wood fiber. The fiber component decreased the mass loss of plasticizer, therefore, the thermal stability of tapioca starch materials was improved.

Thermal degradation curves of wood flour and rice brand could be shown in figure 5. 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°C the predominant pathway results in reduction in the degree of polymerization, elimination water, and carbonyl, carboxyl, hydroperoxide groups. Above the transition point, the major thermal degradation of cellulose involves the increased aromatic character in the tar. The greater thermal stability of lignin has generally been attributed to the high degree of condensation. The residual weight of lignin is mainly composed of phenolic materials.

The final temperature of the decomposition was 449,83°C. The first step of weight loss could be attributed to the loss of loosely bound water, accompanied by the formation of volatile disintegrated materials. The second step was mainly caused by the heat decomposition of the molecules, and the products were composed of small molecular carbon and hydrocarbon. The TG curve of rice bran exhibited two mass loss steps. The initial mass loss below 100°C may be due to the evaporation of absorbed moisture. The second mass loss occurring between 175°C and 450°C may be due to the degradation of different constituents in the rice bran such as sugar and fiber present along with tapioca starch.

3.2. FOURIER TRANSFORM INFRARED SPECTROMETRY (FTIR)

3.2.1. Tapioca Starch with Plasticizer, Mineral and Fiber Filler

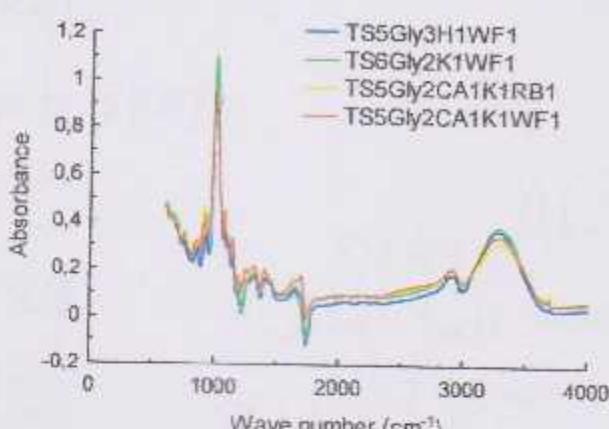


Figure 2. FTIR Tapioca starch with plasticizer and mineral and fiber filler

Figure 6 the spectral structure between 1077 and 1078 cm^{-1} has been assigned to C-O groups in wood cellulose. The spectral structure at 1015 and 1050 cm^{-1} and 3080-3500 cm^{-1} regions were assigned to C-O and OII groups, respectively in wood (combination of cellulose, hemicellulose and lignin). The band at 1508-1512 cm^{-1} was assigned to lignin. More importantly, the bands between 1680 and 1800 cm^{-1} were assigned to the carbonyl functional groups.

4. CONCLUSIONS

Much of the tapioca starch which including amylose and amylopectin that hydrophilic hydroxyl groups branches are mainly attribute to the crystallinity of granule, the plasticizer decreases its inherent brittleness by reducing intermolecular forces and glass transition temperature, changes crystalline to an amorphous structure.

The thermal decomposition on certain temperature of tapioca starch indicate the thermo stability of the matrix sample.

Strong hydrogen bonds enhanced the bonding so that the thermal stability of the plasticized tapioca starch film increased.

Adding fiber and mineral filler has increased the thermal degradation temperature of the tapioca starch matrix, so this can be used as a function of the reinforcing filler.

The broad band shows the structure of tapioca starch around 3267-3291 cm^{-1} .

The tapioca starch with plasticizer, mineral and fiber filler showed stretches associated with free inter and intramolecular bound hydroxyl group due to the hydrogen bonded hydroxyl groups that contribute to the complex vibration.

5. FUTURE WORK

Further steps in the development will focus on starch blend with kaolin and halloysite filler with the liquefaction of wood flour and rice bran as polyols plasticizer and filter.

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