

Synthesis of Cellulose Stearate Ester as Wet Strength Agent for Synthesis of Bio-polybag from Oil Palm Empty Fruit Bunch

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Abstract

Biodegradable polybags are an alternative to overcome the weakness of synthetic polybags because of their degradation properties. Oil palm empty fruit bunches contain a lot of cellulose so that they can be used as a biodegradable polybag. Wet Strength serves to increase the physical strength of bio-polybags when exposed to water (in wet conditions) so that water content stability is required. In this study, Cellulose Stearate Esters were synthesized in an effort to increase the stability of the water content in bio-polybags. Cellulose Stearate Esters are synthesized through a transesterification reaction between -Cellulose isolated from Oil Palm Empty Fruit Bunches (EFB) with methyl stearate. The synthesis of cellulose stearate esters was carried out by refluxing for 2 hours using methanol solvent with various catalysts Na₂CO₃ 5, 10, 15, 20 mg and with volume variations of methyl stearate 5, 10, 15. And the best variation was determined based on the degree of substitution test, namely with variations Na₂CO₃ catalyst 20 mg and volume of methyl Stearate 15 ml, amounting to 1.95. The result of the synthesis, namely cellulose stearate is supported by the FT-IR spectrum in the wavenumber region of 3468.01 cm⁻¹ indicating an OH group, 3062.96 cm⁻¹ indicating a CH stretching group, 1695.43 cm⁻¹ indicating a C=O group, cm⁻¹ indicating a CH bending group, 1095.57cm⁻¹ indicates a COC group, 609.51cm⁻¹ indicates a (CH₂)n>4 group. The results of surface morphology analysis using SEM showed that the surface of cellulose stearate looked homogeneous, more regular and had denser cavities than -Cellulose.

Keywords: Cellulose Stearate Ester, Methyl Stearate, Empty Fruit Bunches, Bio-polybags.

1. Introduction

Indonesia is the largest palm oil producing country in the world with production above 40.56 million tons in 2018. Based on data from the Central Statistics Agency (BPS) from the December 2019 publication, the area of oil palm plantations in Indonesia reached 14.32 million hectares. This palm oil processing industry produces a very large amount of solid waste such as Palm Oil Bunches (EFB). The average production of oil palm empty fruit bunches ranges from 22% to 24% of the total weight of fresh fruit bunches processed at the Palm Oil Mill [1].

The composition of cellulose which is quite large in Oil Palm Empty Fruit Bunches (EFB) makes this EFB potential to be used as raw material for the manufacture of cellulose esters. Various methods have been introduced to make these cellulose esters. Cellulose is reacted with Fatty acid, acyl chloride, Fatty acid vinyl and fatty acid anhydride to produce a degree of substitution (DS), (DS number: 0.3 - 1.3) depending on the fatty acid branch chain used [2]. OPEFB consists of 20.6–33.5% hemicellulose [3], 23.7–65.0% cellulose [4] and 14.1–30.5% lignin [5].

Due to its high cellulose and hemicellulose content, OPEFB is emerging as a potential source in the pulp and paper industry. The chemical properties of OPEFB fibers are similar to hardwoods but with a shorter fiber length (0.53 mm) and the cellulose content is almost comparable to the cellulose content of wood fibers (38–49%) [6], bamboo (26–43%) [7] and bagasse (32–44%) [8].

Hemicellulose, cellulose and lignin in lignocellulosic biomass are arranged in a complex hierarchical structure that forms the recalcitrant properties of microbial and chemical pretreatment [9]. Effective dissolution of these components is very important to produce value-added products from lignocellulosic biomass. High strength paper is usually made from fiber sources with high cellulose content and low lignin content. The removal of lignin from the pulp increases the brightness of the paper and also makes it more durable and stable. However, conventional pretreatment techniques such as mechanical, chemical, biological and physicochemical or a combination of these methods often require expensive and extreme methods to break down biomass into cellulose, hemicellulose and lignin products [10].

In connection with the Wet Strength Agent, the Wet Strength Agent functions to increase the physical strength of the paper when exposed to water (in wet conditions). Strength in wet conditions is needed on some types of paper such as tissue paper. This increase in strength



occurs by manipulating the hydrogen bonds between the fibers. it is necessary to have water content stability, so we need a cellulose ester, especially with a large DS number and with long-chain fatty acids.

There are several modifications of cellulose, such as etherification [11], acetylation [4], esterification [12] and transesterification. Transesterification is a reaction for the formation of an ester from another ester compound through the exchange of an alkyl group from an ester reacting with alcohol. The transesterification reaction can be catalyzed by acids or bases.

Based on the description above, researchers are interested in further researching cellulose from Oil Palm Empty Fruit Bunches (EFB) which has the potential to be modified through a transesterification reaction with methyl ester stearate, so that cellulose has better properties.

2. Literature Review

Cellulose is the most abundant polysaccharide in nature. This compound has a high molecular weight, regular structure in the form of a linear polymer with -D-glucopyranose repeat units, because of its structural regularity, cellulose is a relatively inexpensive polymer with excellent physical and chemical properties [13]. Long-chain cellulose has stronger physical properties, is more durable against degradation caused by heat, chemical and biological influences [14].

Cellulose is the main component of plant cell walls, where the cellulose content is about 45 - 50% of the dry weight of the plant. Cellulose is composed of anhydroglucopyranose units linked by 1,4 glycosidic bonds to form an unbranched macromolecular chain. Each anhydroglucopyranose unit has three hydroxyl groups [15]. Cellulose has the empirical formula ($C_6H_{10}O_5$)n with n up to 1500 and a molecular weight of up to 243,000 [16].

Cellulose contains about 50-90% crystalline and the rest is amorphous. Cellulose is almost never found in its pure state in nature but is always bound to other materials such as lignin and hemicellulose. Cellulose molecules are glucose microfibrils that are bonded to each other to form very long polymer chains. The presence of lignin and hemicellulose around cellulose is the main obstacle to hydrolyzing cellulose [17].

Currently, cellulose has become a material that is widely used in various applications. This relates directly to the paper industry where cellulose is treated conventionally, such as paper and cardboard. However, although paper and cardboard are the main products of cellulose, this does not mean limiting the use of cellulose for other uses. Cellulose can be chemically modified to create a wider range of derivatives that are used in the industrial sector. These derivatives are further used as coating agents, laminating, optical films, as well as adsorbents. In addition, cellulose derivatives can also be found as additives in building materials, pharmaceuticals, food and cosmetic products [18].

Cellulose ethers and esters are commercially produced under heterogeneous reaction conditions using acid and anhydrous acid as reagents. In other words, the main pathway of ether synthesis is Williamson ether synthesis. The weakness of heterogeneous reactions is the relatively slow reaction rate and the absence of regioselective properties of a reaction so that the accessibility of the free hydroxyl groups of cellulose is a determining factor for selectivity and also for the degree of substitution. More specifically, when the reaction takes place in a homogeneous solution, the regioselectivity of the reaction is determined by the difference in the reactivity of the free hydroxyl groups belonging to the cellulose molecule, not from its accessibility. In addition to the ability to control the selectivity in the reaction, the value of the degree of substitution can be adjusted from the derivative of a compound with a low degree of substitution to that of a compound with a higher degree of substitution. Therefore, the material produced from cellulose derivatives can be adjusted according to the desired path [19].

Cellulose esters are widely used like fibers and plastics, while cellulose ethers are used as binders and additives for special mortars or special chemicals for building and construction, viscosity stabilizers in paints, food, pharmaceutical products and others. Cellulose is also the basic material for making paper. The fiber has high durability [20]. Cellulose derivatives have been widely used in pharmaceuticals such as ethylcellulose, methylcellulose, carboxymethyl cellulose, and other forms. The use of cellulose forms in preparations is due to its inert nature and excellent biocompatibility in humans [21], [22].

The transesterification reaction is widely known commercially in industrial organic reactions. In this reaction, an ester is converted to another ester by exchanging an acid group or an alcoholic group. When transesterification exchanges alcoholic groups, it is called an alcoholysis reaction. In alcoholysis, alcohol is added in excess to obtain a high yield of the desired ester. Recently, the production of alkyl esters, especially methyl esters, from plant oils (such as palm oil, jatropha oil) has become very popular in the process of making biodiesel from renewable feedstocks.

The transesterification reaction is an equilibrium reaction that is usually carried out simply by mixing the reactants. This reaction proceeds so slowly that a catalyst is needed to speed up the reaction so that it can be used commercially. The use of a catalyst only accelerates the occurrence of equilibrium but cannot shift the position of the equilibrium. Strong acids and strong bases are widely used as catalysts. To increase the conversion of the reaction, it is necessary to pay attention to several aspects such as changes in the concentration of reactants and reaction products, volume, pressure and temperature [17].

Transesterification is divided into three types of reactions [23], [24]:

- 1. Interesterification, namely the formation of esters from esters with esters. Interesterification reactions are a way to change the structure and composition of oils and fats by exchanging acyl radical groups between triglycerides and alcoholic acids (alcoholysis), fats (acidolysis), or esters (transesterification).
- 2. Alcoholysis is the reaction of an ester with an alcohol to form a new ester, where the reaction is usually slow but can be accelerated with the help of a catalyst commonly used is an inorganic acid such as HCl and H_2SO_4 .
- 3. Acidolysis is the reaction of the formation of an ester between esters with other esters. Here, the alkyl group of the ester is exchanged with the hydrogen atom of the acid used. The catalyst used will cause proton abstraction which is then followed by the alkyl binding of the ester by the enolate ion formed.

3. Methods

The materials and equipment needed in this research are Empty Palm Oil Bunches (EFB) PT. Ika Bina Agro Wisesa, Aquadest, HNO₃ 3.5%, NaOH 2%, Na₂CO₃, Na₂SO₄, NaOH17.5%, Na₂SO₃ 2%, NaOCl 1.75%, H₂O₂, Stearic Acid, n-hexane, Phenolphthalein Indicator, KOH 0.5 M, 0.5 M HCl, 75% Ethanol and Methanol.

This research consisted of eight stages, namely Isolation of Cellulose from Oil Palm Empty Fruit Bunches, Production of Methyl Stearate, Synthesis of Cellulose Stearate Esters, Determination of the Degree of Substitution (Wurzburg 1978 in Singh 2004), Sample Preparation, Isolation of -cellulose from Palm Oil Empty Fruit Bunches, Production of Methyl Stearate and Production of Cellulose Stearate Esters. Experiments were carried out on the mass of the catalyst used and the volume of methyl stearate. The -Cellulose isolation used in this study was the result of isolation from empty palm oil bunches from the PKS mill PT. Ika Bina Agro Wisesa (IBAS). -cellulose obtained from 75 g isolated from Oil Palm Empty Bunches was 16.51 grams (22.01% of initial weight).

The manufacture of Methyl Stearate is carried out by an esterification reaction between the Stearic Acid used from PT. SOCIMAS with methanol uses an acid catalyst, namely $H_2SO_4(p)$. Methyl Stearate esterification process by refluxing at a temperature of 70-80°C for 2 hours. A clear colored methyl stearate was obtained. Cellulose Stearate is obtained by reacting -cellulose from Oil Palm Empty Fruit Bunches with methyl Stearate using Na₂CO₃ catalyst and methanol as solvent. This study also used 1 g of cellulose and varied the volume of methyl stearate and catalyst. Where the volume variations of methyl stearate are 5,10, and 15, while the catalyst variations are 5, 10, 15, 20 grams, respectively. The results of the synthesis obtained were subjected to a degree of substitution test to determine the best temperature and catalyst, FT-IR -Cellulose Spectrophotometer Analysis and Surface Morphological Analysis with SEM (Scanning Electron Microscopic).

4. Result and Discussion

3.1. Isolation of alpha-cellulose from Oil Palm Empty Fruit Bunches

Cellulose used in this study is the result of isolation from empty palm oil bunches from the PKS mill PT. Ika Bina Agro Wisesa (IBAS). Cellulose was analyzed from the isolation of 75 grams of empty fruit bunches fiber through several stages according to previous research methods. -cellulose obtained from 75 g isolated from Oil Palm Empty Bunches was 16.51 grams (22.01% of initial weight).

In this study, the delignification process was carried out using a 2% NaOH solution at a temperature of 50°C so that the lignin could be dissolved. The dissolution of lignin at this temperature is caused by the degradation of the lignin molecules by strong base nucleophiles (OH-).

In the delignification process, yellow cellulose is produced, so it needs to be bleached. The bleaching process aims to remove the remaining lignin that is still attached. In this process, NaOCl is used as a bleaching reagent. In the bleaching process, white cellulose will be produced but the cellulose still consists of, and -Cellulose. Therefore, it is necessary to separate -Cellulose from and -Cellulose using 17.5% NaOH, where -Cellulose will precipitate while and -Cellulose will dissolve [25]. The results were obtained in the form of -cellulose which is still yellow so it is necessary to bleach it with 10% H₂O₂ to remove the pigment attached to the cellulose. Then it is dried to reduce the water content, so that white -Cellulose is obtained [26]. The results of -Cellulose isolation were then analyzed using the functional group test (FT-IR) and the morphology test (SEM).

3.2. Results of FTIR Spectrophotometer Analysis of -Cellulose EFB

From the FT-IR spectroscopic data of -cellulose from Palm Empty Fruit Bunches (EFB) it gives a spectrum with vibrational peaks in the wave number region of 3431.36 cm-1 which indicates -OH group, 2893.22 cm-1 which indicates C-H stretching group; 1359.82 cm-1 which shows the C-H group.



Fig 1. FT-IR Spectrum - Cellulose from Oil Palm Empty Fruit Bunches

3.3. Results of SEM Surface Morphological Analysis of -cellulose EFB



Fig 2. SEM results of -cellulose EFB (a) 1000x magnification (b) 2000x magnification

SEM (Scanning Electron Microscopic) surface morphology analysis was carried out to see the surface morphology of the isolated - Cellulose from Oil Palm Empty Fruit Bunches (EFB). This analysis will show a picture of how well the interaction of the reagents is used in the modification of cellulose. In this study, the surface morphology analysis of SEM can be seen in Figure 2. with a magnification of 1000 times and 2000 times. The SEM results of -cellulose isolated from Oil Palm Empty Fruit Bunches (EFB) showed a homogeneous and smooth surface morphology.

3.4. Results of FTIR Spectrophotometer Analysis of Cellulose Stearate Esters



Fig 3. Cellulose Stearate Esters FTIR Spectrum

From the FTIR spectroscopic data, the Cellulose Stearate Ester gives a spectrum with vibrational peaks in the wave number region of 3468.01 cm^{-1} which indicates the -OH group, 3062.96 cm^{-1} which indicates the CH stretching group, 1695.43 cm^{-1} . which indicates the C=O group, 1465.90 cm^{-1} indicates the CH bending group, 1095.57 cm^{-1} indicates the COC group, wave number 609.51 cm^{-1} indicates the (CH₂)n>4 group. Figure 4 shows the comparison of the FTIR spectrum of -Cellulose and Cellulose Stearate Ester.



Fig 4. Comparison of FTIR Spectrum of Cellulose and Cellulose Stearate



3.5. Results of SEM Surface Morphological Analysis of Cellulose Stearate Esters

Fig 5. SEM results of cellulose stearate ester (a) 1000x magnification (b) 2000x magnification

SEM (Scanning Electron Microscopic) surface morphology analysis was carried out to see the surface morphology of the Cellulose Stearate Ester. In this study, SEM surface morphology analysis was carried out on sample 12 which had a DS of 1.95 and can be seen in Figure 4.5. From the figure, it can be seen that there has been a change in surface morphology which indicates that there has been a reaction between the OH group on the C2 atom of cellulose and the carbonyl group of methyl Stearate. The surface at 1000 and 2000 times magnification looks homogeneous, more regular and has larger pores than -cellulose before esterification.

3.6. Analysis of Cellulose Stearate Crystal Structure with XRD Analysis

The results of the analysis of the crystal structure of cellulose with the XRD test extracted with 4% NaOH at a temperature of 40°C can be seen in Figure 6 below.



Fig 6. Cellulose Stearate XRD test results

Figure 6 shows the XRD pattern of cellulose stearate which shows a high peak appearing at an angle of 2θ around 22.5° , this area is a typical peak of the cellulose structure [27]. According to Rosli, the characteristic peak of cellulose crystals is at an angle of $22-23^{\circ}$ [28]. High crystallinity of cellulose was obtained by hydrolysis using NaOH. The high crystallinity obtained was due to the removal of hemicellulose and lignin in the amorphous region which led to the arrangement of the cellulose molecules.

3.7. Effect of Na₂CO₃ and Methyl Stearate catalysts on the Degree of Substitution (DS)



Fig 7. Effect of Na₂CO₃ and Methyl Stearate catalysts on the Degree of Substitution (DS)

The results of the study. The degree of substitution was determined by titration with the sample added with 40 ml of 0.5 M KOH. The excess alkali was then titrated with 0.5 M HCl using the PP indicator. The degree of substitution is the average number of groups per anhydroglucose that is substituted by another group. If the replacing group in the form of one anhydroxyl group in each anhydroglucose unit is esterified with one acetyl group, the DS value is 1. If there are three esterified hydroxyl groups, then the DS value is 3 [29]. In the graph, it can be seen that the effect of adding methyl stearate is directly proportional to the value of the degree of substitution. The more volume of methyl stearate added, the higher the DS value [30]. On the effect of the Na₂CO₃ catalyst, the DS value is also directly proportional in the sense that the more catalyst used, the greater the DS value obtained. The best DS value is found in the variation of 20 mg Na₂CO₃ catalyst and 15 ml methyl Stearate volume with a DS value of 1.95. This shows the number of ester groups that are substituted

to the OH group on -cellulose is 1.95. It can be concluded in this study that the greater the value of the degree of substitution, the greater

5. Conclusion

the number of substituted groups.

Cellulose stearate ester is the result of synthesis between -cellulose and methyl stearate with the help of Na₂CO₃ catalyst to produce Stearate Cellulose Ester. The characterization of cellulose stearate, based on FT-IR analysis, obtained a vibration peak in the wave number region of 3468.01 cm-1 indicating an OH group, 3062.96 cm^{-1} indicating a CH stretching group, 1695.43 cm^{-1} indicating a C=O group, cm⁻¹ indicates a CH bending group, 1095.57 cm^{-1} indicates a COC group, 609.51 cm^{-1} indicates a (CH₂)n>4 group. Based on the SEM results at 1000 times magnification, and 2000 times the Cellulose Stearate Ester looks more homogeneous, more regular and has denser cavities than

-cellulose before esterification. This research can be continued by changing the source of fatty acids used or varying the volume of fatty acids that have been used.

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