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Analysis of Cellulose and Cellulose Acetate Production Stages from Oil Palm Empty Fruit Bunch (OPEFB) and Its Application to Bioplastics

Aisyah Hanifah[1*], Efri Mardawati[1,3], S Rosalinda[2], Desy Nurliasari[1,3], Roni Kastaman[1]

[1]*Agricultural Industrial Technology, Universitas Padjadjaran, Jl Raya Bandung-Sumedang KM.21, Sumedang*
[2]*Agricultural Engineering, Universitas Padjadjaran, Jl Raya Bandung-Sumedang KM.21, Sumedang*
[3]*Research Collaboration Center for Biomass and Biorefinery between BRIN and Universitas Padjadjaran*

Key Words : *Bioplastic; Cellulose; Cellulose Acetate; Starch; OPEFB*

Abstract

Oil Palm Empty Fruit Bunch (OPEFB) is a type of solid waste from the palm oil processing industry. The components of OPEFB include cellulose, hemicellulose, and lignin. OPEFB has a large cellulose content, so it possesses the potential to be used as a bioplastic material. The purpose of this research was to examine the stages of the bioplastics' production process and its characterization. The cellulose content of OPEFB as raw material and during the isolation process which includes hydrolysis, delignification, pulping, and bleaching are 39.59%, 56.00%, 59.85%, 61.48%, and 68.20%, respectively. Cellulose isolation produces α-cellulose content of 97.87%. The resulting cellulose acetate has an acetyl content of 25.93%. The bioplastics were then characterized to determine the effect of cellulose acetate, starch, chitosan, and glycerol on the physical and mechanical properties of the plastics. The results of the physical properties characterization include biodegradability, water absorption, and density with values of 78.73%, 38.26%, and 1.2% respectively. The results of the mechanical properties characterization include tensile strength, elongation, and modulus of elasticity with values of 0.729 MPa, 4.13%, and 17.5 MPa, respectively. The functional groups in the bioplastics, which are O-H, C-H, C-O, C=O, and N-H, are produced from the mixing process between cellulose acetate, starch, chitosan, and glycerol.

INTRODUCTION

Currently, the problem of waste is still a major problem for environmental sustainability. Waste management is still considered ineffective in solving the problem of plastic waste so other efforts are needed such as synthesizing raw materials from nature or those that can be degraded by microorganisms as materials for making plastics known as bioplastics. Potential sources of fiber in Indonesia are very abundant, one of which is agricultural waste, namely Oil Palm Empty Fruit Bunch (OPEFB). OPEFB is one of the largest types of solid waste generated from palm oil

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Address
Jalan Urip Sumohardjo km. 05 (Kampus 2 UMI)
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Email :
jcpe@umi.ac.id

***Corresponding Author**
aisyahhanifh@gmail.com

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production. OPEFB accounts for around 25%-26% of the total fresh fruit bunches in the palm oil industry [1] so the number will continue to increase in line with the increasing volume of palm oil production. The abundance of OPEFB is contrary to its limited and inadequate utilization: as organic fertilizer, paper raw material, briquettes, and its fiber as a filler. The constituent components of OPEFB include cellulose, lignin, holocellulose, hemicellulose, water, and other extractive substances. Optimizing the utilization of OPEFB is necessary, considering the potential and abundant lignocellulosic content [1]. The cellulose content in OPEFB is 48.56%; hemicellulose 28.08%; and lignin 23.39% [2]. Its high cellulose content makes OPEFB potential to be processed into more valuable products. Cellulose is obtained by isolating it from OPEFB in several stages: hydrolysis, delignification, pulping, and bleaching [2]. Cellulose is insoluble in most solvents, so it needs to be acetylated with anhydrous acetic acid as a solvent and glacial acetic acid as a diluent and an acid catalyst [1]. Cellulose is an organic polymer that can be used as raw material for making bioplastics by synthesizing it into cellulose acetate. The addition of cellulose acetate with other biopolymer matrices such as starch and chitosan as filler can improve the physical properties of bioplastics [3]. The amylose content in starch gives strength to plastics through its long polymer chain structure [4]. Chitosan is used as a reinforcement to provide resistance to water because it has hydrophobic compounds [5]. In addition, plasticizers are usually added to increase the elastic properties of bioplastics [6]. This study analyzed the stages of cellulose production consisting of hydrolysis, delignification, pulping, and bleaching. Cellulose separation requires analysis because the lignocellulosic components (cellulose, hemicellulose, lignin) can be changed at each stage. Therefore, the purity of the cellulose produced can be processed into cellulose acetate which is used as a raw material for making bioplastics.

MATERIAL AND METHODS

The stages of bioplastic production refer to the study by Febrianti [2] which consists of five stages: raw material preparation, cellulose isolation, cellulose acetate production, bioplastics making, and sample characterization.

Material Preparation

OPEFB is washed thoroughly to remove adhering dirt. The OPEFB was then dried and cut into 5-10 cm pieces. Drying was carried out until the moisture content was 4-5% and then it was reduced in size and sieved up to 80 mesh to produce dry OPEFB powder. An analysis of the lignocellulose content of the material was carried out to determine the potential of the raw material.

Cellulose Isolation

Cellulose in OPEFB is extracted through the stages of hydrolysis, delignification, pulping, and bleaching[7]. The first step is hydrolysis by adding OPEFB powder and 3.5% HNO_3 into an Erlenmeyer on a 1:10 ratio. The hydrolysis process lasted for 2 hours at a temperature of 90^{o}C, after which the results were washed, filtered, and dried before the subsequent analysis of their lignocellulosic content. The second stage, delignification, was carried out by refluxing the hydrolysis results with a solution of 1:5 2% NaOH (w/v) and 1:5 Na_2SO_3 2% (w/v) at a temperature of 100^{o}C for 1 hour. After that, the results were again washed, filtered, and dried to analyze the difference in lignocellulosic content from this stage. The third stage, pulping, was carried out by adding the delignification product and 17.5% (w/v) NaOH with a ratio of 1:10 at 80^{o}C for 30 minutes. The results were again tested for its lignocellulosic percentage after washing, filtering, and drying. Lastly, bleaching was carried out by adding the pulp and 10% H_2O_2 with a ratio of 1:10 at 80^{o}C for 90 minutes. The final lignocellulose and α-cellulose content of the product were measured from the washed, filtered, and dried yields.

Cellulose Acetate Production

The production of cellulose acetate consists of three stages: activation, acetylation, and hydrolysis. A-cellulose will be activated by the addition of 1:2.5 glacial acetic acid then heated at 25^{o}C for 2.5 hours using a 125-rpm magnetic stirrer. Then 1:7.5 anhydrous acetic acid and 1:1.5 H_2SO_4 were added at a temperature of 25^{o}C for 2.5 hours. After that, 1:1 distilled water and 1:2.5 glacial acetic acid were added at 25^{o}C for 30 minutes. Then 1:0.5 sodium acetate was added and stirred for 5 minutes. Washing, filtering, and drying then followed by measurement of the yield and acetyl percentage of the cellulose acetate.

Bioplastics Film Forming

Bioplastics are made from cellulose acetate, starch, chitosan, and glycerol. Cellulose acetate, starch, and glycerol were each diluted by adding 1:25 1% acetic acid, then mixed and stirred on a magnetic stirrer at 70°C for 15 minutes. The mixture was then added with glycerol of 25% of the total mass and stirred for 10 minutes. The now gel-shaped mixture was then poured into a mold and dried at 50°C for 1x24 hours. The bioplastic samples were then tested for physical properties, mechanical properties, and functional groups.

Sample Characterization

The lignocellulose content was determined by observing the difference in the lignocellulose content obtained at each stage using the Chesson method. The characteristics of the resulting bioplastic will be tested for its physical properties including biodegradability, water absorption, and density. Mechanical properties tests include tensile strength, elongation, and modulus of elasticity. The last is the analysis of the functional groups that bind between cellulose acetate, starch, chitosan, and glycerol.

RESULT AND DISCUSSION

Characteristics of Lignocellulose

Based on the Chesson method, OPEFB from this research contains 39.59% cellulose, 15.74% hemicellulose, and 17.57% lignin. The lignocellulosic content in OPEFB is then compared with several other biomass wastes which can be seen in Table 1.

Table 1. Lignocellulosic content of various biomass waste

Biomass	Cellulose (%)	Hemicellulose (%)	Lignin (%)	Reference
OPEFB	39.591±1.369	15.737±3.342	17.574 ±0.436	Analysis results
Snake fruit (salak) fronds	35.32	30.49	20.05	[8]
Rice Straw	29.2-34.7	23.0-25.9	17.0-19.01	[9]
Bagasse	25.0-45.0	28.0-32.0	15.0-25.0	[9]
Peanut Shells	25.0-30.0	25.0-30.0	30.0-40.0	[10]

Table 1 shows that the percentage of OPEFB cellulose content in this research was higher than that of Salak fronds, rice straw, bagasse, and peanut shells. The high cellulose content in OPEFB displays its potential to be processed into cellulose acetate and made into bioplastic raw materials. Raw OPEFB can be seen in Figure 1.

Figure 1. Raw OPEFB

Analysis of Lignocellulose Content

The stages of cellulose isolation successively consist of hydrolysis, delignification, pulping, and bleaching. The cellulose isolation process is an important step in breaking down and reducing the lignin and hemicellulose content. The success of cellulose isolation can be seen through the data of increasing cellulose content and decreasing lignin and hemicellulose content.

Hydrolysis

The hydrolysis aims to degrade lignin bonds and increase the material porosity until the hemicellulose breaks down into simple sugars (glucose, galactose, mannose, hexose, pentose, xylose, and arabinose). The use of HNO_3 causes the hemicellulose content and

extractive substances to be more soluble than other acids[11]. A comparison of lignocellulosic content before and after the hydrolysis process can be seen in Table 2.

Table 2. Lignocellulose content after hydrolysis

Lignocellulose	On Raw Materials	Hydrolysis Results
Cellulose (%)	39.591 ±1.369	56.002 ±2.578
Hemicellulose (%)	15.737 ±3.342	9.412 ±1.431
Lignin (%)	17.574 ±0,436	25.652 ±0.492

The lignin content increased due to heat and acid treatment which made phenolic compounds in plant tissues (e.g., ferulic acid, chlorogenic acid) form cross-links such as the binding of esters to cell wall polysaccharides to form "lignin objects". The lignin object will then be detected as lignin, causing the lignin content to increase.

Delignification

The delignification aims to dissolve components (hemicellulose and lignin) from EFB to obtain cellulose [1]. Lignin contained in OPEFB fibers can cause turbidity and reduce the quality of the cellulose produced [12]. Lignin is removed in the delignification step using 2% NaOH and 2% Na_2SO_3. The comparison of lignocellulosic content before and after the delignification process can be seen in Table 3.

Table 3. Lignocellulose content after delignification

Lignocellulose	Hydrolysis Results	Delignification Results
Cellulose (%)	56.002 ±2.578	59.849 ±2.900
Hemicellulose (%)	9.412 ±1.431	7.027 ±0.312
Lignin (%)	25.652 ±0.492	23.446 ±3.187

It's identified that the content of hemicellulose and lignin decreased. That is in line with the purpose of the delignification process, to degrade the hemicellulose and lignin content to produce high cellulose content.

Pulping

The pulping process started by adding 17.5% NaOH to dissolve β-cellulose and γ-cellulose to produce α-cellulose[13]. The comparison of lignocellulosic content before and after the pulping process is shown in Table 4.

Table 4. Lignocellulose content after pulping

Lignocellulose	Delignification Results	Pulping Results
Cellulose (%)	59.849±2.900	61.478±3.888
Hemicellulose (%)	7.027±0.312	7.050±1.239
Lignin (%)	23.446±3.187	20.772±3.832

Table 4 shows that the hemicellulose content at the pulping stage has increased. That is due to the degradation of γ-cellulose by 17.5% NaOH solution into simple sugars whose structure resembles and detected as part of hemicellulose.

Bleaching

The last process in cellulose isolation is bleaching using hydrogen peroxide to remove the remaining ingredients (pigment and residual lignin). The comparison of lignocellulosic content before and after the bleaching process can be seen in Table 5.

Table 5. Lignocellulose content after bleaching

Lignocellulose	Pulping Results	Bleaching Results
Cellulose (%)	61.478±3.888	68.197 ±3.058
Hemicellulose (%)	7.050±1.239	8.138 ±0.347
Lignin (%)	20.772±3.832	14.837 ±1.928

Based on Table 5, the cellulose content has increased to 68.2%. The hemicellulose content increases, usually it should decrease. That can be caused by errors during the characterization process so that the percentage of results detected is high.

Analysis of α-Cellulose Content

Cellulose is divided into three types based on differences in the degree of polymerization and solubility under specific conditions, namely: α-

cellulose, β-cellulose, and γ-cellulose. β-cellulose and γ-cellulose will be dissolved in sodium hydroxide solution, leaving α-cellulose. The α-cellulose concentration affects the purity of cellulose; the greater it is, the greater the purity [13]. The α-cellulose produced in this research can be seen in Figure 5.

Figure 2. A-cellulose

The α-cellulose was characterized and its percentage yield calculated. The physical appearance of the α-cellulose produced is in the form of fiber powder and is white in color. The overall yield of the cellulose isolation process was 36.1%, lower than the cellulose content produced in the previous study [2] which was 44.01%. The low yield resulted from the size of the raw material being too small, so mass loss occurs during the washing and filtering process. The moisture content of the cellulose produced in this research was 3.648%. These results indicate that the cellulose meets the requirements to enter the cellulose acetate production. The -OH group in water will be more reactive to anhydride reagent than to the -OH group in cellulose, so the moisture content lowers while the reactivity of the -OH group in cellulose increases. That causes the acetylation reagent to enter the cellulose fibers [7]. The cellulose extraction process in this study gave the results of α-cellulose content of 97.87% or it could be said to have a high level of purity. This value is greater than the previous studies which were 94% and 44.01%, respectively. This is due to the immersion of cellulose in 17.5% NaOH which dissolves the components of β-cellulose and γ-cellulose and in the end produces pure α-cellulose. The cellulose content produced in this research met the prerequisites and can be used as a raw material for making bioplastics.

ANALYSIS OF CELLULOSE ACETATE PRODUCTION

Cellulose acetate is an esterification product of cellulose and anhydrous acetate [14]. Cellulose acetate is produced in three steps: activation, acetylation, and hydrolysis. The synthesis of cellulose is carried out first because cellulose is insoluble in most solvents, so it needs to be acetylated with anhydrous acetic acid as a solvent and glacial acetic acid as a diluent and an acid catalyst [1]. The cellulose acetate made can be seen in Figure 3.

Cellulose acetate powder has a white color and is finer than the cellulose one (Figure 3). Cellulose acetate was then analyzed for its moisture content, yield, and acetyl content. The results of the analysis can be seen in Table 6.

Figure 3. Cellulose acetate

Table 6. Results comparison of moisture content, yield, and acetyl content of cellulose acetate

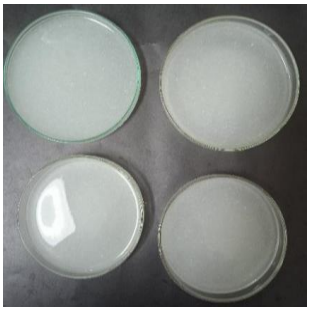
Moisture (%)	Yield (%)	Acetyl (%)	Reference
0.321 ±0.265	119.900 ±19.73	25.932 ±2.939	Analysis results
1.210	88.560	39.580	[2]
4.470	145.354	43.194	[7]
4.430	153.800	40.360	[15]

The moisture content of cellulose acetate in this research was smaller than in previous studies. Low moisture content can assist the hydroxyl group of acetate in cellulose to bind with the acetic acid solvent and form a stronger bond. n this study, a yield of 119.9% was obtained, which was within the range of results [2], [7], [15]. That amount comes from the synthesis of acetic acid in the cellulose structure. The value of acetyl content in this research was less than 35% so it was classified as cellulose monoacetate [16].

CHARACTERIZATION OF BIOPLASTICS

Bioplastics are made using cellulose acetate, starch, chitosan, and glycerol. The characteristics of the gel formed are thick and white. The gel was then poured into a glass mold with a thickness of ±0.5 mm and dried at 50^{o}C for 1x24 hours. The gel thickness will decrease after drying due to the evaporation of the solvent in the gel. Changes in the shape of bioplastics before and after drying can be seen in Figure 4.

The bioplastic product is white because the cellulose acetate binds and spreads to the matrix evenly. Bioplastics are flexible because the amount of plasticizer used is too much. The moisture content of bioplastics is still high, namely 11.402%, due to the glycerol that is difficult to evaporate at low temperatures. The plasticizer with these properties causes the physical bond to the polymer to be disturbed, resulting in the emergence of excess liquid [7]. The bioplastics were then characterized for their physical and mechanical properties such as biodegradability, water absorption, density, tensile strength, modulus of elasticity, and elongation.



(a)

(b)

Figure 4. Bioplastic film forming (a) before and (b) after drying Analysis of Bioplastics Physical Properties

A physical characterization test of bioplastics consists of the value of biodegradability, water absorption, and density. The biodegradability test on bioplastics aims to provide a reference for the level of resistance of bioplastic composites to decomposition in the soil within a certain time. Water absorption is a reference for the stability and resistance of bioplastics when exposed to water. The density measured is the polymer chain density. Simultaneously, the biodegradability of polymeric materials is affected by the polymer structure. The amorphous structure will degrade easier than the crystalline structure. The percentage of mass lost during the degradation process in bioplastics reached 78.727%, higher than the biodegradability in previous studies [2]. The biodegradation rate of bioplastics before and after testing can be seen in Figure 5.

(a)

(b)

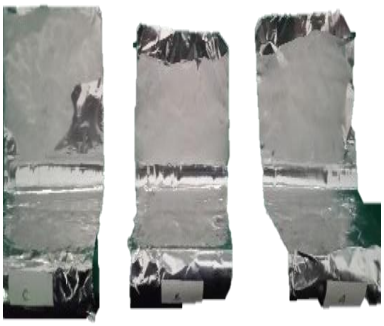
Figure 5. Bioplastics (a) intact and (b) degraded

Based on the pictures, bioplastics degrades after several days of being buried in the ground. Cellulose acetate is easy to decompose [4], following the research [2] that states that the greater the concentration of cellulose acetate, the faster the

bioplastic degrades. The addition of starch to the bioplastic formulation affected the biodegradation of the bioplastic produced due to the amylose and amylopectin content.

Amylose and amylopectin have hydroxyl groups (OH) that decompose into small pieces until they disappear when buried in the soil [17]. The addition of glycerol can stretch the molecular bonds between the O-H groups of cellulose acetate and starch. Glycerol has the property of absorbing water or being hydrophilic so that it affects the degradation process in bioplastics. Chitosan as a reinforcing agent will take longer to degrade than cellulose acetate, starch, and glycerol. That's because chitosan has hydrophobic properties and is resistant to pest attacks, so it is hard to decompose [18]. Water absorption before and after the test is in Figure 6.

(a)



(b)

Figure 6. Water absorption (a) before and (b) after physical properties analysis

Based on Figure 6, after the test, the bioplastic experienced an increase in volume because it binds water. The water absorption capacity of the bioplastics produced in this research was 38.265%, greater than the previous research. The bioplastics' ability to absorb water is related to the density of the bonds formed. The higher the water absorption value, the more easily damaged the bioplastic when exposed to water and the effect on the decrease in the mechanical properties of the bioplastic. Cellulose as a filler material can increase water absorption in the resulting bioplastic. That is because cellulose can form intermolecular hydrogen interactions with water in the amorphous part, while cellulose in the crystalline state has a relatively tighter space so that water vapor permeability can decrease [19]. Water absorption can also increase due to starch that is water-absorbing or hydrophilic. The hydroxyl group in starch can form hydrogen bonds with water, causing high water absorption in bioplastics [4]. Meanwhile, chitosan has hydrophobic properties or is insoluble in water [18]. The crystal structure of chitosan will make the distance between bioplastic molecules closer by filling the pores between cellulose and starch in the formation of bioplastic films [2].

The measured density is at the molecular level of the material, so it certainly affects the polymer properties of bioplastics. Density is affected by the components that make up the bioplastic. The higher the concentration of dissolved solids, the greater the thickness produced [20]. Based on this research, bioplastics with starch addition had a higher density (1.201%) than bioplastics from previous research that use no starch [2]. Amylose and amylopectin in starch cause thickening of bioplastics film. That is due to amylopectin and amylose in starch that can gelatinize to produce a viscous paste and increase the thickness of the bioplastic [19].

Analysis of Bioplastics Mechanical Properties

Mechanical characterization of bioplastics consists of their tensile strength, elongation, and modulus of elasticity. Tensile strength was measured to determine the ability of bioplastics to withstand heavy loads. Elongation is an indicator of bioplastics flexibility and is determined by the strain point on the film when it breaks. The modulus of elasticity is the resistance value of the material when it is elastically deformed when a force is applied.

The mechanical properties of bioplastics related to their constituent (cellulose acetate, starch, chitosan, and glycerol). The composition of the bioplastic has an impact on its mechanical properties. That is due to the

physical interaction between the constituent components of cellulose acetate, starch, chitosan, and plasticizers. The addition of starch to the bioplastic composition increased the tensile strength, percent elongation, and modulus of elasticity. Research Febrianti [2] with cellulose acetate, chitosan, and glycerol components as material obtained bioplastics with a tensile strength of 0.198 MPa, an elongation of 1.027%, and a modulus of elasticity of 6.24 MPa. While starch components addition in bioplastics using SNI ISO 1924.2: 2016 resulted in bioplastics with a tensile strength value of 0.729 MPa, elongation 4.13%, and modulus of elasticity 17.5 MPa.

Cellulose acetate can increase the tensile strength of bioplastics because the polymer chains are straight and long. This increase was due to good interfacial adhesion, thus forming strong hydrogen bonds between the starch and cellulose matrices. In addition, the tensile strength of the bioplastic relates to the acetyl content of the cellulose acetate; the higher the acetyl content, the higher the tensile strength will be [4].The amylose content in starch can increase the tensile strength of bioplastics because amylose is a necessary gelatinizing and retrograding agent during film formation. Amylose has hydrogen bonds between each polymer hydroxyl group which forms a junction zone between molecules, increasing the tensile strength in bioplastics [21]. The addition of a glycerol plasticizer can reduce the tensile ability of bioplastics. Glycerol, which can form many hydrogen bonds and interactions, creates distance between the particle chains. This distance causes the attractive intermolecular interactions to become weak [22]. Meanwhile, the high concentration of chitosan influences hydrogen bonding in bioplastic films. Chitosan can form chemical bonds that are very strong and difficult to remove so that the tensile strength is greater [18].

The addition of glycerol to bioplastics can increase elongation due to the nature of glycerol which can stretch the bonds between molecule [18] whereas cellulose, starch, and chitosan can reduce the value. The decrease in elongation value is due to the increase in hydrogen bonding in the bioplastic film. The modulus of elasticity in this research has a value of 17.5 MPa, higher than the research results by Febrianti (2019). According to Sutan et al. [23], the value of the elastic modulus can be influenced by the composition of the bioplastic production material. The addition of starch can increase the value of the modulus of elasticity because the amylopectin content in starch has branched chains that increase the elasticity of bioplastics. On the other hand, cellulose acetate can reduce the value of the modulus of elasticity because the hydrogen bonds formed make the chain longer and cause the bioplastic to have a slightly stiffer nature [7].

Analysis of Bioplastic Functional Group

Fourier transform infrared (FTIR) test was conducted to identify the interaction between starch as a matrix, cellulose as a reinforcing agent, and other constituent components such as chitosan and glycerol. In the FTIR test, the x-axis shows the wavelength of absorption (cm^{-1}) and the y-axis shows the percentage of transmission. The FT-IR spectrum of the researched bioplastic is shown in Figure 7.

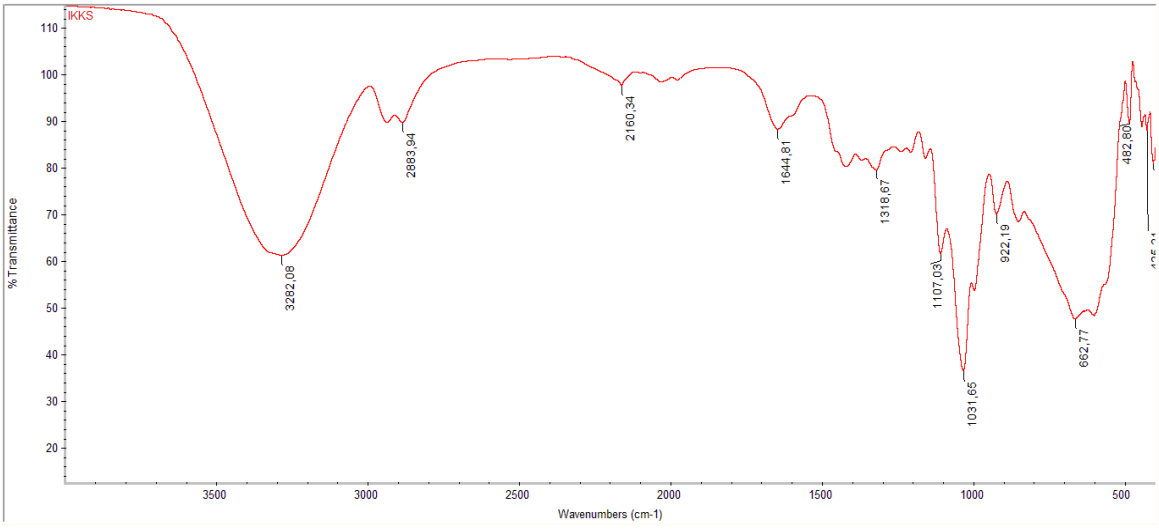


Figure 7. FTIR spectra of the bioplastics sample

The FTIR result shows that the plastic with the composition of cellulose acetate, starch, chitosan, and glycerol has a hydroxyl group (-OH stretching) at a wavelength of 3282.08 cm^{-1}. The identified O-H groups allow the bioplastic to bind to water. The wavelengths at 2883.94 cm^{-1} and 2160.34 cm^{-1} indicate the presence of C-H stretching. The wavelength of 1644.81 cm^{-1} indicates the presence of a C=O functional group (carbonyl group) which identifies the cellulose acetate content in bioplastics. The N-H group was identified at a wavelength of 1318.67 cm^{-1}. The N-H group appears due to the mixing of chitosan in bioplastics. The C-O group was identified at wavelengths of 1107.03 cm^{-1} and 1031.65 cm^{-1}. Cellulose has a peak of 922.19 cm^{-1} which indicates the presence of β-glycosidic. Bioplastics with cellulose acetate as filler material will produce functional groups that still indicate the presence of cellulose acetate, starch, chitosan, and glycerol. According to Febrianti (2019), the presence of O-H, C=O, C-O, C-H, and N-H structures indicates the structure of bioplastics with the composition of cellulose acetate, chitosan, and glycerol. Based on the FTIR results in this research, it was found that the functional groups OH, CO and esters can degrade bioplastics.

CONCLUSION

Cellulose from OPEFB as raw material and after the isolation process which includes hydrolysis, delignification, pulping, and bleaching are 39.59%, 56.00%, 59.849%, 61.48%, and 68.20%, respectively. Cellulose isolation produced α-cellulose content of 97.87%. The cellulose acetate produced has a moisture content of 0.321%, a yield of 119.9%, and an acetyl content of 25.932%. Bioplastics made from cellulose acetate, starch, and chitosan have the physical appearance of white film sheets and are flexible. The physical properties of bioplastics, which are biodegradability, water absorption, and density, have values of 78.73%, 38.26%, and 1.2%, respectively. Meanwhile, the mechanical properties of bioplastics include tensile strength, elongation, and modulus of elasticity with values of 0.729 MPa, 4.13%, and 17.5 MPa, respectively. The functional groups in bioplastics are O-H, C-H, C-O, C=O, and N-H groups produced from the mixing process between cellulose acetate, starch, chitosan, and glycerol.

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