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# Production of Cellulose Acetate from Oil Palm Empty Fruit Bunches Cellulose

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**Abstract**

Cellulose acetate was produced from acetylation process oil palm empty fruit bunches cellulose which underutilized. To obtained cellulose acetate qualified was needed cellulose with high purity level which was produced through several stages are pre-hydrolysis, delignification, pulping and bleaching. The purpose of this research was to making cellulose acetate and determining the exact acetylation time from oil palm empty fruit bunches cellulose. The research was conducted several stages are cellulose extraction, cellulose acetate production with acetylation process and characterization of acetate cellulose. The production process of cellulose acetate was done with acetylation time are 15, 30, 45 and 60 minutes. The results were obtained cellulose acetate by water content range from 4.332 to 4,468%, the highest yield for 45 minutes was 189.699% and the best acetyl content for 30 minutes was 40.108%. FTIR analysis was showing carbonyl group at 1759 $cm^{-1}$ and C-O group at 1234 $cm^{-1}$ region. Morphology of cellulose acetate obtained was shaped cylindrical fibers and had perforated layer.

**Keywords:** Oil Palm Empty Fruit Bunches, Cellulose Acetate, Acetylation

## 1. Introduction

Cellulose acetate is a biopolymer that produced through acetylation process of biomass cellulose. Various types of biomass have been used as source of cellulose on producing cellulose acetate such as cotton wastes (Cheng *et al*. 2010), recycled papers (Filho *et al.* 2008), agricultural wastes (Israel *et al.* 2008), and woods (Sato *et al.* 2008). However, the amounts of biomass are still low so it cannot as the main raw material on production commercial cellulose acetate that derived from woods. One of biomass that underutilized is palm empty fruit bunches. The use of palm empty fruit bunches cellulose can be alternative to wood cellulose that if used excessively can affect the environmental balance.

Oil palm empty fruit bunches have high cellulose content are consists of 67.88% holocellulose and 38.76% α-cellulose that have fiber content 72.67%. Alpha-cellulose is the most high-quality of cellulose (pure). The content of high α-cellulose in oil palm empty fruit bunches can be processed become pulp that can be used as raw material for production of cellulose acetate. So far the oil palm empty fruit bunches have been used to production of bio-oils (Sukiran *et al.*2009), carboxilmethylcellulose (Bono *et al*. 2009), briquetting (Nasrin *et al*, 2011), compost (Amira *et al*. 2011), ethanol (Millati *et al*. 2011), biofuel (Yang *et al*. 2006) and Indonesia Oil Palm Research Center using Oil palm empty fruit bunches wastes to polypot, particle boards and latex fiber.

Cellulose acetate is one of macromolecules from derivative of cellulose. It's one type of natural polymer that has very organized of micro fibril structures. Cellulose acetate has excellent quality with good transparency, tensile strength, heat resistance, low water absorption, and easily biodegradable. The cellulose acetate properties are very need of various industries such as coating, plastics, films, fiber textiles, filter tow, LCDs, photo films, textiles (Puls *et al*. 2011), packaging, membranes and cigarettes (Hinterstoisser *et al.* 2003).

Cellulose acetate quality is very influenced by acetyl content and degree of substitution because it can affect to the resulting product and the solubility of cellulose acetate in solvent. Acetyl content of cellulose acetate is influenced by several factors such as cellulose with anhydride acetic ratio, acetylation time and interaction between treatment factors (Susanti 2003). Production of cellulose acetate needs different acetylation time that is depended on source of cellulose to obtain the appropriate acetyl content. Various research have been done about acetatee cellulose such as microbial cellulose takes 5-10 hours (Safriani 2000), bacterial cellulose from pineapple

waste for 2 hours (Pasla 2006), straw for 2-3 hours (Harrisson *et al.* 2004). The purpose of the research is to making cellulose acetate and determining the exact acetylation time from oil palm empty fruit bunches cellulose.

## 2. Methodologhy

*2.1 Materials and Equipment*

The using materials in the research were oil palm empty fruit bunches from oil palm factory "PTPN VII" in banten and chemical materials on cellulose extraction, $\alpha$-cellulose content analysis and cellulose acetate production.

The using tools in the research were tools on cellulose extraction, $\alpha$-cellulose content analysis and cellulose acetate production. The resulting product was tested morphology with Scanning Electron Microscope (SEM) and functional group transformation with Fourier Transform Infrared (FTIR).

*2.2 Research Methods*

The research was done several stage, (i) extracting cellulose oil palm empty fruit bunches, (ii) producing cellulose acetate, and (iii) characterization of cellulose acetate.

2.2.1 Extraction of Oil Palm Empty Fruit Bunches Cellulose

Extraction was done in 5 (five) stage by preparation of oil palm empty fruit bunches, pre-hydrolysis with $HNO_3$ 3,5%, delignification with NaOH 2% and $Na_2SO_3$ 2%, pulping with NaOCl 1,75% and NaOH 17,5%, and bleaching $H_2O_2$ 10%. The resulting cellulose was tested purity level by measuring $\alpha$-cellulose content (Harahap *et al.* 2012).

2.2.2 Production of Cellulose Acetate

Production of cellulose acetate was done some stage by activation, acetylation, hydrolysis, sedimentation and drying.

1. Activation, the resulting cellulose was added galcial acetic acid with ratio 1:10 and shaken at 38 $^0$ C for 60 min. Then it was added 2 % sulfuric acid and shaken at 38 $^0$ C for 45 min.
2. Acetylation, the results of the activation process was done acetylation process by adding anhydrid acetic acid with anhydrid and glacial ratio 3:2 (Safriani 2000). Then it was shaken at temperature 38 $^0$ C with acetylation time according to treatment (15, 30, 45 and 60 minutes).
3. Hydrolysis, acetylation process was stopped by adding aquadest and glacial acetic acid ratio 1:2 and shaken at temperature 50 $^0$C for 30 minutes.
4. Sedimentation, the results of hydrolysis process was put in centifuge with speed 1500 rpm for 15 min then sedimented into aquadest and filetered until aceic acid flavour was lost.
5. Drying stage, the resulting sediment was dried at temperature of 105 $^0$ C.
6. Analysis of Cellulose Acetate was done by measuring the cellulose acetate yield (ASTM, 1991), water content (ASTM D-678-91), acetyl content (ASTM D-678-91)

*2.3 Characterization*

2.3.1 Fourier Transform Infrared (FTIR)

The chemical investigation of functional groups in oil palm empty fruit bunches cellulose and cellulose acetate were studied by using Fourier Transmission Infrared Spectroscopy (Spectrum Perkin Elmer). The sample disc was prepared by mixing and compressing the sample and KBr at a 1:1 ratio. FTIR spectra were produced after fifty times by scanning at a 4 $cm^{-1}$ resolution for transmission wavelength range 4000 to 450 $cm^{-1}$.

2.3.2 Scanning Electron Microscope (SEM)

The surfaces of the samples (oil palm empty fruit bunches cellulose and cellulose acetate) were coated with a thin (approximately 20 nm thick) of gold in an ion sputter, model JFC-1100E (Jeol Ltd.). SEM images of oil palm empty fruit bunches cellulose and cellulose acetate were recorded at 100x and 2,000× magnification using an acceleration voltage of 20 kV.

## 3. Result and Discussion

### *3.1 Extraction of Oil Palm Empty Fruit Bunches Cellulose*

Cellulose extraction was done some stage by preparation of palm empty fruit bunches, prehydrolysis, delignification, pulping and bleaching. Palm empty fruit bunches have a structure which is very hard, thorny and still contains palm oil after it was processed from the factory. Preparation of oil palm empty fruit bunches is done by cleaning from the shells and the remaining oil then dried in the sun. Dry palm empty fruit bunches was separated fibers then cut to form short fibers size of 5-10 cm which aims to facilitate the delignification process of cellulose.

Palm empty fruit bunches fibers contain components other besides cellulose (45.95%) are lignin (22:23%), holocellulose (66.07%) and extracted materials (7.78%) (Darnoko *et al*, 2001). Cellulose can be obtained from oil palm empty fruit bunches by delignification process. Delignification was done in two stages, pre hydrolysis with cooking oil empty fruit bunches fiber by $HNO_3$ 3.5% that aims to removing other components besides cellulose and leaving fibrous solids. Delignification was continued by adding mixture of NaOH 2% and $Na_2SO_3$ 2% that aims to remove residual lignin which dissolved and wasted into the solution of dark brown color. The resulting cellulose was pure white. To removing brown color of cellulose then bleaching process was done by NaOCl 17.5%. Hypochlorite ion was a strong oxidant which capable of breaking ether bonds in lignin structure, so that increasing the degree of white pulp (Harahap *et al.* 2012).

The purity cellulose level was determined by content of α-cellulose from the resulting cellulose that can be obtained by addition of NaOH 17.5%. Cellulose can be divided into three types; α-cellulose, β-cellulose and gamma-cellulose. The addition of NaOH 17.5% causes swelling of the cellulose structure that will unlock the cellulose fibers so that β-cellulose and gamma-cellulose were dissolved and resulting α-cellulose of yellowish white. Swelling of cellulose can increase the accessibility -OH groups so that reaction process of cellulose was easily formed. Last stage was bleaching process using $H_2O_2$ 10% which aims to obtain the white cellulose and then dried in the oven at 105 $^0$C. The resulting cellulose can be shown in Figure 1.

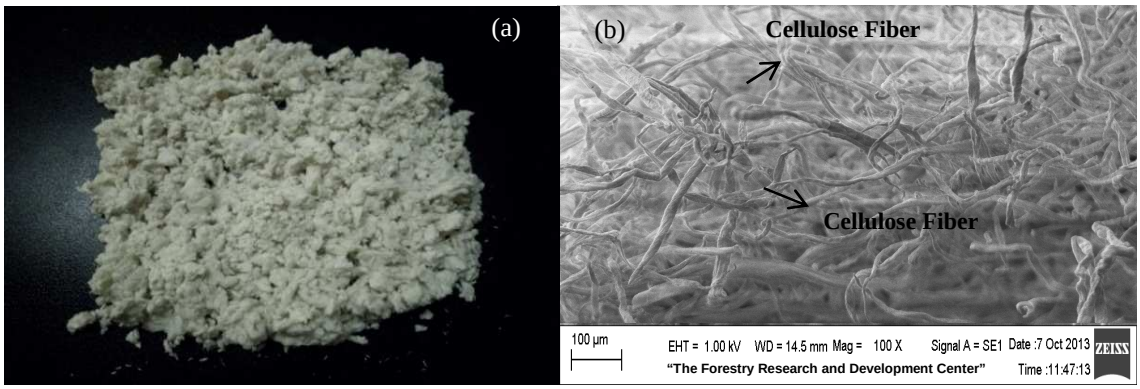



Figure 1. (a) Cellulose and (b) Cellulose with SEM 100x

Production of high cellulose acetate quality was very influenced by the level of cellulose purity. The more level of purity cellulose was increased, the obtain cellulose was increased also. The resulting cellulose in the research was purified by dissolving cellulose in NaOH 17.5% so that can be obtain α-cellulose content of 94.8%. This shows the cellulose has high level of purity so that it can be used as cellulose derivative products like cellulose acetate. The resulting cellulose has water content 4.8%. Low water content influence acetylation process in production of cellulose acetate because –OH group in water more reacts with anhydrous reagent than –OH group in cellulose. The more water content was decreased, -OH group reactivity was increased also so that facilitate acetylation reagent into cellulose fibers.

### *3.2 Production and Characterization of Cellulose Acetate*

The obtaining cellulose acetate was shaped white powder. According of Fenger and Wegener (1995) in Susanti (2003), cellulose acetate was caused by oxidative changes of cellulose molecules so that it results the color compounds during acetylation. The color changes from white to brown has seen when addition anhydride acetic.

In this research, cellulose acetate was produced several stages by activation, acetylation, hydrolysis, sedimentation and drying. Before the acetylation process, the activation process was done to expand the surface area of cellulose fibers and reduce the intermolecular hydrogen bonds so that facilitate acetylation process with anhydride acetic. Activation was done by using glacial acetic acid as activator and sulfate acid as catalyst. This statement like Safriani (2000) that acetylation was done in acid condition by adding anhydride acetic and glacial acetic acid ratio (2:3) and sulfate acid as catalyst (5-20 % of the cellulose weight).

Table 1. Result Cellulose Acetate Analysis of Yield, Water, and Acetyl Content with Acetylation Time Variation.

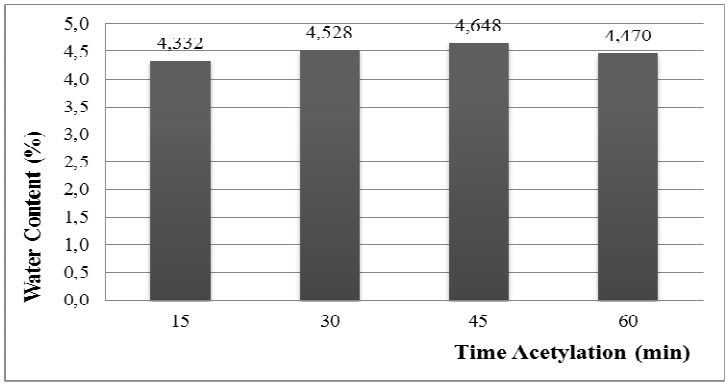
| Treatment | | Water Content (%) | Yield (%) | Acetyl content (%) |
|---|---|---|---|---|
| Acetylation time (min) | 15 | 4,332 | 57.325 | 37.525 |
| | 30 | 4.528 | 156.202 | 40.108 |
| | 45 | 4.648 | 189.699 | 42.261 |
| | 60 | 4,470 | 145.354 | 43.194 |

Source : The Results of Cellulose Acetat, 2013.

3.2.1 Water Content

Water content of cellulose acetate in the research from 4.332 to 4.468% (Figure 2). In the figure 2 was shown that the longer of acetylation process, the higher of water content but it was not significant effect on the water content of the resulting cellulose acetate. Water content of cellulose acetate was very low because it was done to drying of production process. The drying of temperature and long was done at temperature of 105 $^0$C for 6 hours.

Low water contents also were influenced by storage factor. Cellulose acetate was not hygroscopic properties so




that it can be changed to depending on storage conditions.

Figure 2. Water Contents of Cellulose Acetate

3.2.2 Yield

Yield of cellulose acetate was ratio between weight of the resulting cellulose acetate with weight of the using cellulose in acetylation process. The obtaining yield was from 57.255 to 189.699 % (Figure 3). The longer of acetylation process, the higher of yield of cellulose acetate. Acetylation process which was long causes cellulose degradation process with anhydride acetate increased so that the resulting cellulose acetate increased. Yield of cellulose acetate was directly proportional with water content because the higher of water content, yield was increased also.

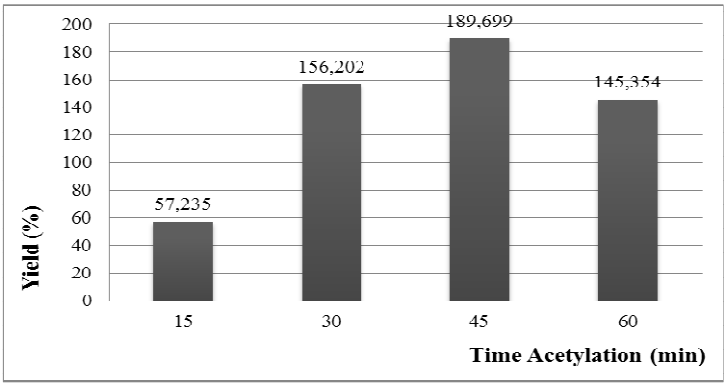



Figure 3. Yield of Cellulose Acetate

3.2.3 Acetyl Content

Acetyl content was measure of the esterified acetate amount in cellulose chains that will determine the value of substitution degree. The degree of substitution is the average amount of -H atoms on the hydroxyl group (-OH) which was converted to acetyl groups in each anhydroglucose residue (Arifin 2004). Acetyl content of the resulting cellulose acetate was 37.525 to 43.194% (Figure 4). The amount of the obtaining acetyl content was increased with increasing duration of acetylation process. Acetylation time was long cause cellulose degradation occurs gradually because cellulose containing water content was very low (4.332 to 4.468%), but -OH group on the water more easily react with acetic anhydride reagent than -OH group cellulose.

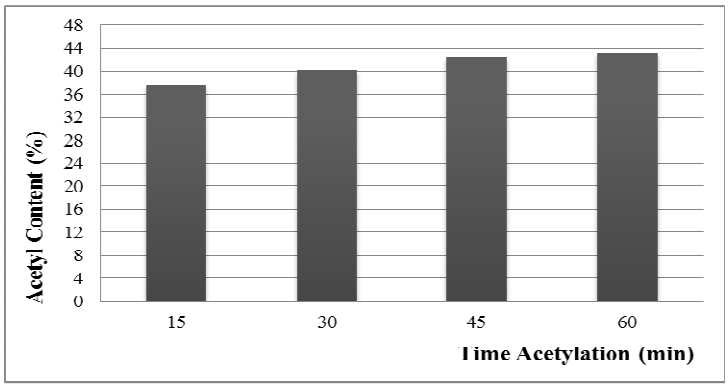



Figure 4. Acetyl Content of Cellulose Acetate

Value of the esterification degree the resulting cellulose acetate was influenced by acetyl content. According to Ullmann's Encyclopedia (1999) in Safriani (2000) that the degree of esterification or the amount of acetic determines the solubility and compatibility with plasticizers, resins and others which affect the mechanical properties. Cellulose acetate can soluble in organic solvents depending on the degree of esterification so that can be obtained good and homogeneous solution so that was resulted quality product. Relationship between the degree of substitution, acetyl content, and solvent was shown in Table 1.

Table 2.4 Relations Degree of Substitution, Acetyl Content, Solvent and Application of Cellulose Acetate

| Degree of Substitution | Acetyl Content (%) | Common Solvents | Application |
|---|---|---|---|
| 0.6-0.9 | 13.0-18.6 | Water | - |
| 1.2-1.8 | 22.2-32.2 | 2-Methoxy-ethanol | Plastic |
| 2.2-2.7 | 36.5-42.2 | Acetone | thread, film |
| 2.8-3.0 | 43.0-44.8 | Cloroform | fabric, wrapping |

Source: Fengel and Wegener 1995.

Good cellulose acetate was cellulose acetate type with its good quality according to Indonesia nation standard by acetyl content parameter of 39-40%, intrinsic viscosity (solvent acetone) 1.5-1.8 dl / g and stability against heat was not occur carbonization when heated (180 $^0$C for 8 hours). Acetyl content and solubility of cellulose acetate in organic solvents was major parameter in determining good cellulose acetate. The cellulose acetate category was obtained by acetylation time of 30 minutes with acetyl content of 40.108%.

*3.3 Chemical Characterization by FTIR*

Cellulose and cellulose acetate was tested by FTIR that aims to analyze functional groups changes from cellulose become cellulose acetate with looking the forming spectrum (Figure 5).

The results of functional groups analysis on cellulose uses FTIR can be seen in Figure 5a. In the figure 5a was shown O-H groups at 3348 cm$^{-1}$ and C-O groups 1065 cm$^{-1}$. The groups were type of alcohol compound that was the forming cellulose group. There was also C-H functional groups at 2901 cm$^{-1}$ which were hydrocarbons compounds.

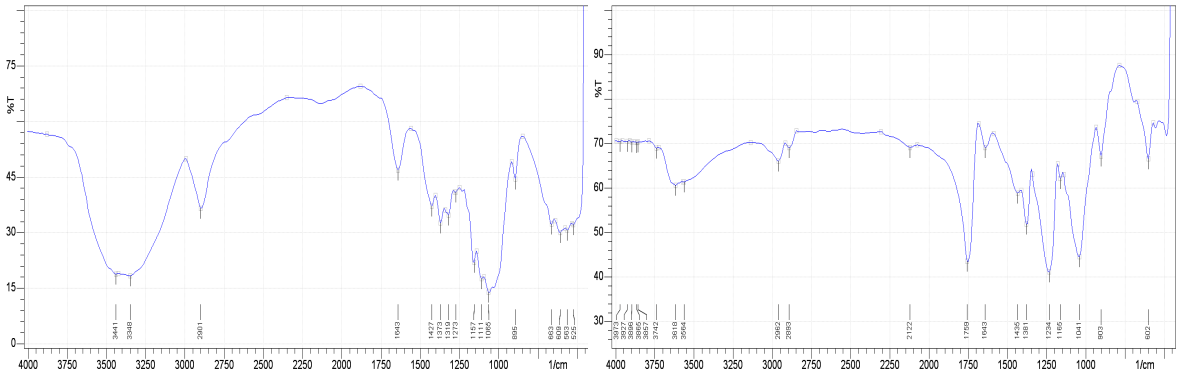



Figure 5. FTIR Analysis of (a) Oil Pa (a) ıpty Fr (b) :hes Cellulose and (b) Cellulose Acetate

The results of functional groups analysis on cellulose uses FTIR can be seen in Figure 5a. In the figure 5a was shown O-H groups at 3348 cm$^{-1}$ and C-O groups 1065 cm$^{-1}$. The groups were type of alcohol compound that was the forming cellulose group. There was also C-H functional groups at 2901 cm$^{-1}$ which were hydrocarbons compounds.

The results of analysis of functional groups on cellulose acetate using FTIR can be seen in Figure 5b. The result was shown O-H groups that not perfectly acetylated like seen at 3618 cm$^{-1}$ with the level of depth peak was quite low. This condition was comparable with acetyl content value cellulose acetate so that can be stated that there are –OH groups on the not substituted cellulose. C-O group was showed at 1234 cm$^{-1}$ which was ether groups and C-H groups at 2962 cm $^{-1}$. The establishment of C=O in cellulose acetate by wave numbers at 1759 cm$^{-1}$ was showed carbonyl group. The carbonyl groups were groups which make up the cellulose acetate.

The second spectrum in Figure 5b was showed that change of functional groups on cellulose after was be through acetylation process into cellulose acetate. Differences of functional groups between cellulose and

cellulose acetate was the establishment of C=O group (carbonyl groups) that was resulted from acetylation process with using anhydride acetic acid. Moreover, reduction in the quantity of OH groups in cellulose acetate was caused the OH group substituted by acetyl group. The emergence of acetyl group was verified with the new absorption peaks suvh as carbonyl goups and C-O acetyl groups. The FTIR spectra was demonstrated that the acetylation process oil palm empty fruit bunches cellulose into cellulose acetate has been successfully carried out.

### *3.4 Morphological Characterization by SEM*

The Result of SEM Characterization with 2000x magnification was shown in Figure 6.

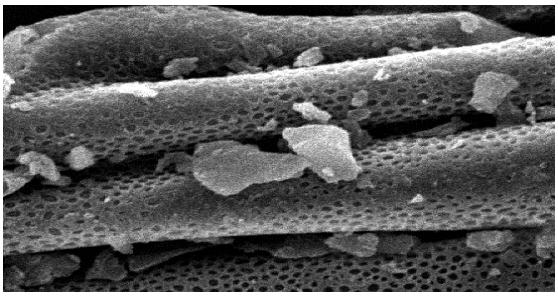


Figure 6. SEM Analysis of Cellulose Acetate with magnification 2000x

SEM image was seen morphology of micron-sized fibers. Fiber morphology was showed fiber that was shaped cylindrical fibers and had perforated layer. Palm empty fruit bunches cellulose (figure 1b) was showed good fiber of the same size, while cellulose acetate (figure 6) was showed different surface with cellulose, the fiber changes with the perforated layer. That was caused effect acetylation process that have dissolved cellulose in anhydride acetic so that shaped micron-sized fibers. Morphology of the resulting fiber had differences with cellulose acetate that was resulted by Harahap *et al* (2012) had shaped blank space fiber because the effect of time acetylation was shorter so that acetyl content was different.

## 4. Conclusion

Conclusions in the research was

1. Oil palm empty fruit bunches cellulose had water content of 4.8% and α -cellulose content of 94.8%. Low water content influence acetylation process in production of cellulose acetate because –OH group in water more reacts with anhydrous reagent than –OH group in cellulose.
2. Water content of cellulose acetate range from 4.332 to 4.468%. and highest yield of cellulose acetate by time acetylation for 45 minutes at 189.699%. This was showed yield directly proportional with water content because the higher of water content, yield was increased also
3. The best acetyl content of cellulose acetate by acetylation time for 30 min at 40.108%.
4. The changes functional groups in cellulose after acetylation process into cellulose acetate. The differences was seen with the formation of C = O group at 1759 $cm^{-1}$ and C-O group at 1234 $cm^{-1}$ that was carbonyl groups formed by acetylation process.
5. Morphology of cellulose acetate was shaped cylindrical fibers and had perforated layer.

Based on the research conclusion, as empirical implication, it is suggested that:

1. Cellulose acetate from oil palm empty fruit bunches can be developed into the industry and compete with the cellulose acetate from wood affects the environment ecosystem.
2. Cellulose acetate can be used as raw material for the production of some products such as bioplastics, biomembran, fabric and biofilms.

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