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*by* Ropalia Ropalia

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## Characterization of cellulose acetate functional groups synthesized from corn husk (*Zea mays*)

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**Abstract.** The use of masks is very important to reduce transmission of the COVID 19 virus. Therefore, an innovation is needed from mask materials is that are environmentally friendly, have good filtration quality and have anti-virus agents. An alternative way to provide masks with good filterability using a raw material of cellulose acetate. Cellulose acetate has fibrils that are bonded together so that it can form dense fibers. Fiber is a semipermeable layer that functions as a particle filtration. Therefore, this study aims to get cellulose from corn husks via delignification. The research method consisted of extracting cellulose from corn husks and further synthesizing cellulose acetate. FTIR results showed an absorption peak at wave numbers 3349  $\text{cm}^{-1}$ , 1728  $\text{cm}^{-1}$ , 1252  $\text{cm}^{-1}$ , and 1031  $\text{cm}^{-1}$ . These peaks indicated the presence functional groups of OH, C=O, aryl ether, and C-O. This functional group indicates a cellulose acetate compound.

### 1. Introduction

Currently, the use of masks is part of a comprehensive series of prevention and control measures that can limit the spread of respiratory viral diseases, including (COVID-19). Masks can be used to protect healthy people (wear yourself when in contact with an infected person). In addition, masks also function as source control (worn by infected people to prevent further transmission) [1]. The type of mask that is recommended is a mask consisting of 3 layers. The inner layer consists of an absorbent material such as cotton (fiber), the middle layer consists of polypropylene, and the outer layer of a non-absorbent material, such as a polyester blend [2,3]. However, the disadvantage of masks containing polypropylene and polyester is that they are difficult to degrade in nature, so they are not environmentally friendly.

According to Oceans Asia Environmental Group data, by 2020 it is estimated that 52 billion masks will be produced globally. The masks produced are disposable masks so that more than 1.5 billion waste masks enter the world's oceans. Therefore, an effort is needed to replace disposable masks with masks that are more environmentally friendly but have good quality filtering properties. As an alternative way to provide masks with good filtering ability by using raw materials from cellulose.

Cellulose is a source of biopolymer which is quite abundant on earth. Cellulose has fibrils that are bonded to each other so that they can form dense fibers [4]. Fiber is a semipermeable layer that functions as particle filtration [5]. One of the biomass wastes that has a high enough cellulose content is corn husk [6]. so in this study, cellulose extraction from corn husk waste will be carried out and how the delignification process is carried out so that good cellulose is obtained to be used as fiber in masks

### 2. Experimental

#### 2.1. Extraction cellulose from corn husk

Corn husk cellulose extraction consists of several stages, namely sample preparation and cellulose extraction. At the preparation stage, the corn husk sample was cleaned of impurities by washing. The results of the washing are then dried in the sun. The dried corn husks were then ground to a size of 20 mesh. Then in the alkaline step of cellulose extraction, the dry corn husk powder was heated in 1 M



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NaOH solution at 80 °C for 4 hours. Then the residue is filtered and washed with excess distilled water. Then the bleaching process was carried out, where the residue that had been washed with excess distilled water was then added with a solution of NaOH, glacial CH<sub>3</sub>COOH, N<sub>2</sub>O<sub>2</sub>, and heated in a water bath at a temperature of (80-90 °C) for 4 hours. After that, it was filtered and the residue was washed with excess distilled water. Then the residue was heated at 50 °C for 10 hours. Cellulose from the extraction process was characterized by FTIR to determine its functional groups.

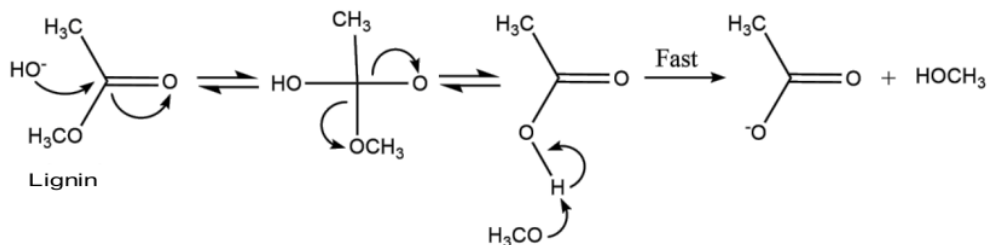
### 2.2. Synthesis of cellulose acetate

Cellulose was reacted with 25 mL of glacial acetic acid for 1 hour at room temperature with continuous stirring to obtain activated cellulose. In a separate container, 15 mL of acetic acid anhydride and 3 drops of concentrated sulfuric acid were prepared, the temperature was maintained at 0 °C. Then the second mixture was added to the first mixture and stirred for 3 hours at 40 °C. The reaction was then continued for 20 h at room temperature. Then 25 mL of 60% glacial acetic acid was added dropwise. Then filtered with a vacuum filter. The filtrate obtained was dripped with a number of DM water to form a white polymer precipitate. The white precipitate was filtered and neutralized with aqua dm. The cellulose acetate obtained was dried in a vacuum oven at 40 °C. Cellulose acetate from that process was characterized by FTIR to determine its functional groups.

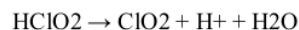
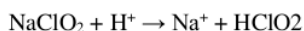
## 3. Result and Discussion

The process of preparing cellulose from corn husks was carried out in several stages, namely washing, drying, refining and sifting. Corn husks that have been cleaned, then dried to remove the moisture content in them. The purpose of reducing the moisture content is to avoid spoilage. After that, it was refined and sifted with an 80 mesh sieve. Sieving aims to produce a fine powder size with a large surface area so that the contact of the solvent molecules with the active compounds contained in the sample is faster. After that, the corn husk cellulose extraction process was carried out in several stages, namely treatment with alkali and bleaching. Treatment with alkali (such as NaOH solution) aims to reduce lignin. With the reduction of this lignin, the bond between hemicellulose and cellulose will be broken. So that hemicellulose will dissolve in NaOH solution and separate from cellulose deposits [7,8].

Hemicellulose must be removed, because hemicellulose can reduce the mechanical properties of the bioplastic to be synthesized. In addition, hemicellulose will also reduce optical properties (transparency) because of the weak properties of hemicellulose [9,10]. The following chain bond breaking reaction that occurs between lignin and cellulose using alkali (NaOH) can be seen in the following reaction equation

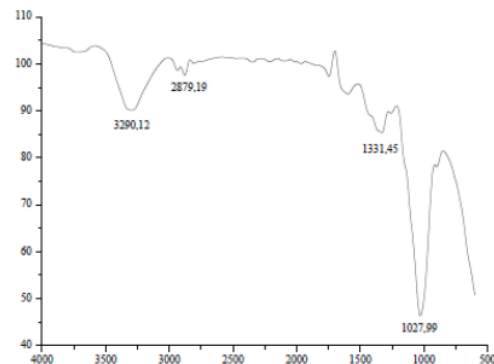


The purification process aims to increase the purity of the cellulose. This purification was carried out using NaClO<sub>2</sub> and CH<sub>3</sub>COOH. The bleaching process is carried out by the following mechanism:



When the pH value is low, the chlorite ion changes to chloric acid (HClO<sub>2</sub>). If the pH of the solution is added to acetic acid, it will form ClO<sub>2</sub> which is a corrosive compound. This compound is responsible for the bleaching of cellulose [9]. The cellulose yield obtained was 20%.

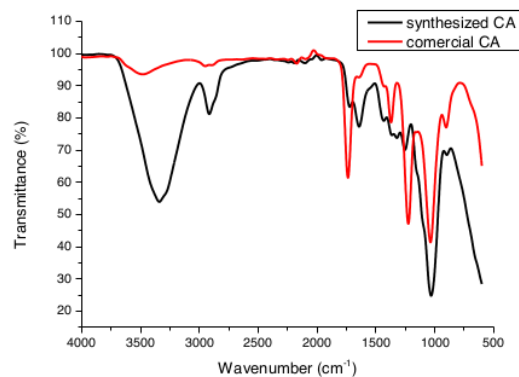
The extracted cellulose was carried out by FTIR test to determine the functional group. The results of FTIR characterization can be seen in the Figure 1.



**Figure 1.** FTIR of cellulose

From the results of the FTIR analysis, there is an absorption peak at a wave number of  $3290\text{ cm}^{-1}$ . This absorption indicates the stretching vibration of the OH functional group originating from the cellulose and possibly from the water molecules adsorbed on the cellulose. Absorption at a wave number  $2879\text{ cm}^{-1}$  is a stretching vibration from C-H. The presence of absorption at a wave number of  $1331\text{ cm}^{-1}$  indicates the presence of a C-H group (alkane). The C-O-C (polysaccharide) functional group was indicated by the absorption at a wave number of  $1027\text{ cm}^{-1}$ . In the FTIR spectrum of cellulose above, there is no absorption peak at the wave number  $1527\text{--}1535\text{ cm}^{-1}$ . The wavelength in this range indicates the presence of a C=C group on the aromatic ring of lignin (Wiradipta, 2017). Thus, this indicates that the extracted cellulose is free from lignin and hemicellulose. The results of the characterization above are in accordance with the results of research conducted by Abdel-Halim [11] regarding the cellulose extracted from sugarcane bagasse.

The extracted cellulose will be synthesized into cellulose acetate (CA). The synthesized cellulose acetate was characterized by FTIR to see its functional groups. The FTIR of commercial cellulose acetate was used as a comparison.



**Figure 2.** FTIR cellulose acetate

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**Table 1.** FTIR functional group analysis of corn husk cellulose acetate and commercial cellulose acetate

Wavenumber (cm <sup>-1</sup> )		Functional group	Range [12]
Corn husk CA	Commercial CA		
3349	3479	-OH	3600-3200
1728	1737	C=O	1760-1690
1365	1371	C-H	1480-1350
1252	1224	Aryl ether	1320-1210
1031	1036	C-O	1300-1000

Based on the FTIR results in the figure 2, it shows that the absorption pattern between commercial cellulose acetate and synthetic cellulose acetate is not much different. the absorption of the OH group in corn husk cellulose is wider and sharper than commercial cellulose acetate. This is due to the presence of the hydroxyl group of cellulose which is not substituted by the acetyl group. This is presumably due to the presence of water content in the material resulting from the less than perfect drying process. The characteristics of the absorption band for cellulose acetate are strain vibrations of the C=O group, C-O ester of the acetyl group, C-H, and aryl ether.

#### 4. Conclusion

Based on the results of the study, it was concluded that cellulose acetate had been successfully synthesized from corn husk waste. This is indicated by the appearance of IR absorption at a wave number of 3349 cm<sup>-1</sup>, 1728 cm<sup>-1</sup>, 1252 cm<sup>-1</sup>, and 1031 cm<sup>-1</sup>. These peaks indicated the presence functional groups of OH, C=O, aryl ether, and C-O.

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