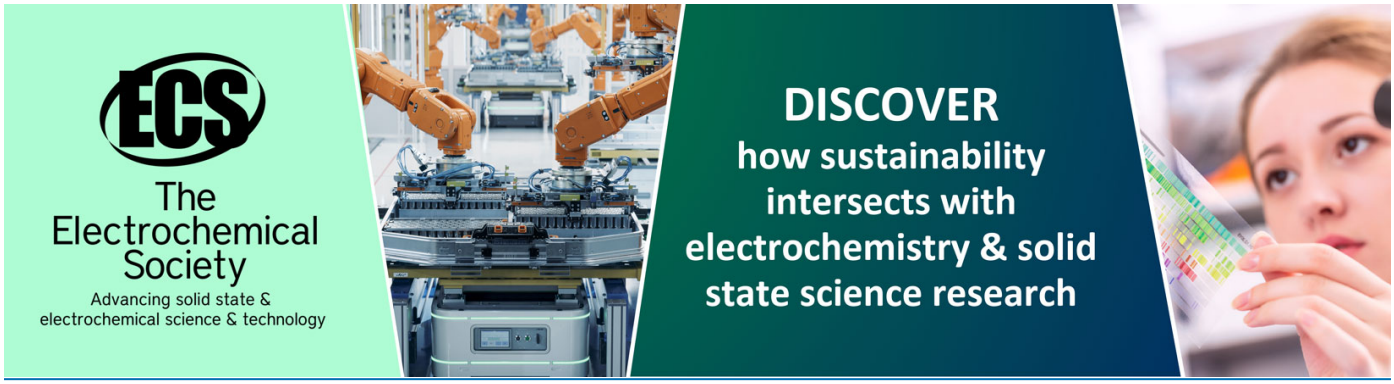
Preparation and characterization of cellulose acetate from cotton



Preparation and characterization of cellulose acetate from cotton

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Abstract. The preparation and characterization of cellulose acetate from cotton was conducted in this study. Cotton was hydrolyzed using various concentrations of sulfuric acid (10, 20, 30 and 40%). The bleaching process was performed by using H_2O_2 solution. The acetylation process was conducted using acetic acid solution. The obtained cellulose acetate was then characterized using Fourier Transform Infrared Spectroscopy (FTIR) in order to observe the functional groups, Scanning Electron Microscopy (SEM) to examine the surface morphology and X-Ray Diffraction (XRD) to observe the crystallinity of cellulose acetate. The results of FTIR analysis confirmed the formation of cellulose acetate. SEM images showed the irregular sizes of cellulose acetate. XRD patterns exhibited that the acetylation process increased the crystallinity of cellulose.

1. Introduction

Cellulose acetate is one of the important additives in the fields of pharmaceuticals, food, cosmetics and other industries [1]. The utilization of cellulose acetate as a filler is in great demand by researchers in order to increase the performance of *drug delivery system* (DDS) applications. As an additive in DDS applications, cellulose acetate can improve the performance of DDS. However, commercial cellulose acetate is expensive, meaning DDS applications have high production costs. Therefore, in this work we prepared cellulose acetate from cotton that easily found, inexpensive and environmentally friendly.

The main content of cotton is cellulose that can be extracted through hydrolysis process using acid solutions. The different concentrations of acid will produce different crystallinity of cellulose. Therefore, in order to obtain the high crystallinity of cellulose, the cotton was hydrolyzed with various concentration of H_2SO_4 (10, 20, 30 and 40%).

In order to produce cellulose acetate, the extracted cellulose was reacted with acetic anhydride (AcOH). The acetylation of cellulose particles was conducted using the slurry method where the cellulose particles were dispersed using distilled water as a dispersing agent and sodium hydroxide was used as a catalyst at room temperature [2-4]. The obtained cellulose acetate was characterized by Fourier Transform Infrared Spectroscopy (FTIR) in order to confirm the formation of cellulose

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acetate, Scanning Electron Microscopy (SEM) to study its morphology and X-Ray Diffraction (XRD) to determine its structure.

2. Materials and Methods

2.1. Materials

The cotton was obtained from a local area of Aceh, Indonesia. The collected cotton was washed and dried at 70°C for 24 h. Before hydrolysis process, the cotton was bleached according to procedure reported by Rashid *et al* [5]. All chemicals were analytical grade and were used as received.

2.2. Hydrolysis of cotton

Hydrolysis of cotton was conducted using various concentration of sulphuric acid (10, 20, 30 and 40% v/v). This process was performed in 500 ml erlenmeyer flask containing one gram bleached cotton and sulphuric acid solution. The mixture was stirred continuously at 250 rpm for 90 min. Cold distilled water was added to the mixture (to stop the hydrolysis process). The obtaining residues were washed repeatedly with distilled water untill neutral pH was reached (pH of 6-7) and then dried at 50ºC for 24 h.

2.3. Acetylation of cellulose obtained from cotton

The acetylation process was performed using acetic acid, where two grams of cellulose was added to Erlenmeyer flask containing 70 mL distilled water and then stirred to produce slurry. NaOH (1 M, 1 mL) was added to the slurry (pH 8). Acetic acid (4.8 mL) was added to the mixture and stirred continuously for 1 h at 40°C. After that, the pH was adjusted to initial pH (pH 8) at room temperature. After 30 min (acetylation reaction), the reaction was stopped by adding HCl (pH 5.5). Finally, the acetylated cellulose was washed repeatedly with distilled water until neutral pH was reached and then dried at 50ºC [3].

2.4. Characterization

The products were characterized using Cary 630 Fourier transform infrared spectroscopy (Agilent Technologies) at wavenumber of 4000-600 cm^{-1} to examine the functional group, Scanning Electron Microscopy (SEM) using JSM-6510A/JSM6510LA (Analytical/Analytical low vacuum SEM) in magnification of 100x and 500x for the morphology studyand X-Ray Diffractometry (XRD) using Shimadzu XRD-700 Series X-Ray to obtain the diffractogram pattern.

3. Results and Discussion

3.1. X-Ray Diffractometry (XRD)

XRD patterns of cellulose extracted from cotton with various concentrations of H_2SO_4 in hydrolysis process are shown in Figure 1. XRD patterns of hydrolyzed cotton with different concentrations of sulfuric acid exhibit typical peaks of cellulose at $2\theta = 15$ and 22º. The XRD pattern of cotton (H_2SO_4, 0%) is also similar with cellulose because the main content of cotton is cellulose. However, it shows a broader peak and lower intensity than the diffraction patterns of the hydrolyzed cotton. H_2SO_4 concentration in hydrolysis process influenced the crystallinity of cellulose. The highest crystallinity was obtained at 30% of H_2SO_4 concentration, where the 2θ was 15.38 and 21.86º.

The obtained cellulose with the highest crystallinity was then modified through acetylation process in order to obtain cellulose acetate. The comparison of XRD pattern of cellulose and cellulose acetate is shown in Figure 2. Figure 2 shows the XRD pattern of cellulose acetate is not significantly different from XRD pattern of cellulose extracted from cotton. XRD pattern of cellulose acetate exhibits 2 peaks at $2\theta = 15.70$ and 22.44º which are also typical peaks for cellulose. However, the peak intensity of cellulose acetate was relatively higher than cellulose. It probably influenced by the addition of the acetyl groups to the main polymer chain of cellulose.

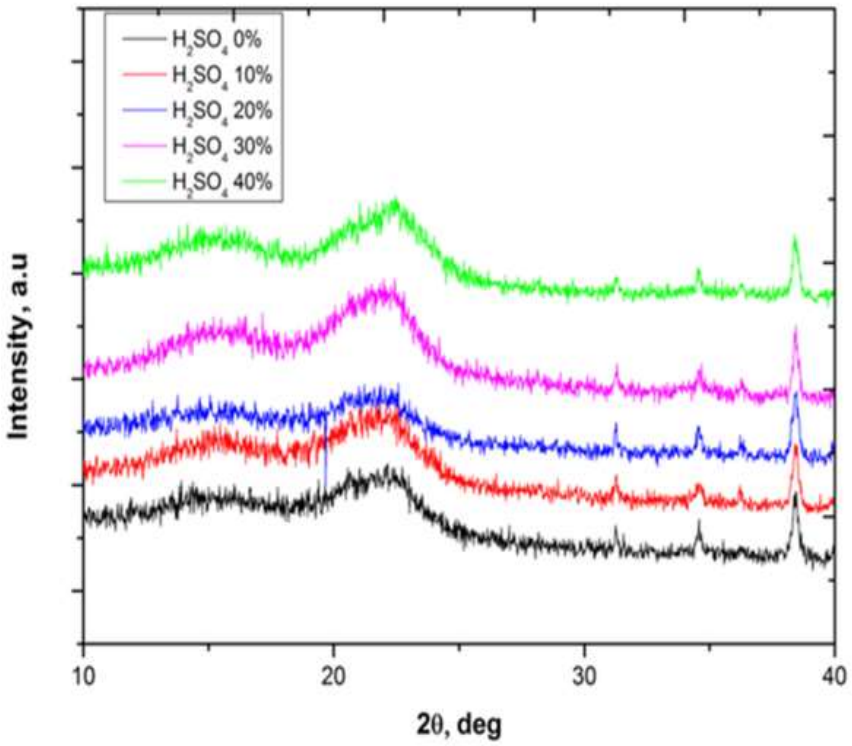

Figure 1. The XRD patterns of hydrolized cotton using various concentrations of H_2SO_4 (0, 10, 20, 30 and 40% v/v)

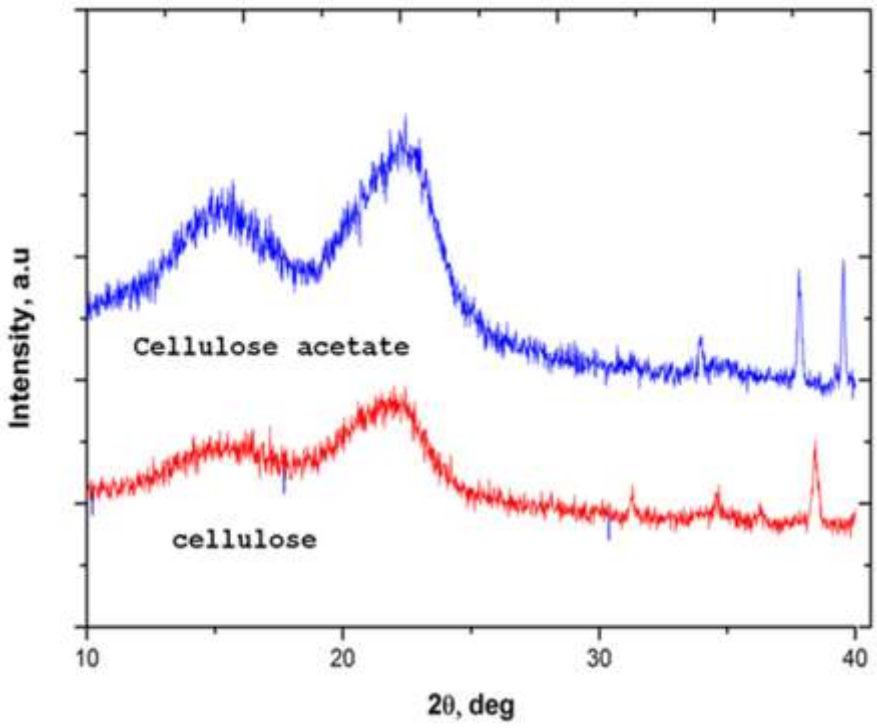

Figure 2. The XRD patterns of cellulose and cellulose acetate

3.2. Fourier transform infrared spectroscopy (FTIR)

The FTIR spectra of cellulose and cellulose acetate are shown in Figure 3. Absorption bands of cellulose extracted from cotton show typical absorption bands of cellulose. Absorption band at wavenumber 3388.93 cm^{-1} attributed to OH vibration [6-10]. Absorption band around 1600 cm^{-1} represents a strong bond between H_2O and cellulose [2].

After acetylation process, a broader absorption band of OH vibration was observed and it shifted to lower wavenumber (3376.4393 cm^{-1}). Moreover, the increase in intensities were also observed at wavenumbers 1737.86 and 2902.87 cm^{-1} which corresponded with C=O and CH vibrations, respectively [11]. These results confirmed the acetylation process of cellulose was successfully performed.

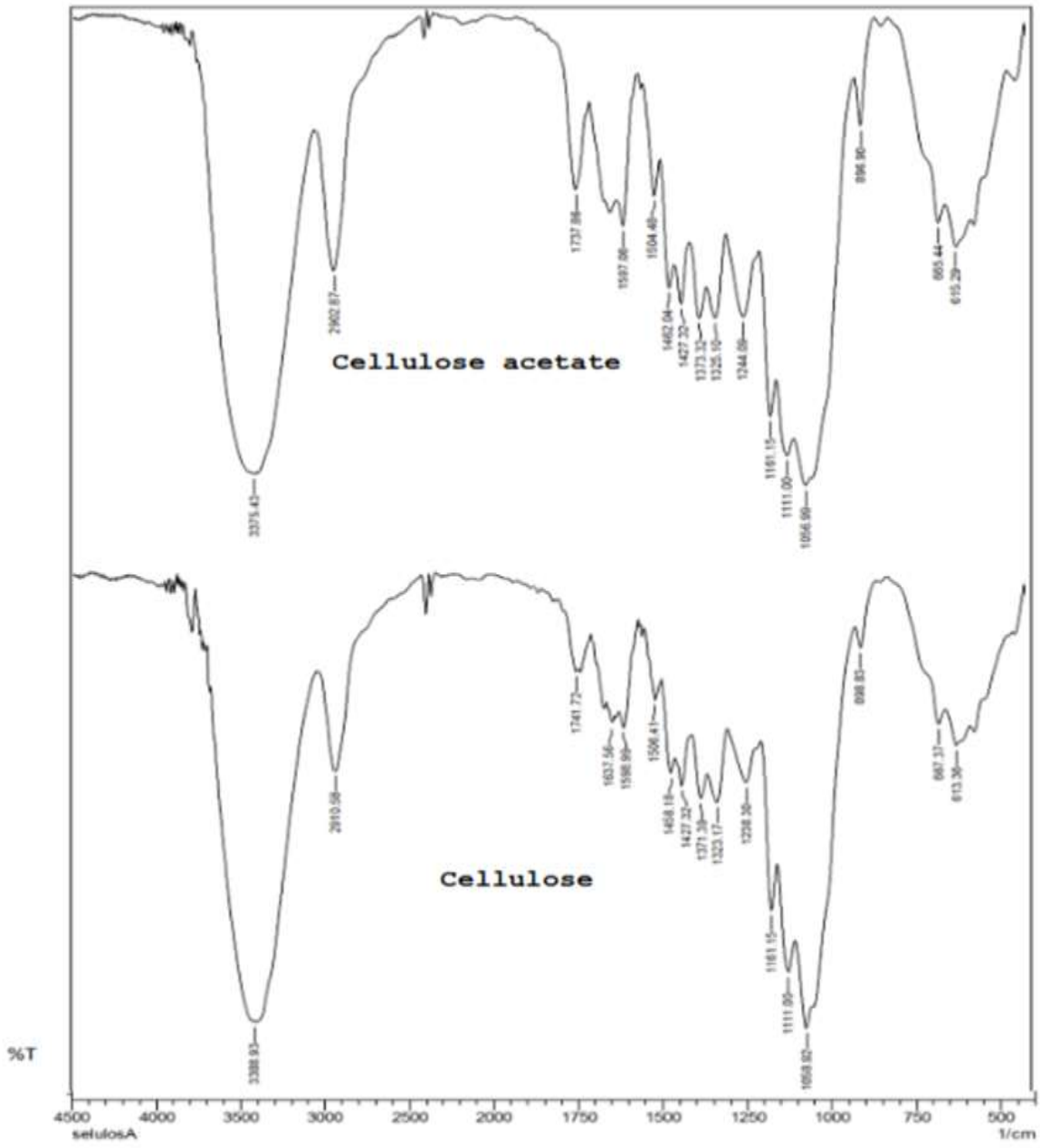

Figure 3. FTIR spectra of cellulose and cellulose acetate

3.3. Scanning Electron Microscopy (SEM)

The morphology of cellulose and cellulose acetate were analyzed using SEM. Figure 1 shows SEM images of cellulose and cellulose acetate prepared from cotton with different magnifications (100× and 500×).

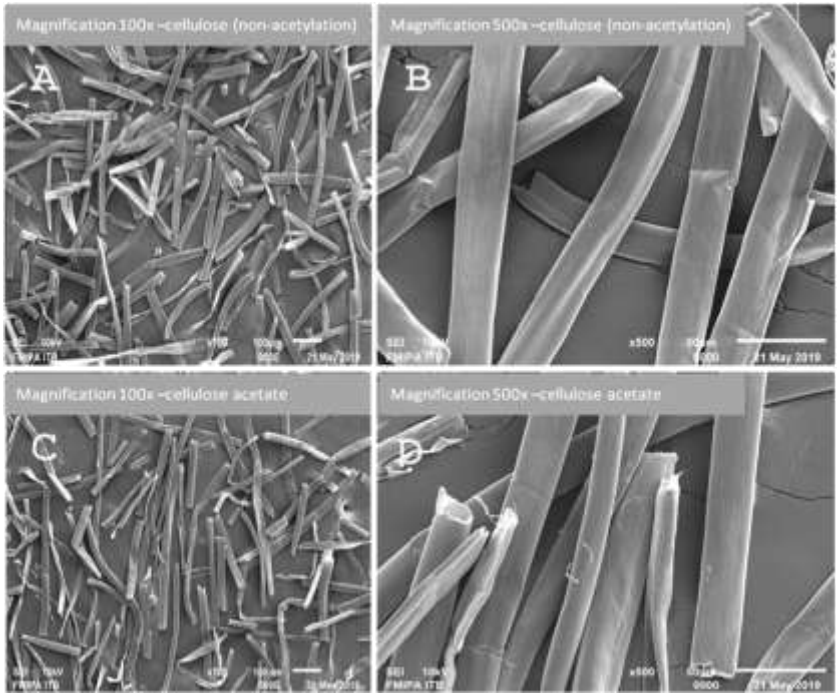

Figure 4. SEM images of cellulose (A, B) and cellulose acetate (C, D)

The SEM image of cellulose extracted from cotton shows elongated thread like structure which was the initial structure of cotton. The structure of cotton was almost retained after hydrolysis (cellulose) or after acetylation processes (cellulose acetate). It was probably due to strong hydrogen bonding between hydroxyl groups contained in cellulose. Similar result was also found by Rashid *et al* [5]. This result also agreed with the XRD analysis, where the XRD pattern of cellulose and cellulose acetate was not significantly different.

4. Conclusion

The preparation and characterization of cellulose acetate from cotton has been successfully performed. The highest crystallinity of cellulose was obtained through hydrolysis process of cotton using H_2SO_4 30%. FTIR spectra of cellulose and cellulose acetate showed some differences that confirmed the formation of cellulose acetate. XRD patterns and SEM images of cellulose and cellulose acetate were only slightly different due to strong hydrogen bonding of hydroxyl groups.

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