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Synthesis and Characterization of Cellulose Acetate Derived from Corn Stalk Cellulose Using FTIR Analysis

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ABSTRACT

The synthesis and characterization of cellulose acetate were carried out using cellulose derived from corn stalks. Cellulose acetate was obtained through the esterification of cellulose, a natural biopolymer commonly sourced from wood or plant fibers. During the acetylation stage, the hydroxyl groups in cellulose were replaced with acetate groups from acetic anhydride. The acetylation process involved varying the volume of glacial acetic acid (CH_3COOH) at 25, 35, 45, 55, and 65 milliliters and reaction times of 1, 1.25, 1.5, 1.75, and 2 hours, using 0.5 milliliters of 98 percent sulfuric acid as a catalyst. Cellulose was first extracted by delignification using 17.5 percent sodium hydroxide, followed by bleaching with 4 percent sodium hydroxide and 4 percent hydrogen peroxide. The results showed that increasing the volume of acetic acid led to an acetyl content of up to 40.4 percent and a degree of substitution of 2.5. Extension of the acetylation time to 1.5 hours also increased the yield of cellulose acetate to a maximum of 70.2631 percent before decreasing at longer durations. These optimal results, including 70.2631 percent yield, 40.4 percent acetyl content, and a degree of substitution of 2.5, were achieved with 65 milliliters of acetic acid and 1.5 hours of acetylation. FTIR analysis confirmed successful acetylation by identifying hydroxyl (O–H), carbonyl (C=O), and ester (C–O) functional groups.

INTRODUCTION

Cellulose acetate is a polymer produced through the esterification of cellulose with acetic anhydride (Lou, 2022). It is widely used in textiles, cigarette filters, plastics, films, paper coatings, and membranes due to its favorable physical and optical properties. Its solubility in acetone, thermoplastic behavior, and biodegradability make it both industrially valuable and environmentally friendly (Faizal, 2022). Cellulose acetate membranes have been widely studied for their applications in pervaporation, particularly for the separation of water-ethanol mixtures, showing good separation performance and potential for industrial use (Zhu, 2001). Based on the number of acetate groups attached, cellulose acetate is classified into monoacetate, diacetate, and triacetate (Lou, 2022). Physically, cellulose acetate appears as a white solid with a density of 1.52 g/cm³ and a degree of substitution ranging from 0.6 to 3.5 (James, 1999).

Recent research trends in cellulose acetate synthesis from agricultural waste show a growing emphasis on sustainability and the use of various agricultural residues. Previous studies have focused on rice husk ash, which yielded a high degree of substitution of 1.01 with a yield of

120% under specific conditions (Ma'ruf, 2023). Research on sugarcane bagasse has achieved a yield of 79.31%, with an acetyl content of 36.19% and a degree of substitution of 2.11 (Anjawarti, 2023). Meanwhile, palm fiber has been shown to produce a yield of 49%, an acetyl content of 39.97%, and a degree of substitution of 2.5 (Utami, 2021). In more recent studies, Thongsomboon et al. (2023) compared sugarcane bagasse and rice straw, identifying their potential as valuable cellulose sources for sustainable material development. Bamba et al. (2022) focused on cocoa pod husk, achieving significant characterization of cellulose triacetate. Additionally, Plakantonaki et al. (2024) explored upcycling peach waste, showing promising results for dissolving pulp production. In Indonesia, there is increasing attention on utilizing rice straw, coconut husks, and corn residues as cost-effective, locally available biomass. Furthermore, research is shifting towards eco-friendly methods, such as ultrasound-assisted acetylation, that reduce environmental impact, reflecting a broader trend toward sustainable and efficient cellulose acetate production.

Corn is an annual crop with a growing cycle of 80 to 150 days. Major production centers include Central Java, East Java, and Madura (Ikhtiarini et al., 2022). In 2022, East Java produced 4.952 million tons of corn from 817,449 hectares of farmland, with an average productivity of 57.41 quintals per hectare. Up to 95 percent of the corn plant becomes waste, yet only a small portion is utilized. Agricultural residues, such as stalks and husks, contain approximately 44 percent cellulose (Setyaningsih, 2020). Corn stalks, which are often burned after harvest, hold potential as raw material for cellulose production. The cellulose content in corn stalks ranges from 35 to 50 percent of dry weight (Afriyanti et al., 2020).

The synthesis process begins with cellulose extraction from dried corn stalks through delignification using 17.5% sodium hydroxide. The optimal temperature for this step is approximately 100.38 °C to ensure effective lignin removal without damaging the cellulose (Sartika & Firmansyah, 2022). This is followed by a bleaching stage using 4% sodium hydroxide and 4% hydrogen peroxide to purify the cellulose (Susi et al., 2023). This step removes pigments and enhances whiteness (Permadani & Silvia, 2022). It is designed to operate at 90 °C or higher for optimal hydrogen peroxide performance, but must remain below 130 °C to prevent the cellulose from dissolving in the solution (Bajpai, 2016). Acetylation is then carried out using acetic anhydride and glacial acetic acid with a sulfuric acid catalyst to substitute hydroxyl groups with acetyl groups (Nurhayati, 2019).

Factors that affect acetylation include temperature, reaction time, stirring speed, and solvent volume. The optimal temperature for acetylation is 50°C, yielding 79.31% with an acetyl content of 36.19% (Anjarwati, 2023). Prolonged reaction time can reduce acetyl content, with the highest yield achieved after three hours and the highest acetyl content at one hour (Rahmatullah, 2020). Stirring at 350 rpm results in 39.2% acetyl content, though higher speeds may reduce acetyl content (Siswati & Wachidah, 2021). Increasing the volume of glacial acetic acid improves yield, with optimal results at 100 milliliters (Anjarwati, 2023). Studies on lignin biopolymers confirm that these factors, such as temperature and solvent volume, significantly influence acetylation (Hosseini et al., 2020). Additionally, optimizing stirring speed and reaction time is critical for improving acetylation efficiency (Zhang et al., 2021).

This research aims to examine the effects of acetic anhydride volume and acetylation time on the characteristics of cellulose acetate derived from corn stalks, focusing on yield, acetyl content, degree of substitution, and FTIR analysis.

RESEARCH METHODS

In this study, the main materials used were corn stalks obtained from Tuban, East Java, and acetic acid (CH_3COOH), which was purchased in Surabaya, East Java. Supporting chemicals used in the study included sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), distilled water (aquadest), sulfuric acid (H_2SO_4), hydrochloric acid (HCl), phenolphthalein, and ethanol (C_2H_5OH). The first stage was the preparation of corn stalk powder. The collected corn stalks were sorted to obtain good-quality material. The sorted stalks were then crushed or size-reduced to

facilitate further processing. After that, they were washed to remove impurities and soaked in water or distilled water for 24 hours. The soaked corn stalks were dried until a constant weight was achieved. Once dried, the material was ground into finer particles to form a powder. The resulting corn stalk powder was sieved using a 100-mesh screen to obtain uniform particle size. The final product was corn stalk powder, which served as the raw material for cellulose synthesis. The second stage, the synthesis of cellulose from corn stalks through delignification and bleaching processes, is shown in Figure 1.

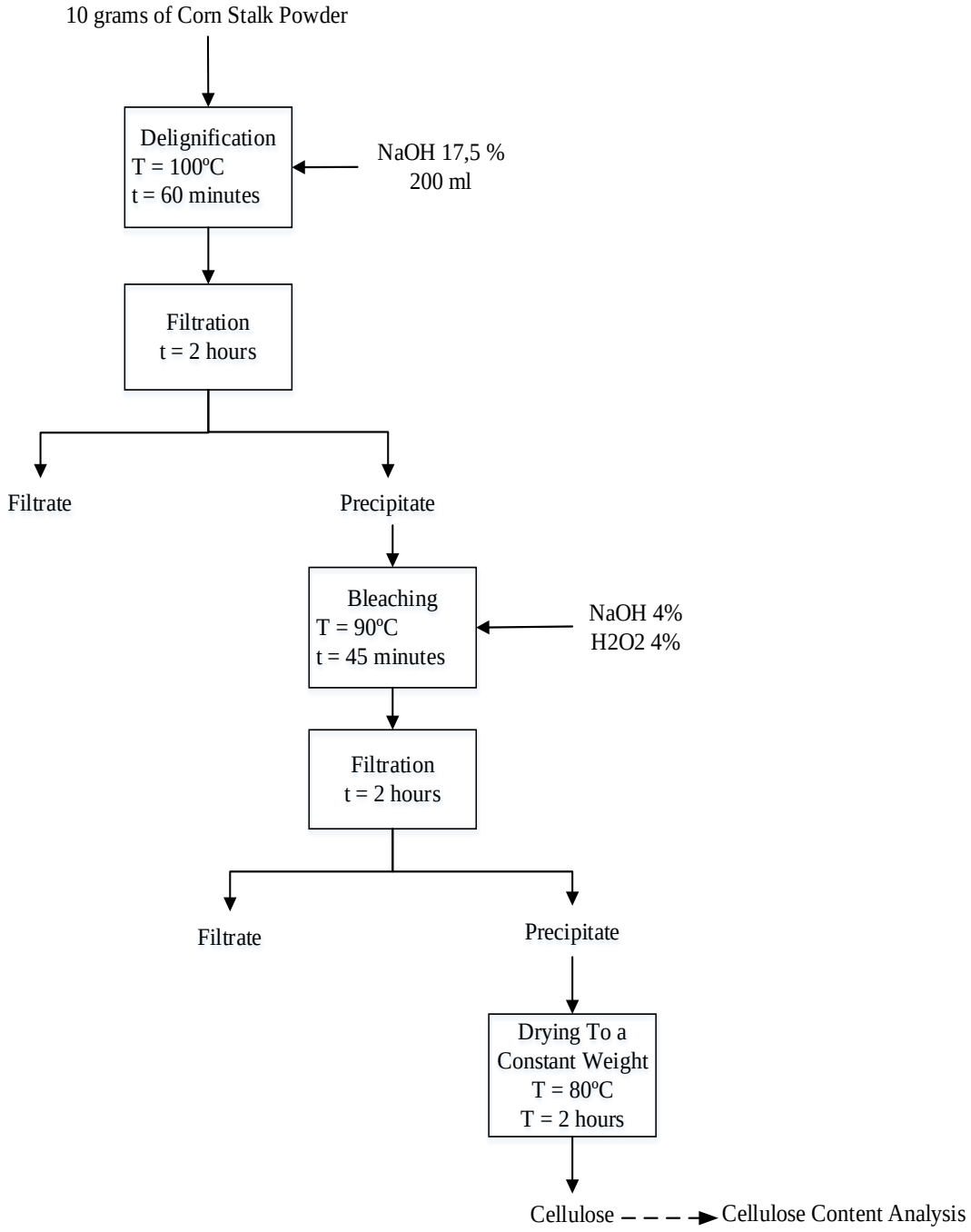

Figure 1. Synthesis Process of Cellulose from Corn Stalks

Corn stalk powder was mixed with 200 mL of 17.5% NaOH solution and heated at 100 °C for 30 minutes for delignification. The mixture was then filtered to separate the liquid from the solid. The filtrate was bleached using a solution of 4% NaOH and 4% H_2O_2 at 90 °C for 45 minutes.

After bleaching, the mixture was filtered again to separate the precipitate. The precipitate was dried at 80 °C until a constant weight was reached. The final product obtained from this process was cellulose derived from corn stalk powder. The third stage was the acetylation process, as shown in Figure 2.

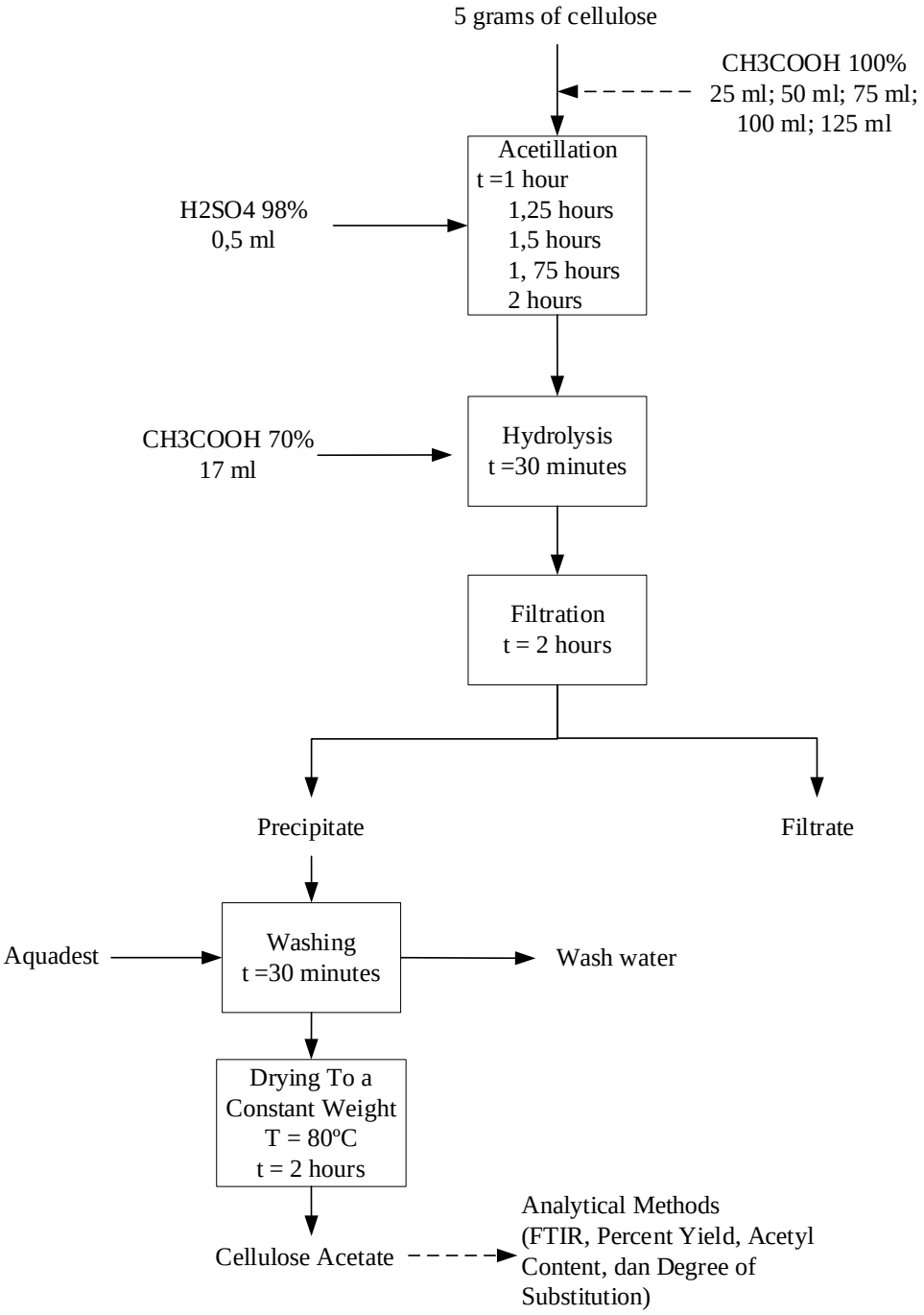

Figure 2. Acetylation Process

Five grams of cellulose were reacted with varying volumes of glacial acetic acid (CH_3COOH): 25, 35, 45, 55, and 65 mL. The acetylation was carried out for 1, 1.25, 1.5, 1.75, and 2 hours, respectively. During the reaction, 0.5 mL of 98% H_2SO_4 was added as a catalyst. After each acetylation step, the mixture was filtered to separate the precipitate from the filtrate. The filtrate was hydrolyzed using 70% acetic acid for 30 minutes, followed by another filtration. The resulting precipitate was washed with distilled water until neutral pH was reached, then filtered again. The washed precipitate was dried at 80 °C until a constant weight was achieved. The final

product was cellulose acetate. The product was then characterized for yield percentage, acetyl content, degree of substitution, and FTIR analysis.

RESULTS AND DISCUSSION

Cellulose from corn stalks was synthesized as a raw material for cellulose acetate production through two main stages: delignification to remove lignin, followed by a bleaching process. The resulting cellulose was tested for its cellulose content. The test was conducted at the Nutrition Laboratory of the Faculty of Public Health, Universitas Airlangga, yielding a cellulose content of 35.84%. (Afriyanti, 2020) which reported that the cellulose content in agricultural waste varies between 35–50% of dry weight. This cellulose content was then used to calculate the required cellulose mass for each variable.

Effect of Acetylation Time and Acetate Volume on Yield

The product yield percentage was obtained by comparing the product mass to the initial mass of the material. The initial mass was determined by weighing the sample at the start of the acetylation process and multiplying it by the previously obtained cellulose content. Meanwhile, the product mass was derived from the precipitate obtained after the second filtration in the acetylation process.

Table 1. Results of analysis of percentage yield of cellulose acetate at various acetylation times (hour) and acetic acid volumes (ml)

Time (hour)	Volume (ml)	Percent Yield
1 jam	25 ml	12,1448
	35 ml	18,4868
	45 ml	29,3198
	55 ml	38,1503
	65 ml	37,9750
1,25 jam	25 ml	25,2072
	35 ml	39,4382
	45 ml	42,2896
	55 ml	51,3627
	65 ml	55,6987
1,5 jam	25 ml	53,4249
	35 ml	65,7223
	45 ml	63,4869
	55 ml	68,5318
	65 ml	70,2631
1,75 jam	25 ml	41,7535
	35 ml	57,0996
	45 ml	55,9569
	55 ml	61,5854
	65 ml	65,1498
2 jam	25 ml	34,3163
	35 ml	45,5604
	45 ml	49,3637
	55 ml	45,7793
	65 ml	48,1567

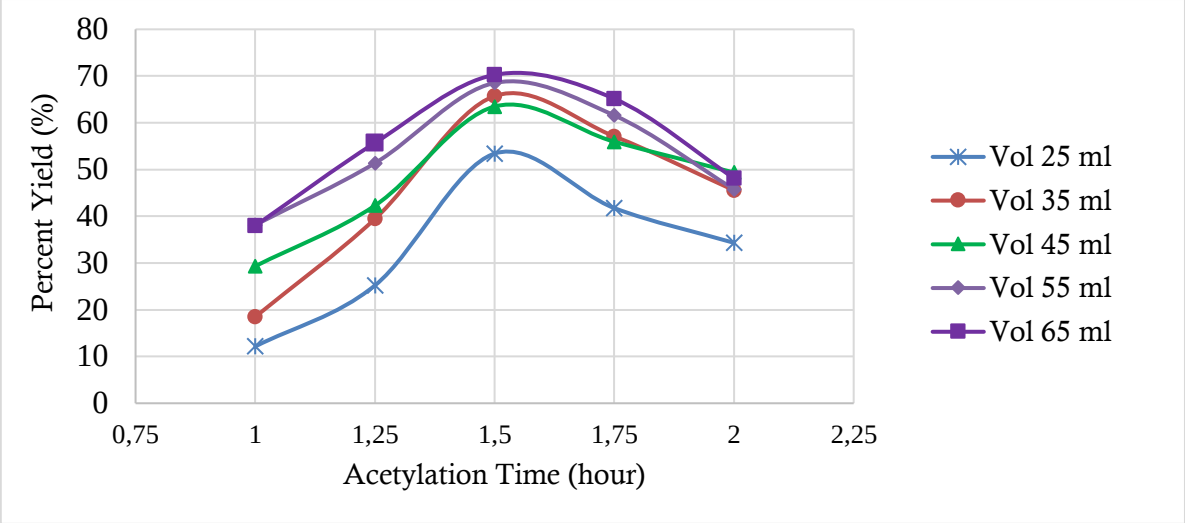

Figure 3. Relationship Between Acetylation Time, Acetic Acid Volume, and Product Yield

The effect of acetylation time on product yield is shown in Figure 1. The analysis revealed that longer acetylation times generally increased the yield. This is because a longer reaction time provides more opportunities for reactant molecules to react and form the desired product. This finding is consistent with research by (Vashti, 2024), which states that the acetylation of cellulose by glacial acetate increases over time, enhancing the acetyl-to-cellulose acetate ratio and thus increasing the product yield. At 1.5 hours, the product yield reached its optimum condition. At this point, nearly all reactive hydroxyl groups had been successfully substituted by acetyl groups, producing the highest yield. However, after this optimum point, a decline in cellulose acetate yield was observed. This is because, beyond the optimum time, structural degradation of cellulose acetate may occur, converting it into undesired byproducts. These results align with the study by (Siswati & Wachidah, 2021), which found that prolonged acetylation beyond the optimum time can degrade cellulose acetate into glucose, reducing its mass.

The effect of acetic acid volume on yield showed an increase in yield with higher volumes of glacial acetic acid. The lowest yield (12.1448%) was obtained at 25 mL, while the highest yield (70.2631%) was achieved at 65 mL. This is because a sufficient reagent volume ensures complete substitution of hydroxyl groups without reducing reaction efficiency. An optimal volume also improves solution homogeneity, increasing the likelihood of effective collisions between reactant molecules and the substrate. According to (Faizal et al., 2022), an adequate acetic acid volume enhances solution homogeneity, promoting better contact between reagent molecules and the substrate.

Effect of Acetylation Time and Acetate Volume on Acetyl Content

The acetyl content indicates the amount of acetate esterified onto the cellulose chain and plays a role in determining the degree of substitution (DS) (Utami, 2021). Acetyl content analysis was conducted to identify the type of cellulose acetate produced whether monoacetate, diacetate, or triacetate.

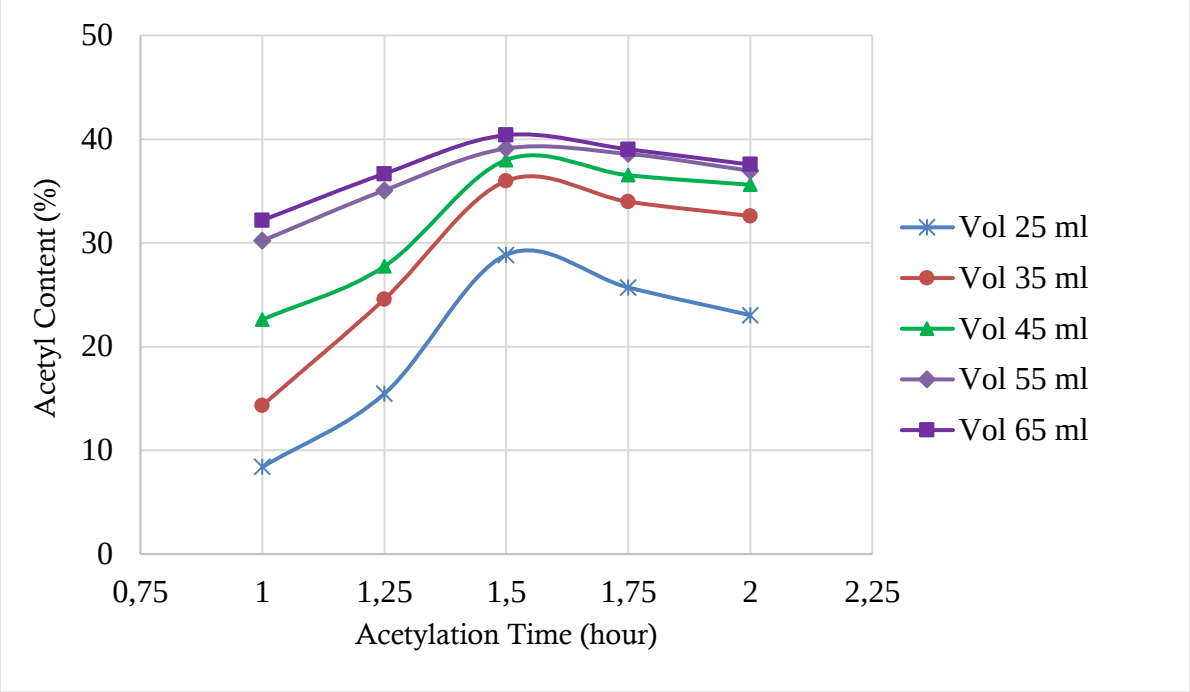

Figure 4. Relationship Between Acetylation Time, Acetic Acid Volume, and Acetyl Content

The effect of acetylation time on product yield can be seen in Figure 4. The analysis results show that the longer the acetylation time, the higher the acetyl content tends to be. This occurs because a longer reaction time provides more opportunities for reactant molecules to react and form cellulose acetate. According to (Anjarwati, 2023), the initial stage of acetylation is characterized by a high rate of acetyl group formation due to the abundance of hydroxyl groups. Within the range of 1.25 to 1.5 hours, the acetyl content continued to increase until reaching a maximum value of 40.3860%. This condition indicates that the reaction achieved optimal efficiency, with nearly all hydroxyl groups being converted into acetyl groups. At the 1.5-hour interval, the product yield reached its optimum condition. (Vashti et al., 2024) stated that the optimal duration allows the formation of stable cellulose acetate, placing the reaction efficiency at its peak. However, after reaching the optimum condition at 1.5 hours, a decrease in acetyl content was observed. This decline is caused by the degradation of the cellulose structure, where the formed acetyl groups begin to undergo structural damage. According to (Widodo et al., 2022), excessively long reaction times not only damage the cellulose structure but also increase the risk of byproduct formation, such as glucose.

The effect of acetic acid volume on acetyl content shows an increase in acetyl content with each addition of glacial acetate volume. The lowest acetyl content of 8.3948% was obtained using 25 mL of acetic acid. In the volume range of 35 mL to 65 mL, the acetyl content continued to rise until reaching a maximum value of 40.3860%. An excess of reagent ensures the saturation of hydroxyl groups on the substrate, allowing the esterification reaction to proceed optimally. According to (Vashti et al., 2024), sufficient acetic acid volume ensures even distribution of reagent molecules, giving each hydroxyl group on the cellulose an equal opportunity to react.

Based on the acetyl content analysis, it can be concluded that the types of cellulose produced were cellulose monoacetate and cellulose diacetate. This is indicated by an acetyl content of less than 35% for cellulose monoacetate and 35%–43% for cellulose diacetate. In an industrial context, cellulose acetate must meet the quality standards set by the Indonesian National Standard (SNI). According to SNI 06-2115:1991, the quality requirements for cellulose acetate must have an acetyl content between 39% and 40%. In this study, cellulose acetate that met the SNI standard was produced by adding 55 mL of acetic acid with a 1.5-hour acetylation time, yielding an acetyl

content of 39.0962%. Additionally, cellulose acetate produced with 65 mL of acetic acid and a 1.75-hour acetylation time had an acetyl content of 39.0189%.

Effect of Acetylation Time and Acetate Volume on Degree of Substitution

The degree of substitution (DS) value is strongly influenced by the acetyl content of cellulose acetate, where the acetyl content shows a positive correlation or direct proportionality with the degree of substitution. The degree of substitution is used to determine the number of hydroxyl (-OH) groups that have been replaced by acetyl (-OCH3CO) groups, serving as an indicator for the formation of cellulose acetate (Utami et al., 2021).

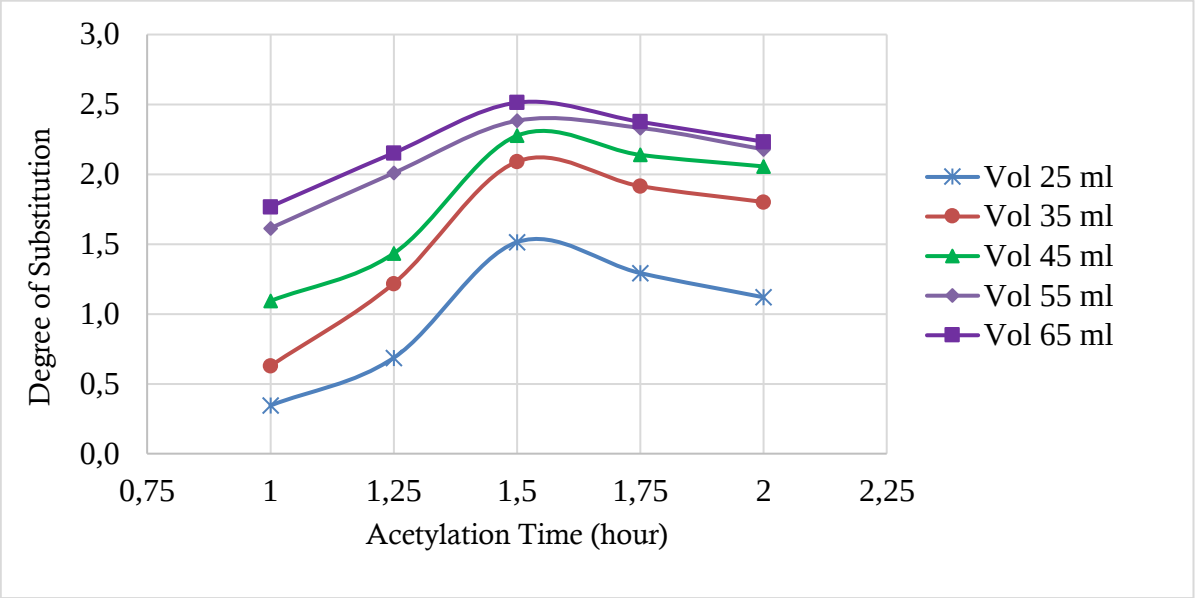

Figure 5. Relationship Between Acetylation Time, Acetic Acid Volume, and Degree of Substitution

The effect of acetylation time on product yield is shown in Figure 5. The results indicate that longer acetylation times generally increase the degree of substitution (DS), as extended durations allow more interaction between acetic acid and cellulose hydroxyl groups. This enhances the substitution process, with acetyl groups progressively replacing hydroxyl groups (Vashti, 2024). The DS continued to rise between 1.25 and 1.5 hours, reaching a peak of 2.5127. After this point, the DS decreased due to structural degradation of cellulose acetate (Utami et al., 2021). Thus, 1.5 hours is considered the optimal acetylation time for achieving maximum DS without damaging the structure.

The effect of acetic acid volume on the degree of substitution shows an increase in the degree of substitution with each addition of glacial acetate volume. At 25 mL of acetic acid, the degree of substitution produced was relatively low, ranging from 0.3 to 1.8. This is due to the insufficient amount of acetic acid to react completely with the hydroxyl groups on the cellulose. When the acetic acid volume was increased to 35 mL, the degree of substitution rose significantly. A larger volume ensures better solution homogeneity, increasing the chances of contact between reagent molecules and the substrate. Research by (Widodo et al., 2022) explains that increasing the acetic acid volume improves the diffusion of acetate molecules into the cellulose structure, making the reaction more efficient.

FTIR Analysis

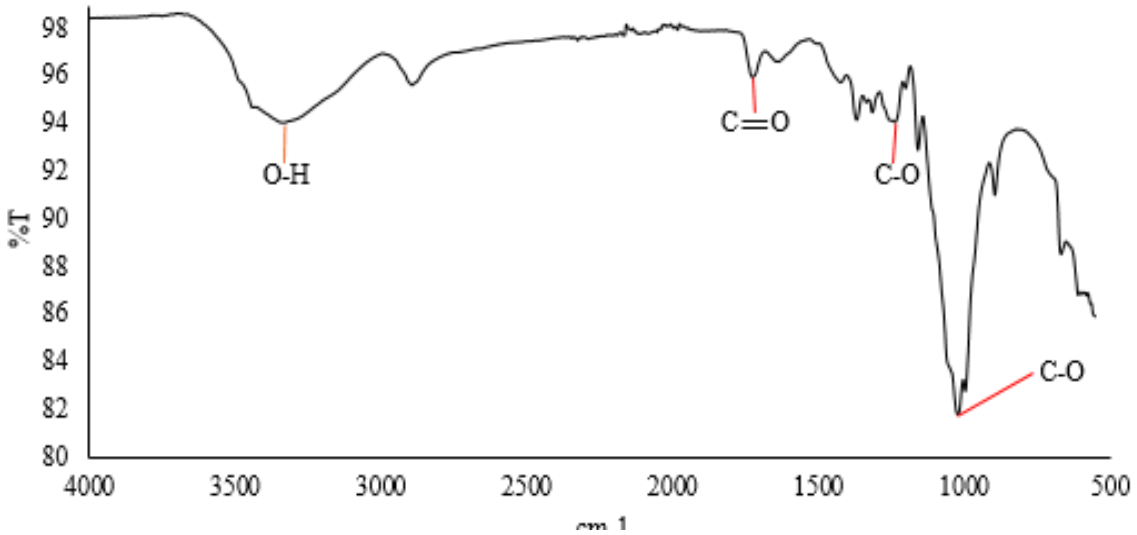

Figure 6. FTIR Spectrum of Cellulose Acetate

Analysis using the FTIR instrument was conducted to observe characteristic absorption regions and changes in functional groups after the acetylation process. This qualitative analysis aimed to determine the success of the acetylation process. The functional groups forming the chemical structure of cellulose acetate are C-O, O-H, C-H, and C=O. The results showed absorption peaks for hydroxyl groups (O-H) at a wavelength of 3333.68 cm^{-1}, carbonyl groups (C=O) at 1724.5 cm^{-1}, and ester groups (C-O) from acetyl groups at 1021.86 cm^{-1}. Based on the data obtained, the tested samples contained acetyl groups with typical absorption characteristics, namely carbonyl (C=O) and acetyl (C-O) group absorptions. This means the acetylation process in this study was successful, as evidenced by the presence of functional groups forming cellulose acetate in the tested samples.

CONCLUSION

Based on the research result, cellulose acetate with the highest yield of 70.2631% was obtained by adding 65 mL of acetic acid and an acetylation time of 1.5 hours. Meanwhile, the highest acetyl content of 40.4% and a degree of substitution of 2.5 were also achieved under the same conditions. The longer the acetylation time and the higher the volume of acetic acid used, the greater the yield, acetyl content, and degree of substitution obtained.

To improve product quality, future researchers are advised to consider the influence of other variables that may affect the characteristics of cellulose acetate. This study demonstrates that corncobs can be utilized as an eco-friendly raw material for cellulose acetate production. By converting agricultural waste into value-added products, this process not only reduces waste but also supports the availability of raw materials for various commercial applications, such as textiles, photographic films, and biodegradable plastics.

The use of corncobs also opens opportunities for sustainable production with more efficient costs and renewable resources. These findings can be further developed for industrial-scale applications, particularly in the bioplastics and textile industries, thereby contributing to greener and more sustainable manufacturing processes. Future research could also explore the economic aspects and scalability of cellulose acetate production from corncobs.

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