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Cellulose Acetate From Cassava Stalks : Effect Of Acetic Anhydride Volume And Acetylation Time

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ABSTRACT

Cassava stalks waste can be used as a material for making particleboard, crafts, briquettes, charcoal, bioethanol, and producing alpha-cellulose. Alpha-cellulose produced from cassava stems can be used as Cellulose Acetate. This study aims to obtain cellulose acetate from cassava stems and find relatively good conditions based on variations in acetylation time and volume of acetic anhydride. The process of making cellulose into cellulose acetate is carried out in several stages, namely pre-treatment, cellulose isolation, and acetylation. In the pre-treatment process, cassava stems will be reduced in size and softened to facilitate fiber extraction. Next, cellulose isolation is carried out by delignification and bleaching to obtain alpha-cellulose. In the acetylation stage, cellulose is first activated with glacial acetic acid before being acetylated with acetic anhydride. In this study, the effect of acetylation time (30, 60, 90, 120, 150 minutes) and the effect of adding volume (10, 12.5, 15, 17.5, 20 ml) on the acetyl content and degree of substitution of cellulose acetate from cassava stlaks were investigated. Based on the results of the study, the relatively good acetyl content and degree of substitution were obtained at a volume of 20 ml and a time of 150 minutes, with an acetyl content of 34.8% and a degree of substitution of 1.987. The synthesized cellulose is classified as monoacetate cellulose, which can be used in the manufacture of plastics and paints.

INTRODUCTION

Cassava is one of the food crops in Indonesia, in addition to rice and corn. Lampung Province is one of the national cassava production centers, which ranked first in producing an average harvest of 7.74 thousand tons with a harvested area of around 295.55 thousand hectares in 2012-2016. Increasing cassava production will certainly increase the resulting cassava waste. The resulting waste includes cassava peels (45%), leaves (29%), and cassava stems (29%) from a 9 m^2 plot. The utilization of cassava is mainly for food, while only 10% of the cassava stem can be used for replanting, and 90% of the cassava stem will become waste (Amien, 2021). In addition to being used for seeds through cuttings, cassava stem waste can be used as a material for making particleboard, handicrafts, briquettes, charcoal, bioethanol, absorbent media, and producing alpha-cellulose (Santy et al., 2019). Cassava stem waste contains a high amount of α-cellulose. Based on analysis, cassava stems contain 56.82% α-cellulose, 21.72% lignin, 21.45% ADF, and 0.05-0.5 cm fiber length (Sumada, 2011). Alpha-cellulose produced from cassava stems can be used as cellulose acetate.

In recent years, there has been a growing global trend toward converting biomass waste into value-added products such as bioplastics, driven by increasing environmental concerns and the urgent need for sustainable alternatives to conventional petroleum-based plastics. The accumulation of plastic waste, coupled with the depletion of fossil fuel resources, has prompted researchers and industries to explore biodegradable solutions derived from renewable sources. Among the most promising feedstocks are agricultural residues, which are abundant, low-cost, and rich in lignocellulosic content suitable for polymer synthesis (Maneking, 2020). Cassava peels, banana peels, corn cobs, coconut fiber, potato peels, sugarcane bagasse, green bean peels, and avocado seeds have been widely studied for their potential in bioplastic production due to their high cellulose and starch content (Aritonang, 2024). Utilizing these types of biomass not only offers a sustainable pathway to reduce environmental pollution but also adds economic value to agricultural waste streams, supporting circular economy principles and green technology development.

Cellulose acetate is cellulose with its hydroxyl groups replaced by acetyl groups. Cellulose acetate has many useful applications in the industrial world. Cellulose acetate is used as an adhesive for topographic films and can also be used in the textile industry, such as paint, and is used in the production of cigarette filters, biodegradable plastics, and membrane production (Fengel, 1984). Cellulose acetate is made by reacting cellulose with acetic acid through an acetylation process, which replaces the hydroxyl groups on cellulose with acetyl groups (Utami, 2021). Cellulose acetate is a promising derivative polymer of cellulose because it is easily formed, non-toxic, highly stable, hydrophilic, and obtained from renewable sources. The molecular formula of cellulose acetate is [C6H7O2(OCOCH3)3]x. Cellulose acetate is a relatively inexpensive chemical compound because it is usually made from agricultural by-products. In addition, the production technology of cellulose acetate does not require additional chemical or mechanical processes, so it does not require many chemicals. Cellulose acetate is conventionally produced by acetylating the hydroxyl groups of cellulose using acetic anhydride, acetic acid as a solvent, and sulfuric acid as a catalyst (Rahmayetty, 2023).

While several studies have explored the synthesis of cellulose acetate from various biomass sources, the optimal acetylation conditions appear to vary significantly depending on the raw material used. Utami (2021), for instance, reported that synthesizing cellulose acetate from palm oil fiber cake yielded optimal results at an acetylation time of 1.5 hours. In contrast, Darmawan (2018), using empty palm oil bunches, found the optimal acetylation time to be 2.5 hours. These variations highlight the need for biomass-specific optimization in the acetylation process. However, there remains limited research focusing on cassava stalks, particularly regarding how acetylation time and acetic anhydride volume influence the chemical properties of cellulose acetate. This gap underscores the necessity of studying the acetylation behavior of cassava stalk-derived alpha-cellulose to produce cellulose acetate.

Based on previous research, cellulose acetate has been synthesized from cassava stems by Lismeri in 2016. The research focused on the pre-treatment process, specifically the delignification process, which yielded relatively good results with a cellulose-to-sodium sulfite ratio of 1:12. The results will be used for the production of cellulose acetate. Based on previous research on the synthesis of cellulose acetate, it is known that there is an effect of time and volume of acetic anhydride on the acetylation process. Therefore, this research will focus on determining the effect of acetic anhydride volume and acetylation time on the acetyl content produced in cellulose acetate from cassava stems using the cellanase process.

RESEARCH METHODS

Prearation of Research Materials

In this research, the main raw material, cassava stems, was obtained from Cerme, Gresik. The cassava stems were peeled, cut, and reduced in size to increase their surface area. The cassava stems were then dried in an oven at 105°C for 1 hour. The cassava stems were hydrolyzed using

aquadest with a material-to-aquadest ratio of 1:6 at 100°C for 1 hour. The cassava stem fibers were separated from the hydrolyzate filtrate through filtration using filter paper. The cassava stem fibers were then dried in an oven at 105°C for 1 hour. The raw material was tested for its cellulose, hemicellulose, and lignin content

Isolation of Cellulose Process

The cassava stem fibers from the pre-treatment process underwent delignification to reduce lignin content using sodium sulfite (Na_2SO_3) with a concentration of 20% and a material-to-sodium sulfite ratio of 1:12 at 100°C for 3 hours. The cassava stem fibers were separated from the filtrate (black liquor) resulting from delignification using filter paper. The cassava stem fibers were washed with aquadest to remove any remaining filtrate and neutralize the pH. The cassava stem fibers were then dried in an oven at 105°C for 1 hour. The bleaching process was carried out using hydrogen peroxide (H_2O_2) with a concentration of 2% and a material-to-hydrogen peroxide ratio of 1:6 at 60°C for 2 hours. The filtrate resulting from bleaching was separated from the alpha-cellulose using filter paper. The cassava stem fibers were washed with aquadest to remove any remaining filtrate and neutralize the pH. The cassava stem fibers were then dried in an oven at 105°C for 1 hour to reduce moisture content. The material was tested to determine the results of the cellulose isolation process, including Alpha cellulose, hemicellulose, and lignin content.

Acetylation Process

10 grams of alpha cellulose, after the cellulose isolation process, were added to 250 ml of glacial acetic acid for activation at 50°C for 3 hours and a stirring speed of 125 rpm. Acetic anhydride and 3 drops of sulfuric acid as a catalyst were added at 50°C with a stirring speed of 250 rpm, followed by cooling to 25°C. The main variables in this process are the acetylation time (30, 60, 90, 120, 150 minutes) and the volume of acetic anhydride (10, 12.5, 15, 17.5, 20 ml), which are adjusted to evaluate their effect on the final product. 2 ml of aquadest and 5 ml of glacial acetic acid were added for hydrolysis at 25°C for 30 minutes with a stirring speed of 150 rpm. 1 gram of sodium acetate was added at 25°C for 5 minutes with a stirring speed of 150 rpm. The cellulose acetate was separated from the filtrate using filter paper. The cellulose acetate was washed with aquadest until pH = 7 and then dried in an oven at 105°C for 2 hours

Analysis Method

The raw material of cassava stem fibers that had undergone pretreatment, including hydrolysis, delignification, and bleaching, was re-tested to determine its composition after the pretreatment process. The testing was conducted using High-Performance Liquid Chromatography (HPLC) to determine the composition of the material after pretreatment. The formed cellulose acetate was tested for its acetyl content using HPLC, and the degree of substitution was calculated based on ASTM D871-96 (2010). Additionally, FTIR testing was conducted to determine the functional groups present.

RESULTS AND DISCUSSION

Raw Material and After Pre-treatment Analysis Results

Tabel 1. Raw Material Content Analysis

Composition	Content(%)
Cellulose	48,72
Hemicellulose	17,74
Lignin	23,89

Tabel 2. After Pre-tratment Content Analysis

Composition	Content(%)
Cellulose	49,78
Hemicellulose	14,58
Lignin	8,27

Based on the table 1 and 2 , it can be seen that the hydrolysis, delignification, and bleaching processes can reduce the content of hemicellulose and lignin. After the pretreatment process, the hemicellulose content decreased by 3.16%, while the lignin content decreased significantly by 15.62%.

The effect of acetic anhydride volume and acetylation time on acetyl content.

Tabel 3. Acetyl Content Analysis Results

Acetic anhydride Volume	30 minutes	6o minutes	90 minutes	120 minutes	150 minutes
10	19,59	20,13	20,42	21,71	22,75
12,5	23,91	24,74	25,76	26,05	26,53
15	24,84	25,51	26,19	26,78	27,39
17,5	25,65	27,29	28,94	30,55	32,18
20	26,39	28,44	30,42	32,64	34,80

The highest acetyl content was 34.80% with an acetic anhydride volume of 20 mL and an acetylation time of 150 minutes. A similar trend was also shown in the other results, which can be seen in the graph below.

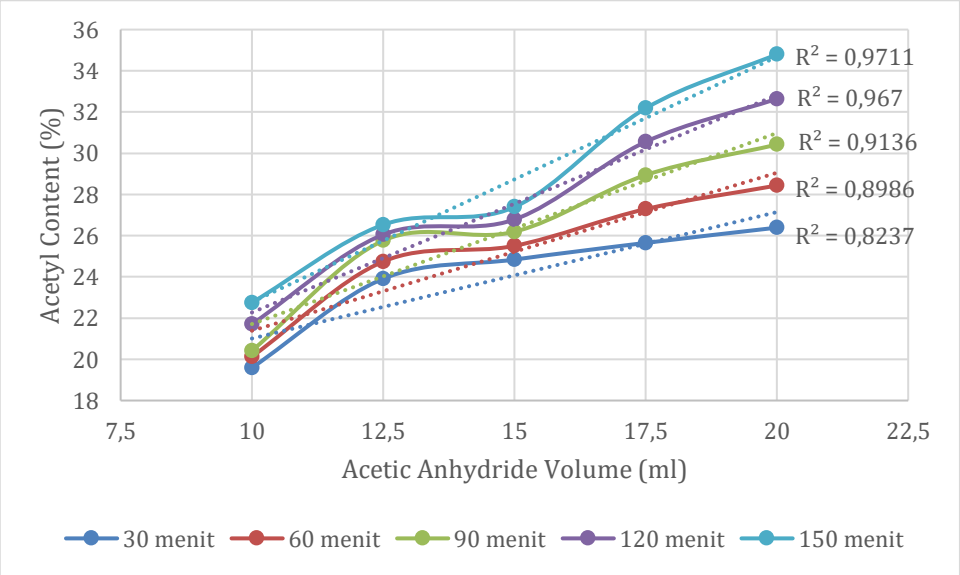

Figure 1. The effect of varying acetic anhydride volume at different acetylation times on acetyl content.

Based on Figure IV.1, the relationship between varying acetic anhydride volume at different acetylation times on acetyl content shows a correlation between acetic anhydride volume and acetylation time on acetyl content, where the higher the acetic anhydride volume and acetylation time, the higher the acetyl content. The higher the acetyl content, the higher the degree of substitution, which will affect the characteristics of the resulting cellulose acetate (Seto & Sari,

2013). This is consistent with Darwis et al. (2003), who stated that the acetyl content of cellulose acetate will increase with an increasing ratio of acetic anhydride. This is because the higher the acetic anhydride volume, the more acetyl groups will collide with each other, resulting in a more complete reaction and increasing the possibility of acetyl groups replacing hydroxyl groups on cellulose (Anjarwati, 2023). The analysis results also show the effect of varying acetylation time on acetyl content at different acetic anhydride volumes. From the figure above, it can be explained that the acetyl content for acetic anhydride volumes of 10 mL, 12.5 mL, 15 mL, 17.5 mL, and 20 mL increases with longer acetylation times. The percentage of acetyl increases up to a maximum at an acetylation time of 150 minutes. This is consistent with Gaol's research (2013), which found that increasing the acetylation time increases the percentage of acetyl up to a maximum at an acetylation time of 150 minutes. This is because longer acetylation times will break down the cellulose acetate structure into glucose acid, resulting in a decrease in acetyl content (Darmawan, 2018). Furthermore, a higher acetyl content is closely associated with an increase in the melting point of cellulose acetate. This occurs because the thermal resistance of cellulose acetate is greater than that of unmodified cellulose. As more hydroxyl groups in the cellulose structure are substituted with acetyl groups, the resulting polymer exhibits enhanced thermal stability, thereby elevating its melting point (Gaol, 2013). A relatively good acetyl content was obtained under optimal conditions, i.e., at an acetylation time of 150 minutes and an acetic anhydride volume of 20 mL, which was 34.80%. This is close to the characteristics of commercial cellulose acetate (39-40%), which meets the Indonesian National Standard (SNI). From the results of this study, it can be inferred that if a higher solvent concentration (>30%) is used, it is likely that the acetyl content will increase to meet the SNI category. Additionally, the acetyl content will indicate the utility of each type of cellulose acetate. Based on the results, cellulose acetate of the monoacetate type was obtained with an acetyl content below 35%. Cellulose monoacetate can be used in the production of plastics and paints.

The effect of acetic anhydride volume and acetylation time on Degree of Substitution.

Tabel 4. Degree of Substitution Calculation Results

Acetic anhydride Volume	30 minutes	6o minutes	90 minutes	120 minutes	150 minutes
10	0,913	1,176	1,236	1,290	1,340
12,5	0,944	1,230	1,280	1,402	1,484
15	0,961	1,297	1,327	1,521	1,631
17,5	1,038	1,317	1,367	1,642	1,806
20	1,103	1,350	1,409	1,769	1,987

The degree of substitution calculation results are used to determine the type of cellulose acetate obtained. A degree of substitution value of 0-2 is classified as cellulose monoacetate with an acetyl content of less than 35%. Cellulose monoacetate can be applied in the production of plastics and paints.

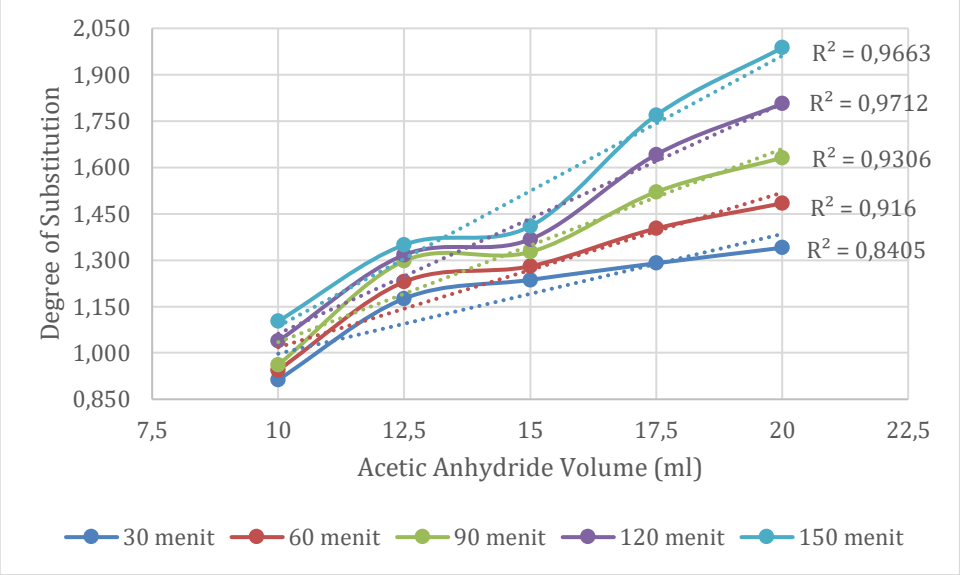

Figure 2. The effect of varying acetic anhydride volume at different acetylation times on degree of substitution.

Based on Figure IV.2, the relationship between varying acetic anhydride volume at different acetylation times on the degree of substitution shows a correlation between acetic anhydride volume and acetylation time on the degree of substitution, where the higher the acetic anhydride volume and acetylation time, the higher the degree of substitution. The higher the acetic anhydride volume, the greater the degree of substitution produced. This occurs because the higher the concentration, the more particles there are, causing molecular movement to become faster. As a result, the contact between reactants and materials becomes more frequent, providing a greater opportunity for acetyl groups to substitute hydroxyl groups (Utami, 2021). The analysis results also show the effect of varying acetylation time on the degree of substitution at different acetic anhydride volumes. The results show that the degree of substitution increases with longer acetylation times. This is because longer acetylation times allow more cellulose acetate to be acetylated, increasing the acetyl content. The increased acetyl content affects the degree of substitution. The degree of substitution increases with increasing acetyl content (Anjarwati, 2023). From the figure above, it can be explained that the degree of substitution for acetic anhydride volumes of 10 mL, 12.5 mL, 15 mL, 17.5 mL, and 20 mL increases with longer acetylation times. The highest degree of substitution was obtained under optimal conditions, i.e., at an acetylation time of 150 minutes and an acetic anhydride volume of 20 mL, which was 1.987. The degree of substitution is used to determine the number of hydroxyl groups (-OH) replaced by acetyl groups (-OCH3CO) as a reference for the formation of cellulose acetate (Utami, 2021). This means that the higher the degree of substitution, the more hydroxyl groups are replaced by acetyl groups to form cellulose acetate. The degree of substitution of cellulose acetate ranges from 0 to 3.5, and an increase in the degree of substitution will increase the melting point of cellulose acetate (Gaol, 2013). Additionally, the degree of substitution will indicate the utility of each type of cellulose acetate. Based on the results, cellulose acetate of the monoacetate type was obtained with an acetyl content below 35% and a degree of substitution below 2. Cellulose monoacetate can be used in the production of plastics and paints.

FTIR Analysis Result

The results obtained from the study were analyzed using FTIR (Fourier Transform Infrared Spectroscopy) procedure at the Pharmacy Laboratory, Department of Pharmacy, Faculty of Pharmacy, Airlangga University, Surabaya. The figure shows that there are characteristic absorption peaks for cellulose acetate, which are the absorption peaks of the carbonyl group C=O and the ester group C-O from the acetyl group.

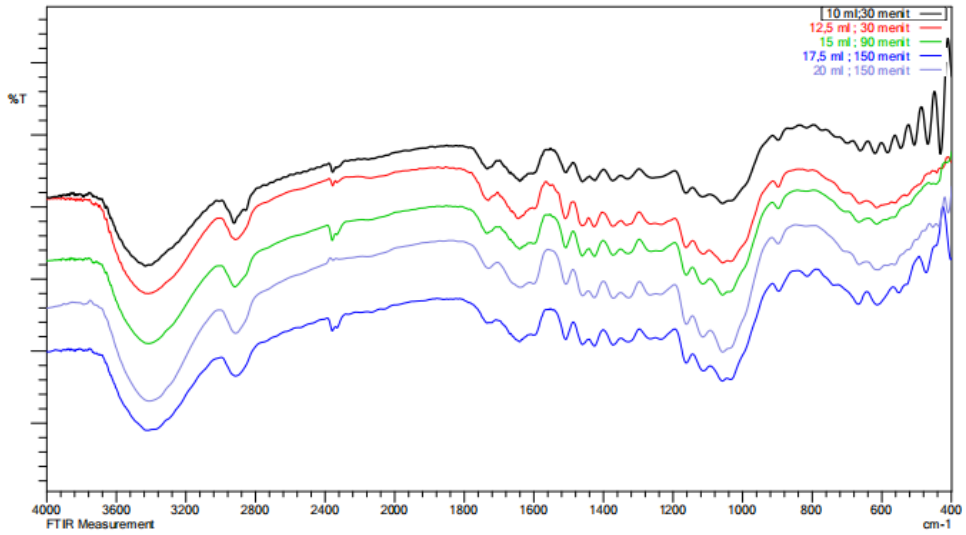

Figure 3. The Result of FTIR Analysis

Figure IV.3 shows that the five samples have a carbonyl group C=O in the form of carboxylic acid in the spectrum of cellulose acetate, which is located in the wavenumber range of 1730.15 - 1734.01 cm-1. The ester group C-O in the form of acetyl is located in the wavenumber range of 1234.44 - 1265.3 cm-1. The absorption of the C-H stretch group in the form of alkyl is present at 2914.44 - 2924.09 cm-1. The C-H bending group is located in the wavenumber range of 1371.39 - 1425.4 cm-1. According to Matius (2023), the FTIR analysis of cellulose acetate is considered successful when peaks corresponding to the carbonyl group (C=O) and the ester group (C–O) from the acetyl group appear, indicated by wavenumbers in the range of 1730–1751 cm-1 (C=O) and 1227–1267 cm-1 (C–O), which are characteristic of cellulose acetate. The test results show that the five samples have characteristic absorption peaks for cellulose acetate, which are the absorption peaks of the carbonyl group C=O and the ester group C-O from the acetyl group.

CONCLUSION

A relatively good acetyl content of 34.8% and the best degree of substitution of 1.987 were obtained from the variables of 150 minutes acetylation time and 20 mL acetic anhydride volume. The best cellulose acetate in this study, which has an acetyl content of 34.8%, still does not meet the Indonesian National Standard (SNI) 06-2115-1991 for cellulose acetate, which requires an acetyl content of 39%. The highest yield percentage of 99.5% was obtained at 150 minutes acetylation time and 12.5 mL acetic anhydride volume. The best cellulose acetate in this study, with an acetyl content of 34.8% and a degree of substitution of 1.987, indicates that it is a cellulose monoacetate that can be used in the plastics and plants industry. The cellulose acetate in this study has carbonyl group C=O and ester group C-O from the acetyl group, which are characteristic absorption peaks for cellulose acetate. The resulting cellulose monoacetate can be utilized as a key material in the production of bioplastics. Cellulose acetate possesses high commercial value due to its excellent physical and optical properties. Additionally, its biodegradable nature makes it an attractive and environmentally friendly option. Further research on the implementation of cellulose monoacetate in bioplastic production is warranted. Further studies are required to optimize the volume of acetic anhydride used to produce cellulose acetate that conforms to the Indonesian National Standard (SNI). Moreover, characterization of the resulting cellulose acetate, including its mechanical properties, is essential to determine its potential applications.

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