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Synthesis of Cellulose Nanocrystals from Rice Husk Using Nitric Acid Hydrolysis

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Abstract

Rice husk residue has excellent potential as a raw material for making cellulose nanocrystals because of its relatively high cellulose content. This research focuses on determining the most effective hydrolysis conditions, especially the reaction time and nitric acid (HNO_3) content, in the synthesis of cellulose nanocrystals. This study also aims to determine the effect of variations in nitric acid concentration and hydrolysis time on the amount of product produced (yield) and the final size of cellulose nanocrystals. The process of making these nanocrystals involves three main stages, namely the removal of lignin using a 17,5% NaOH solution, followed by annealing using a 10% H_2O_2 solution, and finally breaking the cellulose bonds (acid hydrolysis) with a concentrated nitric acid solution of between 2% and 6% for different time periods (30 to 15 minutes). Product characterization using FT-IR and SEM has been carried out to confirm the presence of cellulose and determine the size of the nanocrystals formed. Experimental results show that the smallest cellulose crystal size achieved is between 0,93 to 65,10 µm, which was obtained through hydrolysis using 6% nitric acid for 150 minutes.

Keywords: nitric acid, acid hydrolysis, cellulose nanocrystals, rice husk

INTRODUCTION

As an agricultural country, Indonesia has a large population working in agriculture, with rice being one of the main crops (Mujiyanti et al., 2021). BPS data shows that the country's rice production reached 54,75 million tons in 2022, and the Jombang area recorded a figure of more than 337,17 tons. The rice milling process produces waste in the form of rice husk, approximately 20-30% of the total weight. Unfortunately, most of this husk is not used optimally and is often thrown away. Efforts to use rice husk waste to reduce environmental negative impacts are still limited. If left to decay naturally, agricultural waste tends to decompose slowly, which can cause pollution and potentially harm human health (Listiana et al, 2021).

Cellulose is an organic compound that is abundant in nature. The cellulose structure consists of long straight chains, consisting of hundreds to thousands of D-glucose units linked by β-(1,4) glycosidic bonds (Souhoka & Latupeirissa, 2018). In nature, cellulose generally occurs in the form of lignocellulose, a mixture of cellulose, lignin, and hemicellulose. As a natural polymeric material that is biodegradable and renewable, cellulose has a variety of uses (Rizkiana et al., 2024).

The main composition of rice husk consists of cellulose (38%), hemicellulose (18%), lignin (22%), and silica (22%) (Solihudin et al., 2020). The high cellulose content in rice husk opens the opportunity to produce it into cellulose nanocrystals. This nanomaterial is obtained from cellulose with diameter dimensions between 5-30 nanometers and is elongated with a size of 20-100 nanometers (Kargarzadeh et al., 2017). Cellulose nanocrystals act as a basic material in the manufacture of medicines (Aulia & Gea, 2013). Furthermore, due to its very small size, this material becomes an essential main component in biopolymer nanocomposites, providing high surface reactivity, easy degradation ability, and superior mechanical properties (Young's Modulus 1,50 GPa and tensile strength 1,7 GPa) (Kaur et al., 2021).

There are various ways to produce cellulose nanocrystals, one of which is acid hydrolysis (Purwanti & Dampang, 2017). The acid hydrolysis technique is considered superior because it can produce nanocrystals with a high level of crystallinity, optimal thermal stability, and the potential to reduce energy consumption during the process (Muljani et al., 2023). This acid hydrolysis process involves three main steps: delignification (lignin removal), bleaching, and hydrolysis. The purpose of delignification is to reduce the lignin content in lignocellulosic materials, which consist of lignin, hemicellulose, and cellulose. Generally, delignification is carried out using a base solution such as sodium hydroxide (NaOH). In this reaction, hydroxide ions (OH^-) break the bonds in the basic structure of lignin, thus making the lignocellulose structure more open. Meanwhile, sodium ions (Na^+) will bind the decomposed lignin, forming sodium lignate compounds more soluble in water. The bleaching process is carried out to remove lignin residues that are still present in the fiber. The remaining lignin can change the color of the resulting cellulose product (Lismeri et al., 2019). The bleaching agents commonly used are H_2O_2 or NaOCl.

The acid hydrolysis process in the manufacture of cellulose nanocrystals generally involves the use of strong acids such as sulfuric acid (H_2SO_4), hydrochloric acid (HCl), and nitric acid (HNO_3). The use of strong acids aims to dissolve the amorphous part of the cellulose structure. Although sulfuric acid (H_2SO_4) is often chosen to produce cellulose nanocrystals, studies have shown that its use can cause excessive degradation, decreasing the amount of product obtained. A study by (Gülsu & Yüksektepe, 2021) offers an alternative by using nitric acid (HNO_3), which has the advantages of being non-corrosive, easily soluble in water, and effective in removing the amorphous part of cellulose, thus potentially increasing the yield of nanocrystal isolation.

Factors affecting hydrolysis include temperature, hydrolysis time, acid concentration, and stirring speed. High acid concentration accelerates the process of decomposing cellulose fibers, affecting cellulose size (Artati et al., 2013). The yield tends to decrease as the hydrolysis time increases (Mardina et al., 2016). Based on the description, this study focuses on hydrolysis time and acid concentration as variables. Given the limitations of research on the synthesis of cellulose nanocrystals from rice husks, this study is expected to broaden insights and contribute to related research fields. The main objectives of this study are to identify the ideal combination of hydrolysis time and nitric acid (HNO_3) concentration in producing cellulose nanocrystals, analyze how variations in nitric acid concentration and hydrolysis time affect the amount of product produced, and determine the size of the cellulose nanocrystals formed.

METHODOLOGY

Materials and Instrumentals

This study used several materials, including rice husks that had been sieved to a size of 100 mesh and obtained from a rice mill located in Jombang, East Java. Other chemicals used were sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), nitric acid (HNO_3), and distilled water, all of which were obtained from local chemical stores. The types of equipment used during this research are visually illustrated in Figure 1.

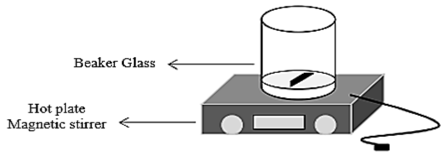

Figure 1. Cellulose Nanocrystal Synthesis Process Tool Series

The leading equipment used in this study consisted of a hot plate magnetic stirrer and a glass beaker that functioned as a container for the delignification, bleaching, and acid hydrolysis processes. Meanwhile, the supporting equipment used included an oven, grinder, sieve, analytical balance, dropper, glass funnel, measuring cup, filter paper, Erlenmeyer flask, pH paper, stirrer, and thermometer.

Methods

Sample Preparation

Rice husks are dried under sunlight to reduce their water content. After drying, they are crushed or milled and sieved using a 100-mesh sieve. Husks that do not pass the sieve (oversize) will be ground again. Rice husk flour was analyzed for hemicellulose, cellulose, and lignin content using the Chesson-Datta method. The flow of the raw material preparation process is shown in Figure 2.

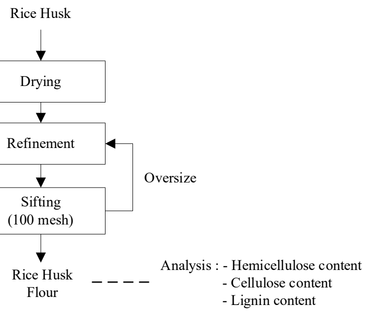

Figure 2. Sample Preparation Flowchart

Cellulose Isolation

This study mixed 15 grams of rice husk flour with 200 ml of 17,5% concentrated sodium hydroxide (NaOH) solution. Next, the mixture was stirred using a magnetic stirrer at 150 rpm for one hour at 100°C. After heating and stirring, the mixture was separated by filtration and then washed with distilled water until it reached a neutral state with a pH of 7. The remaining solid material was dried in an oven at 100°C. The next step was bleaching the precipitate using 100 ml of 100% hydrogen peroxide (H_2O_2) solution, which was also stirred with a magnetic stirrer at 80°C for 60 minutes at 150 rpm. This process was continued by filtering and washing with distilled water until the pH reached 7. Then it was dried in an oven at 100°C. The resulting precipitate was then bleached using 100 ml of 10% H_2O_2 solution and stirred with a magnetic stirrer at 80°C for 60 minutes at a speed of 150 rpm. The process was continued by filtration and neutralization using distilled water until reaching pH 7. The resulting precipitate was then dried in an oven at 100°C until cellulose powder was obtained. The flow of the cellulose isolation process is shown in Figure 3.

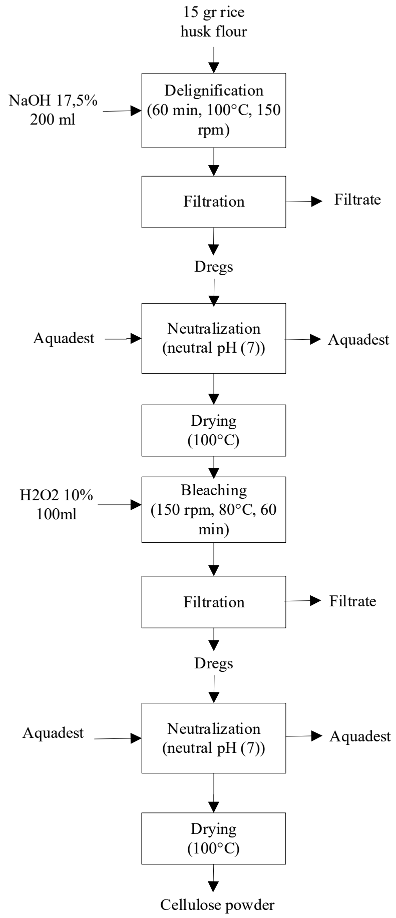

Figure 3. Flowchart of Cellulose Isolation

Synthesis of Cellulose Nanocrystals

Eight grams of cellulose powder were hydrolyzed using 60 ml of nitric acid solution according to the concentration variables of 2%; 3%; 4%; 5%; and 6% and stirred with a magnetic stirrer according to the variables, namely 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes at a temperature of 80°C at a speed of 150 rpm. After the acid hydrolysis process, the solution was filtered to obtain dregs, which were then neutralized to reach pH 7. The dregs obtained were then dried and analyzed to determine the yield, size, and hydroxyl groups. The flow of the cellulose nanocrystal synthesis process is shown in Figure 4.

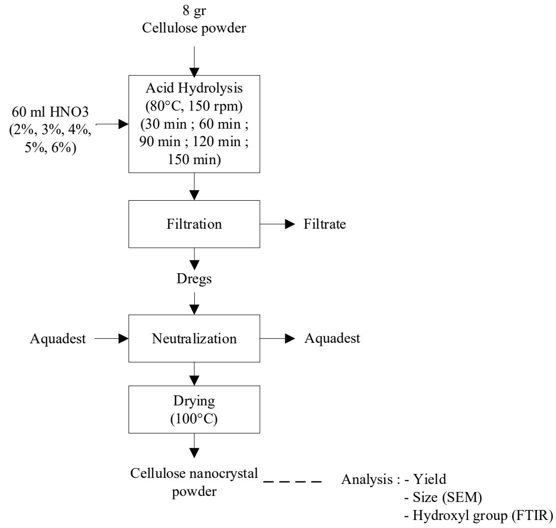

Figure 4. Flowchart of Cellulose Nanocrystal Synthesis

Data Analysis

Yield Analysis

This yield calculation is used to determine the weight of the product produced. To calculate the yield, you can use the following formula:

$$\%\text{Yield} = \frac{W_{\text{final}}}{W_{\text{initial}}} \times 100\% \quad (1)$$

$\%Yield$ is the percent yield, W_{final} is the weight of the product, and W_{initial} is the weight of the material (Sutejo et al., 2023).

RSM *(Response Surface Methodology)*

Response Surface Methodology (RSM) analysis is used to identify the best conditions related to the concentration and duration of acid hydrolysis. In processing the data, Design Expert software version 13 will be used. RSM visualization in graphical form can be generated by the Design Expert after variables and response data are collected. Once the graph is formed, the point indicating the most optimal condition is located at the very bottom of the graph. In addition, this analysis also produces equations that describe the relationship between the two variables studied. These Response Surface Methodology (RSM) analysis is used to identify the best conditions related to the concentration and duration of acid hydrolysis. In processing the data, Design Expert software version 13 will be used. RSM visualization in graphical form can be generated by the Design Expert after variables and response data are collected. Once the graph is formed, the point indicating the most optimal condition is located at the very bottom of the graph. In addition, this analysis also produces equations that describe the relationship between the two variables studied. These equations will form the highest level three and four-dimensional mathematical models, with the general form of each being as follows:

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \cdots + \beta_k x_k + \varepsilon \quad (2)$$

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{11} x_1^2 + \beta_{22} x_2^2 + \beta_{12} x_1 x_2 + \varepsilon \quad (3)$$

y is the predicted response or yield of cellulose, x_1 is the concentration of HNO_3, x_2 is the time of acid hydrolysis, and β is a constant (Hidayat et al., 2020).

SEM *(Scanning Electron Microscope)*

Analysis using Scanning Electron Microscopy (SEM) was carried out to measure the dimensions of the successfully synthesized cellulose nanocrystals (Aulia & Gea, 2013).

FTIR (Fourier *Transform Infra Red)*

Fourier Transform Infrared Spectroscopy (FTIR) analysis was performed to identify the presence of hydroxyl groups in cellulose nanocrystals. After the entire infrared spectrum was generated, the spectral data was interpreted and compared with the information contained in the scientific literature (Salimi et al., 2021).

RESULTS AND DISCUSSION

The lignin removal process is done through a delignification process using NaOH solution. In this process, lignin reacts with Na+ ions to dissolve and form a filtrate. Lignin loss in the fiber is indicated by the appearance of a thick black solution known as leachate. After the delignification process, bleaching is carried out to remove hemicellulose and the remaining lignin.

Lignocellulose Content with the Chesson-Datta Method 1981

The levels of lignin, cellulose, and hemicellulose in the material samples and the effectiveness of the bleaching process were measured using the Chesson-Datta method. This analysis was intended to further confirm the quality of the cellulose before proceeding to the acid hydrolysis stage. The results of the analysis are then summarized and presented in Table 1.

Table 1. Results of Lignocellulose Content Analysis

Sample Type	Level (%)		
	Hemicellulose	Cellulose	Lignin
Material	18,88	42,09	17,99
Bleaching	16,93	47,99	9,08

Based on the results of the analysis, the bleaching stage successfully increased the proportion of cellulose in the material, as indicated by the decrease in lignin and hemicellulose levels. The main purpose of bleaching in cellulose synthesis is to bleach the delignified fibers while removing residual lignin, hemicellulose, color-causing compounds (chromophores), ash, and pectin, thereby producing cellulose with a high level of purity (Wildan et al., 2010). The increase in cellulose content to 47,99% after bleaching indicated the success of this stage, while the hemicellulose and lignin contents decreased to 16,93% and 9,08%, respectively. The compositional analysis of this lignocellulose confirmed that the bleaching process produced cellulose of sufficient quality to be used as a raw material in the synthesis of cellulose nanocrystals via acid hydrolysis.

Cellulose Nanocrystal Yield

The formation of cellulose nanocrystals in this study used the acid hydrolysis method with varying concentrations of nitric acid (HNO_3) solution. The two main factors that affect this hydrolysis process are the hydrolysis duration and the acidity level of the solution. The acid concentrations used included 2%, 3%, 4%, 5%, and 6%, while the hydrolysis times tested were 30 minutes, 60 minutes, 90 minutes, 120 minutes, and 150 minutes. A visualization of the correlation between acid concentration and hydrolysis time on the product yield can be seen in Figure 5.

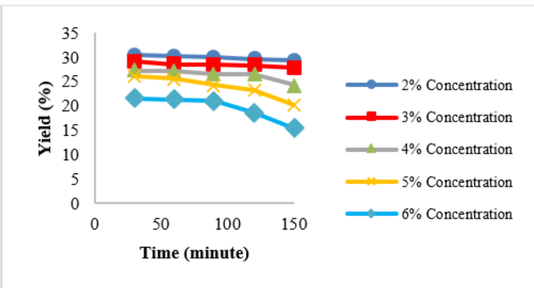

Figure 5. Relationship between Acid Concentration and Hydrolysis Time on Cellulose Nanocrystal Yield

The data in Figure 5 illustrates that using low concentrations of hydrolytic acid for a short time produces a larger amount of product. On the other hand, high acid concentrations and long hydrolysis times tend to produce lower yields. This finding is consistent with research (Khadafi et al., 2023), which states that exposure to high acid concentrations for an extended period can trigger cellulose degradation to other sugar compounds. The breakdown of cellulose to by-products directly reduces the amount of cellulose obtained (yield), causing a decrease in yield.

The results of observations at various concentrations and hydrolysis times show differences in the color of the resulting products. Cellulose samples treated with the lowest acid concentration and shortest hydrolysis time display the lightest color. On the other hand, increasing the acid concentration and extending the hydrolysis time correlate with darker sample colors. This phenomenon indicates that more components are dissolved during the process, which decreases the amount of cellulose nanocrystals due to significant cellulose degradation. This statement is supported by the view (Nasution et al., 2020), which states that an acid concentration that is too high can cause total damage to the cellulose structure. Furthermore, research (Lestari et al., 2023) identified the characteristics of successful cellulose isolation in the form of a tasteless, white and odorless powder. Based on these criteria, the cellulose used in this study is considered to have met the expected quality standards.

Response Surface Methodology (RSM) analysis

The synthesis process of cellulose nanocrystals was analyzed using the Response Surface Methodology (RSM) method through 25 experiments designed with the help of Design Expert 13 software. Before the design was carried out, two main variables that affect the results (Y) were determined, namely acid concentration (A) and time (B). The experimental data were then explained to obtain optimum conditions in the nitric acid hydrolysis process. Further information regarding the variables and levels used in this study can be seen in Table 2.

Table 2. Determination of Factors and Levels

Variable	Symbol	Level Code		
		-1	0	1
HNO_3 Concentration (%)	A	2	4	6
Hydrolysis Time (min)	B	30	90	150

Each factor in the experiment is divided into three levels: low (–1), medium (0), and high (+1). Based on the data obtained, Analysis of Variance (ANOVA) is used to determine the influence of each factor on the response.

Analysis of Variance (ANOVA) aims to analyze the significant influence of each factor on the results of cellulose nanocrystals. A summary of the analysis results obtained through Design Expert 13 software is shown in Table 3.

Table 3. Results of Analysis of Variance (ANOVA)

Analysis of Variance						
Source	Sum of Squares	df	Mean Square	F-Value	p-Value	Significance
Model	390,00	9	43,33	414,00	< 0,0001	Significant
A-concentration	17,27	1	17,27	165,00	< 0,0001	Significant
B-time	11,32	1	11,32	108,11	0,0002	Significant
AB	5,09	1	5,09	48,62	< 0,0001	Significant
A^2	12,54	1	12,54	119,81	< 0,0001	Significant
B^2	3,76	1	3,76	35,96	< 0,0001	Significant
A^2B	0,2055	1	0,2055	1,96	0,1815	Not significant
AB^2	3,18	1	3,18	30,38	< 0,0001	Significant
A^3	0,7096	1	0,7096	6,78	0,0199	Significant
A^3B	1,32	1	1,32	12,65	0,0029	Significant
Residual	1,57	15	0,1047			
Cor Total	391,57	24				

The results of the Analysis of Variance (ANOVA) presented in Table 3 show the P-Value of the model of <0,0001 and 0,0002, both of which are smaller than the significance level (α) of 0,05. This indicates that the model used is statistically significant. This supports the alternative hypothesis (H1), which states that at least one factor significantly affects the response yield. The factors that have a considerable effect are the concentration of HNO3 (A) and the hydrolysis time (B), with P-Values of <0,0001 and 0,0002, respectively, both of which are smaller than 0,05. Based on the analysis of the effect on the response yield, the concentration of HNO3 (A) is more dominant than the hydrolysis time (B).

A 4th Order Polynomial (quartic) model was obtained based on the ANOVA data. This quartic polynomial model can be used to predict optimal conditions for yield response based on experimental data.This quartic polynomial model can be used to predict optimal conditions for yield response based on experimental data. The 4th Order Polynomial equation formed comes from the influence of each factor on the yield response. The resulting equation is as follows :

$$y = 51,8381 - (17,3300 \times \text{Concentration}) - (0,2335 \times \text{Time}) + (0,1712 \times \text{Concentration x Time}) + (4,3350 \times \text{Concentration}^2) + (0,0004 \times \text{Time}^2) - (0,0396 \times \text{Concentration}^2 \times \text{Time}) - (0,0002 \times \text{Concentration} \times \text{Time}^2) - (0,3869 \times \text{Concentration}^3) + (0,0032 \times \text{Concentration}^3 \times \text{Time})$$

The predicted results (RSM) can be obtained from the equation above, as shown in Table 4.

Table 4. Results of Cellulose Nanocrystal Synthesis Experiment

No	HNO_3 Concentration (%)	Hydrolysis Time (min)	RSM Yield (%)	Eksperimen Yield (%)
1	2	30	30,7059	30,6020
2	2	60	29,9889	30,2626
3	2	90	29,2719	29,9732
4	2	120	28,5549	29,6865
5	2	150	27,8379	29,4359
6	3	30	28,5398	29,0992
7	3	60	28,3028	28,5758
8	3	90	27,7058	28,5337
9	3	120	26,7488	28,2997
10	3	150	25,4318	27,9437
11	4	30	27,4315	27,2418
12	4	60	27,0265	27,1119
13	4	90	25,9015	26,6413
14	4	120	24,0565	26,5297
15	4	150	21,4915	24,2835
16	5	30	25,6356	26,1022
17	5	60	24,9906	25,6408
18	5	90	23,2656	24,3006
19	5	120	20,4606	23,1741
20	5	150	16,5756	20,2226
21	6	30	21,4067	21,5272
22	6	60	21,0257	21,2834
23	6	90	19,2047	21,0078
24	6	120	15,9437	18,6644
25	6	150	11,2427	15,4970

Based on the prediction data in Table 4, there is a difference between the results obtained through the Response Surface Methodology (RSM) and the experimental results. To illustrate this difference, a graph is needed that compares the actual response (from the experiment) with the predicted response (the results of the RSM calculation). If the data points are evenly distributed along the line, the data is usually distributed, and the RSM model provides good predictions. Conversely, if the points are clustered on one side, this indicates a mismatch between the model used and the data obtained.

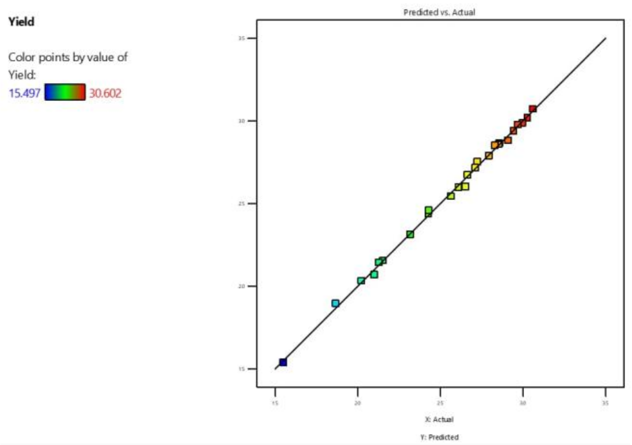

Figure 6. Normal curve plot of Experiment yield against RSM yield

Figure 6 shows the distribution of residual points that are not all exactly on the standard line, but the distribution is still along the black line. The plot and response surface on the effect of HNO_3 concentration (A) and nitric acid hydrolysis time (B) on the yield response are seen in Figures 7 and 8 below.

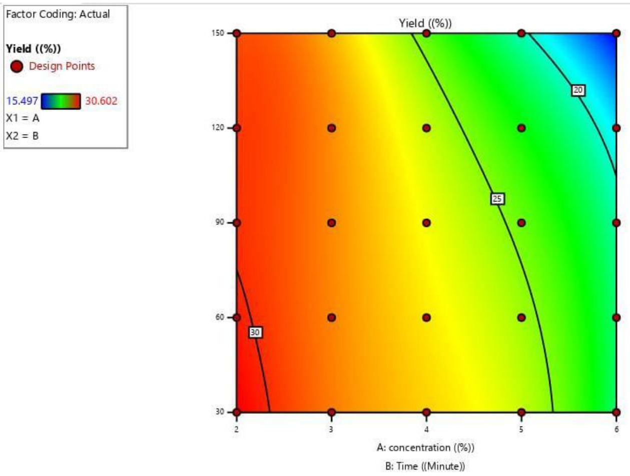

Figure 7. Contour plot Yield

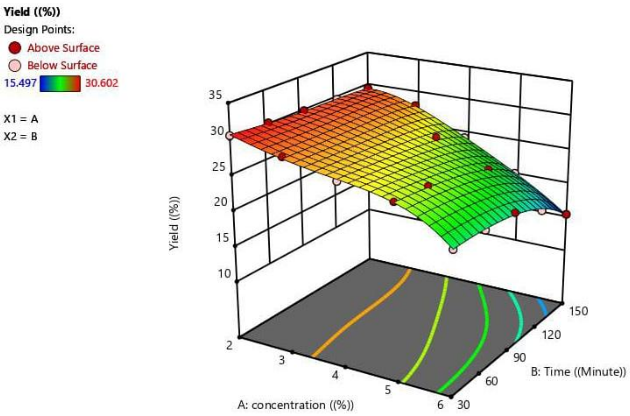

Figure 8. Response surface Yield

Optimal conditions for maximizing yield were predicted using Design Expert 13 software, based on data from 25 trials. The results of determining the optimal conditions for yield response are presented in Table 5.

Table 5. Optimal Concentration and Time Based on RSM Analysis

Solution				
Point	Concentration HNO_3 (%)	Time (min)	Yield (%)	Composite Desirability
Maximum	2,023	34,973	30,604	1,000
Minimum	6	150	15,59	0,994

Table 5 shows that the optimal solution for the yield response was achieved at a hydrolysis time of 34,973 min with an HNO_3 concentration of 2,023%. On the other hand, the minimum yield response was observed at 150 min of hydrolysis with an HNO_3 concentration of 6%. The composite attractiveness or response accuracy obtained was 1,000 and 0,994. The attractiveness value is used to assess the accuracy of the solution. An attractiveness value closer to 1,0 indicates greater accuracy in the solution results provided by the program. The values of 1,000 and 0,994 indicate that the software's optimum solution accuracy yielded prediction accuracies of 100% and 99,4%, respectively.

Fourier Transform Infrared (FT-IR) Analysis

The wavelength spectrum of the cellulose sample from rice husk in Figure 9 is compared with the literature data in Table 6 to identify the functional groups present in the cellulose sample. The absorption band at wave number 3336,86 cm^{-1} indicates the presence of O–H stretch vibrations originating from cellulose. The band at 2918,31 cm^{-1} indicates C–H stretch vibrations also originating from cellulose. The C–H bend vibrations of cellulose were identified in the absorption bands at 1420,90 cm^{-1}, 1367,76 cm^{-1}, and 1315,15 cm^{-1}. Meanwhile, the bands at 1277,34 cm^{-1}, 1157,68 cm^{-1}, 1058,56 cm^{-1}, and 1027,27 cm^{-1} indicate the C–O stretch of cellulose, and the band at 1200,47 cm^{-1} is related to the C–O stretch of lignin. The absorption bands at 997,23 cm^{-1} and 894,80 cm^{-1} indicate the vibration of C=C-H or Ar-H bend associated with β-glycosidic. This confirms that cellulose has been obtained from the isolation of rice husk.

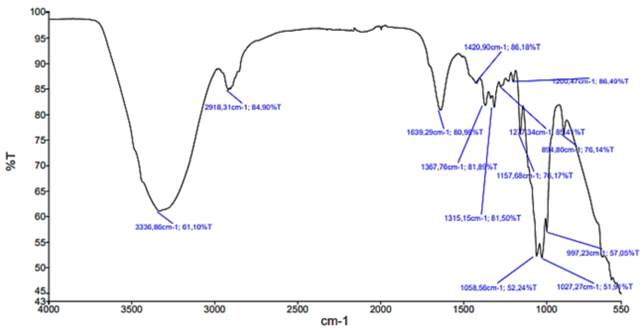

Figure 9. Fourier Transform Infrared (FT-IR) Graph

Table 6. FT-IR analysis of functional groups of cellulose nanocrystals

Sample	Number of Waves	Functional Group	Group	Library sources (Putri, 2018)
Cellulose Nanocrystals	3336,86 cm^{-1}	O-H stretch	Cellulose	3000 - 3750 cm^{-1}
	2918,31 cm^{-1}	C-H stretch	Cellulose	2700 - 3000 cm^{-1}

	1420,90 cm^{-1}	C-H bend	Cellulose	1300 - 1475 cm^{-1}
	1367,76 cm^{-1}			
	1315,15 cm^{-1}			
	1277,34 cm^{-1}	C-O stretch	Cellulose	1000 - 1300 cm^{-1}
	1200,47 cm^{-1}	C-O stretch	Lignin	
	1157,68 cm^{-1}	C-O stretch	Cellulose	
	1058,56 cm^{-1}	C-O stretch	Cellulose	
	1027,27 cm^{-1}	C-O stretch	Cellulose	
	997,23 cm^{-1}	C=C-H, Ar-H bend	β-glycosidic	650 - 1000 cm^{-1}
	894,80 cm^{-1}			

Scanning Electron Microscope (SEM) Analysis

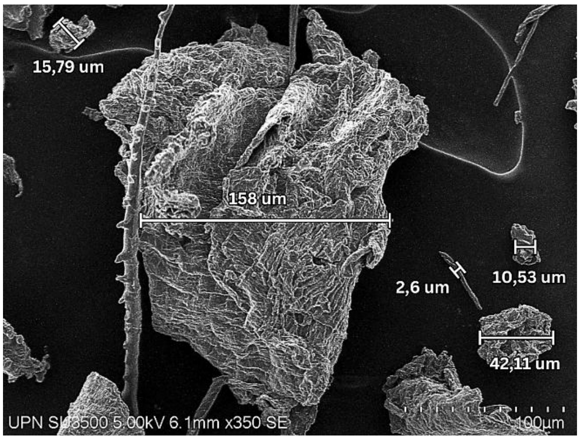

Figure 10. Scanning Electron Microscope (SEM) Results

A Scanning Electron Microscope (SEM) was used to analyze the fiber diameter after acid hydrolysis. Three of the 25 samples analyzed were selected for SEM testing: those treated with 2% concentration for 30 min, 4% for 90 min, and 6% for 150 min. The experimental results and Response Surface Methodology (RSM) showed that 2% concentration for 30 min produced the highest yield, while a 6% concentration for 150 min produced the lowest yield. In addition, to assess whether acid hydrolysis effectively reduced the fiber size, 4% concentration for 90 min was also analyzed. SEM examination was performed at a magnification of 100 µm.

The measurement results of the three samples showed that hydrolysis with 2% nitric acid for 30 minutes produced fibers with diameters ranging from 7,14 µm to 81 µm. Cellulose treated with 4% nitric acid for 90 minutes produced fiber diameters ranging from 2,6 µm to 158 µm. Meanwhile, hydrolysis with 6% nitric acid for 150 minutes produced fibers with diameters ranging from 0,93 µm to 65,10 µm.

SEM analysis showed that increasing the acid concentration effectively reduced the size of cellulose, as evidenced by the reduction in fiber dimensions. However, the cellulose obtained in this study was still classified as microcellulose and did not meet the criteria for nanocellulose. Based on its morphology, cellulose treated with a concentration of 2% for 30 minutes showed a rope-like structure, indicating that it was still classified as a cellulose fiber, not cellulose crystals. In contrast, cellulose treated with a concentration of 4% for 90 minutes and a concentration of 6% for 150 minutes appeared in agglomeration formation, which can be classified as cellulose crystals.

The duration of the hydrolysis process has a significant influence on the formation of cellulose nanocrystals. The longer the hydrolysis time, the greater the possibility of collisions between water and cellulose molecules, resulting in greater cellulose degradation and increased glucose production (Utami et al., 2014). According to Joseph et al., 2023, the optimal hydrolysis time using nitric acid is 2 hours at a temperature of 80°C. Meanwhile, (Rubleva et al., 2019) research found that a hydrolysis time of 3 hours was required to produce cellulose nanocrystals with nitric acid. This study adopted the hydrolysis time suggested by Joseph et al., 2023. However, the cellulose produced did not reach the size of nanocrystals. Therefore, the hydrolysis time used in this study was still insufficient to achieve optimal results (Wildan et al., 2010).

CONCLUSION

The cellulose produced in this study has not yet reached the nanoscale. The smallest particle sizes obtained ranged from 0,93 to 65,10 µm during hydrolysis using nitric acid at a concentration of 6% for 150 minutes. This process resulted in a relatively low yield of approximately 15%.

ACKNOWLEDGMENT

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