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A One-Pot Method for The Synthesis of Nanocellulose from Palm Oil Empty Bunches Using Fe-Cr Combination Catalyst

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Abstract. Oil Palm Empty Fruit Bunches (OPEFB) are one of the byproducts of Crude Palm Oil (CPO) production. Due to its high cellulose content, OPEFB can be processed into nanocellulose, one of its applications. The objective of this study was to determine the procedure for synthesizing nanocellulose from OPEFB using the one-pot method with a combination of Fe and Cr catalysts, as well as the properties and effect of the catalyst composition on the nanocellulose synthesized from OPEFB. In this investigation, OPEFB was processed into nanocellulose in a single vessel. The delignification step was conducted at 90°C for 5 hours with 30%-w/v H_2O_2, and the acid hydrolysis phase was conducted at 75°C for 1 hour with an 8%-w H_2SO_4 solution and $Fe(NO_3)_3.9H_2O$ and $Cr(NO_3)_3.9H_2O$ catalysts. The independent variables were the catalyst composition (3-7%-w/w) and Fe/Cr catalyst weight ratio (0-1). Based on the results, the highest nanocellulose yield was 37%-w, obtained with a catalyst concentration of 5%-w/w with a 0:1 Fe-Cr catalyst ratio. At the same time, the smallest lignin content was 1.711%-wt. which was obtained with a catalyst concentration of 3%-w/w with a Fe-Cr catalyst ratio of 0.5:0.5. Statistical analysis obtained an optimum variable of one-pot method at a catalyst concentration of 3%-w/w with a Fe-Cr catalyst ratio of 0:1. Nanocellulose yield obtained of 30.146%-w and lignin content of 1.610%-w. Meanwhile, from the experimental results at a catalyst concentration of 3%-w/w and a catalyst ratio of Fe-Cr 0:1, a nanocellulose yield of 29.616%-w and a lignin content of 1.799%-w was obtained. Nanocellulose was characterized by PSA, FTIR, TEM, and XRD analyses.

Keywords: Delignification, Nanocellulose, OPEFB, Response Surface Method

INTRODUCTION

Nanocellulose has emerged as a fascinating nanomaterial in recent decades. With remarkable mechanical properties, low weight, biodegradability, and environmental compatibility, nanocellulose offers tremendous potential in various applications in technology and biomedicine. Its ability to enhance the properties of base materials, such as strength, stiffness, and transparency, has made it an attractive material for use in industries such as electronics, paper, composite materials, and biomedicine (Thomas *et al.*, 2020).

One abundant and promising source for

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nanocellulose synthesis is oil palm empty fruit bunch (OPEFB) fibers. EFB fibers are agricultural waste from the palm oil industry, containing high cellulose fibers. In recent years, intensive research has been conducted to exploit OPEFB fibers as a sustainable and cost-effective source of nanocellulose (Fahma *et al.*, 2010; Lani *et al.*, 2014).

Conventional methods for obtaining nanocellulose from OPEFB fibers involve several steps, including OPEFB pretreatment, cellulose fiber extraction, and processing into nanocellulose. Pretreatment aims to effectively remove non-cellulosic components, such as lignin, hemicellulose, and other extractive materials that may impede the nanocellulose synthesis process. The extraction step is performed using enzymatic or chemical approaches. Enzymatic extraction involves using cellulase enzymes to break the cellulose fibers into cellulose nanofibrils. On the other hand, chemical extraction consists of using chemical solutions to remove non-cellulosic components from the OPEFB fibers (Qi *et al.*, 2023). Subsequently, the resulting cellulose fibers are further processed using mechanical techniques to produce nanocellulose with the desired particle size and morphology.

However, these conventional processes still require significant time and energy. A more efficient approach, the one-pot method, has been developed to overcome these limitations. The one-pot method integrates multiple process steps into a single synthesis step. In the context of nanocellulose synthesis from OPEFB fibers, the one-pot approach combines OPEFB pretreatment, cellulose fiber extraction, and processing into nanocellulose into a single efficient synthesis process (Chávez-Guerrero *et al.*, 2022).

As mentioned, bleaching, delignification, and hydrolysis are performed in the same container using the one-pot method. Five hours of delignification and bleaching with 30% H_2O_2 at 90°C. The hydrolysis procedure then utilized 8%-w H_2SO_4 and 0.22 M $Cr(NO_3)_3$ catalyst at 82°C for one hour. Nanocellulose with a size range of 20-60 nm and a yield of 42%-w is produced. Using $Cr(NO_3)_3$ as a catalyst for cleavage of glycosidic bonds in the cellulose matrix is efficacious. Due to the presence of H_3O^+ and Cr^{3+} ions that can penetrate the amorphous zone, the $Cr(NO_3)_3$ catalyst can also be used to enhance the hydrolysis efficiency of depolymerization of cellulose into nanostructured cellulose (Chen *et al.*, 2017). However, the $Cr(NO_3)_3$ catalyst has disadvantages, including the fact that it is relatively expensive and a heavy metal that is detrimental to the environment due to its toxic, genotoxic, and carcinogenic properties at concentrations between 0.24 and 1.8 g/L.

Another catalyst, $Fe(NO_3)_3$ $9H_2O$, is comparatively inexpensive and common, comprising 5%-w of rock sediments (Fairweather-Tait and Sharp, 2021). Based on the Fenton reaction, Fe and H_2O_2 metals function as oxidizing agents that generate oxidation-reduction reactions that break lignin chains and aid in forming nanocellulose. The nanocellulose produced by Elizabeth *et al.* (2024) using the one-pot method and the $Fe(NO_3)_3.9H_2O$ catalyst revealed that agglomeration was still present. This result is demonstrated by the size of nanocellulose, which ranges from 5-70 nm to 3 µm. Microparticles may happen because the final treatment still involves filtering with filter paper and dehydrating in an oven at 40 to 50°C for 10 hours.

Therefore, a combination of Fe and Cr metals was utilized in this investigation to reduce the cost of the Cr metal catalyst and environmental issues related to that metal

waste. In addition, the definitive treatment of nanocellulose involves separation by centrifugation and dehydration by freeze-drying to reduce agglomeration due to drying by heating. The centrifugation conditions were carried out at a speed of 5000 rpm for 15 minutes, followed by freeze drying at 30-40°C and a pressure of 1 bar for 30 minutes.

MATERIALS AND METHODS

Materials

Oil Palm Empty Fruit Bunch (OPEFB) fibers with a particle size of 200 mesh served as the source of cellulose, which was bought from PT Polytech. Hydrogen peroxide (H_2O_2) 30%, which served as the oxidative agent, was bought from a local chemical mart. Iron (III) nitrate nonahydrate ($Fe(NO_3)_3 \cdot 9H_2O$) and chromium (III) nitrate nonahydrate ($Cr(NO_3)_3 \cdot 9H_2O$) were employed as catalysts and were purchased from Sigma Aldrich. Sulfuric acid (H_2SO_4), used as a strong acid catalyst, was purchased from Merck. The experimental procedures utilized Distilled water as a solvent, diluent, and reaction medium. All chemicals used were of analytical grade and were used without further purification.

Methods

Nanocellulose Preparation by One Pot Method

The pre-treatment process must remove impurities and achieve a uniform size of Oil Palm Empty Fruit Bunch (OPEFB). The pre-treatment stages consisted of the following steps: washing and soaking the OPEFB in distilled water for 30 minutes, drying the OPEFB in an oven at 60°C for 6.5 hours, reducing the size of the dried OPEFB using a grinder, filtering the OPEFB through a sieve shaker to achieve a size range of 230-325 mesh. The outcome of this process was obtaining OPEFB with a size of 230-325 mesh. The scheme of the pre-treatment process is illustrated in Figure 1.

The following process was delignification and bleaching. A sample of 5 grams of OPEFB with a mesh size of 230 was placed into a 350 ml four-neck flask. Subsequently, a 100 mL solution of 30% H_2O_2 was added to the flask. The mixture was heated and stirred at 90°C for 5 hours. Following this, various amounts of the catalysts $Fe(NO_3)_3.9H_2O$ and $Cr(NO_3)_3.9H_2O$ were weighed, and an 8% H_2SO_4 solution was prepared. The catalysts were dissolved in the H_2SO_4 solution and

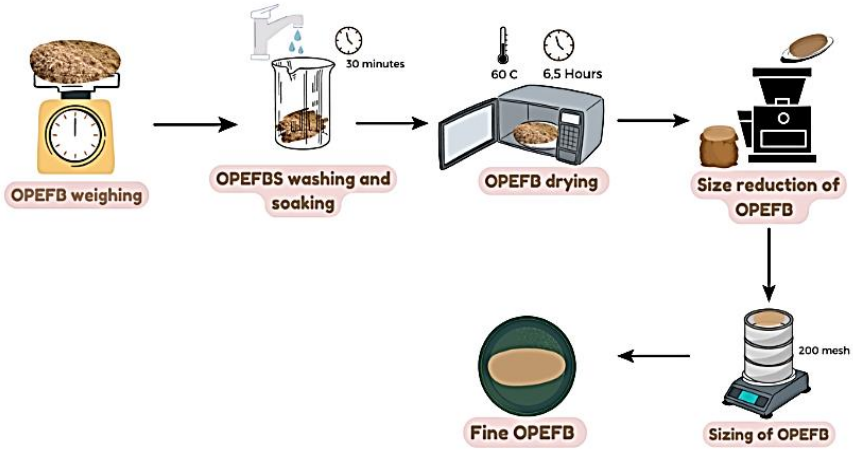


Fig.1: The scheme of the pre-treatment process

added to the same flask. The mixture was then heated at 75°C while stirring at 200 rpm for 1 hour. Afterwards, centrifugation was performed at 5000 rpm and 25°C for 15 minutes. The solution was neutralized to a pH of 7 using distilled water. Furthermore, freeze-drying was conducted. The sample was frozen until the temperature reached -4°C, then dried at 40°C for 30 minutes under a pressure of 1 millibar. The following procedures can be seen in Figure 2.

Experimental Design and Statistical analysis

An experimental design utilizing the Mixed design of Response Surface Methodology (RSM) by Design-Expert® was carried out to evaluate the effect of composition and a combined ratio of catalysts (Frey, 2018). This design is composed of a combination of two catalysts ($Fe(NO_3)_3.9H_2O$ and $Cr(NO_3)_3.9H_2O$), and the composition of the combined catalyst ranges from 3 to 7%-w. The percentages of removed lignin and the amount of nanocellulosic material produced (yield) are the responses that need to be watched and assessed. Catalyst ratios were employed as proportions of the combined catalyst. The total for each mixed attempt was 1, while catalyst proportions ranged from 0 to 1. Table 1 shows the design of this study experiment.

The Design-Expert® was also used to analyze variance (ANOVA) on the lignin removal and nanocellulose yield percentages. The model was determined to be significant at a p-value less than 0.05. Meanwhile residual lack of fit was established at a P-value of more than 0.05. The response was plotted in a 3D surface graph, and the obtained model equations were used in an optimization study to determine the optimal conditions.

Lignin Composition Analysis by NREL

The NREL analysis can measure the amount of lignin in OPEFB before and after the delignification and bleaching processes. The NREL analysis employs two phases (liquid and solid). The liquid phase determines the amounts of lignin that dissolve in acids, whereas the solid phase determines the levels of lignin that are insoluble in acids. Lignin composition was the total amount of both.

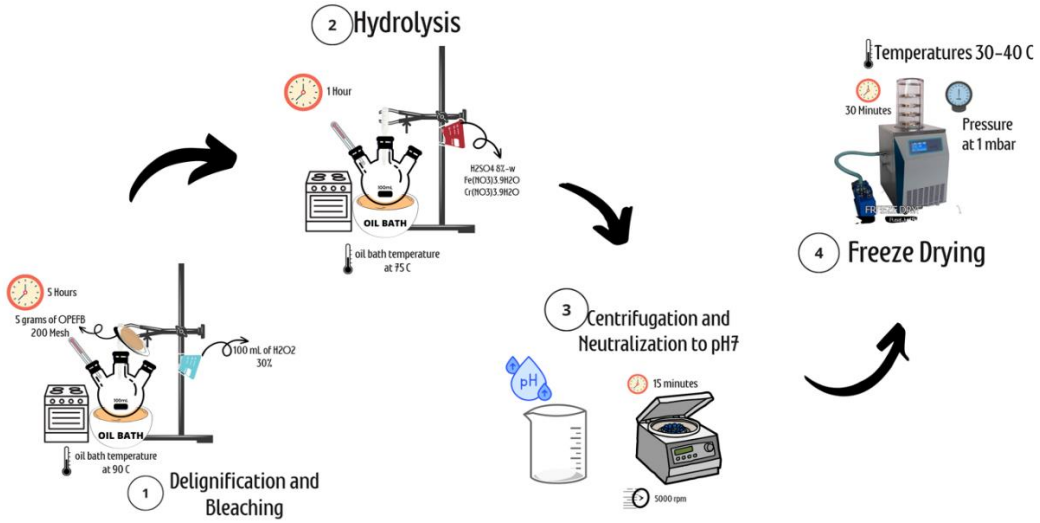


Fig. 2: Nanocellulose synthesis scheme using the One-Pot Method

Table 1. Design of experiment and the result of yield nanocellulose and lignin composition by One Pot Method

Run	Catalyst Rasio (%-b)		Composition of Combination Catalyst to feed (%-b/b)	Yield %-b	Lignin Composition (%)
	Fe	Cr			
Before treatment	0	0	0	-	27.024
1	0	1	7	27.766	3.076
2	1	0	7	5.02	18.886
3	1	0	5	10.076	7.603
4	0.5	0.5	5	12.36	5.304
5	0	1	3	29.616	1.799
6	0.75	0.25	6	11.542	4.315
7	0.5	0.5	7	9.797	2.767
8	1	0	3	11.25	3.403
9	0.5	0.5	3	26.196	1.711
10	1	0	5	9.034	5.512
11	0.25	0.75	4	17.544	2.566
12	0	1	5	37.516	2.87
13	0.5	0.5	5	7.94	5.321
14	0.25	0.75	6	10.306	3.859
15	0.5	0.5	3	12.446	1.716

The gravimetry was used to analyze lignin soluble in acid. In contrast, UV-VIS spectrophotometer analysis, at a wavelength of 205 nm, was used to analyze lignin that is not soluble in acids. The procedure is the same as in our previous research (Elizabeth, 2024).

Transmission Electron Microscope (TEM)

The nanocellulose samples were subjected to transmission electron microscopy (TEM) analysis using an HT7700 instrument. The analysis was conducted at different voltages, with the highest voltage at 120 kV. The measurement was conducted using a solution where distilled water served as the solvent.

X-Ray Diffraction (XRD)

X-Ray Diffraction (XRD) (Bruker D8 Advance) was used to examine the crystallinity of nanocellulose. The sample was examined using Cu K radiation at a voltage of 40 kV and a current of 40 mA. The intensity of the reflections was measured at a scanning range of 10-90 and a scanning speed of 2°/min.

Particle Size Analyzer (PSA)

The Horiba SZ-100 (Z-Type) instrument was used to investigate particle size distribution, employing a scattering angle of 90°. The dispersion medium had a viscosity of 0.897 mPa.s, and the particle distribution was polydisperse.

Fourier Transfer Infra-Red (FTIR)

FTIR (Shimadzu, types of Prestige 21) was used to determine the functional groups of OPEFB that had been carried out in the range of 500–5000 cm-1. A pellet was made by mixing the sample with KBr powder.

RESULTS AND DISCUSSION

Experimental Results

OPEFB was processed through the pre-treatment stage. After drying and milling, it

was filtered to a size of 230-325 mesh. Based on the weight ratio before and after pre-treatment, the OPEFB yield was 23.02%. This process reduced impurities and water content in OPEFB, decreasing yield, as shown in Table 2.

The one-pot method was then used to process the pretreated OPEFB. Table 1 depicts the correlation between nanocellulose yield and lignin composition under various experiment conditions.

Table 2. OPEFB yield during the pre-treatment process

No	Pre-Treatment	Size	Yield (%-b)
1	Initial OPEFB	120 mesh	100.00
2	OPEFB once in the oven	120 mesh	90.48
3	OPEFB after grinding	120-325 mesh	67.46
4	OPEFB after sieving	230-325 mesh	44.44
5	OPEFB after being in the oven again	230-325 mesh	23.02

Effect of Catalyst Concentration and Catalyst Ratio on Nanocellulose Yield

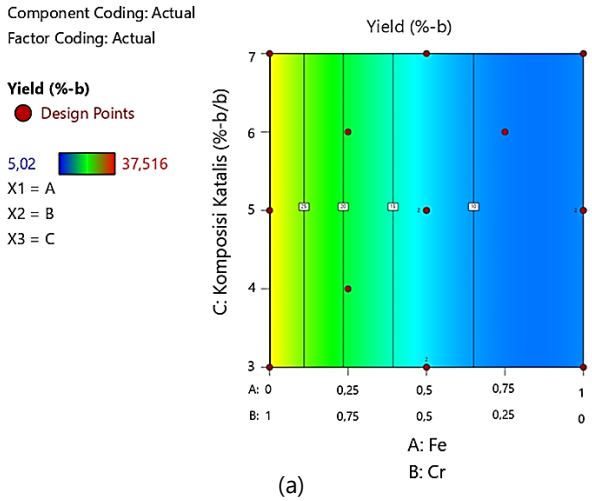
Table 3 shows that the highest yield is achieved at running 12 of 37.516 %-w. The decrease in weight observed in the sample results from the delignification and bleaching procedures, which reduce lignin and hemicellulose content. This reduction in non-cellulosic components enhances the purity of the cellulose. Following the processes of delignification and bleaching, acid hydrolysis is subsequently employed to induce the breakdown of cellulose into nanocellulose, reducing the weight of the sample. Furthermore, the highest nanocellulose yield was achieved while using a catalyst composition of 5% (w/w) with a weight ratio of $Cr(NO_3)_3.9H_2O$: $Fe(NO_3)_39H_2O$, resulting in a nanocellulose yield of 29.616% (w/w).

Table 3 presents the analysis of variance (ANOVA) conducted on the yield of nanocellulose production. The empirical evidence indicates that the many factors involved in the nanocellulose production process significantly impact the resulting nanocellulose yield. The Lack of Fit value in this model is 0.5767, suggesting that the model employed adequately aligns with the experimental data. The experimental findings indicate that the weight ratio of the catalyst

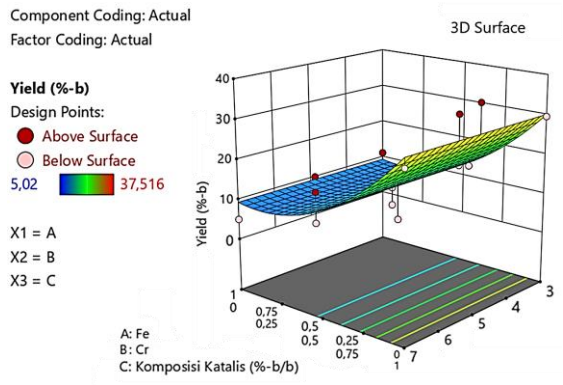
Table 3. Analysis of variance of lignin composition on nanocellulose

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	242.40	8	30.30	15.48	0.0018	Significant
A-Fe	0.5166	1	0.5166	0.2639	0.6258	
B-Cr	0.8752	1	0.8752	0.6065	0.5286	
C- Catalyst Compostion	113.09	1	113.09	57.77	0.0003	
AC	7.18	1	7.18	3.67	0.1039	
BC	46.17	1	46.17	23.59	0.0028	
A2	0.0022	1	0.0022	0.0011	0.9743	
B2	0.0022	1	0.0022	0.0011	0.9743	
C2	23.52	1	23,52	12.01	0.0134	
Residual	11.75	6	1.96			
Lack of Fit	9.56	3	3.19	4.37	0.1284	Not significant
Pure Error	2.19	3	0.7288			
Cor Total	254.15	14				

$Fe(NO_3)_3 9H_2O$ is the primary factor affecting the yield of nanocellulose. A higher weight ratio of the Fe catalyst leads to a decrease in the production of nanocellulose. The statistical analysis supports this conclusion, as the p-value associated with the weight ratio of the Fe catalyst is 0.0057, which is below the predetermined significance level (α) of 0.05. Nevertheless, it is observed that a decrease in the weight ratio of the Fe catalyst leads to an increase in the yield of nanocellulose. In contrast, the impact of the weight ratio of the Cr catalyst and catalyst composition on the nanocellulose yield was negligible.



(a)



(b)

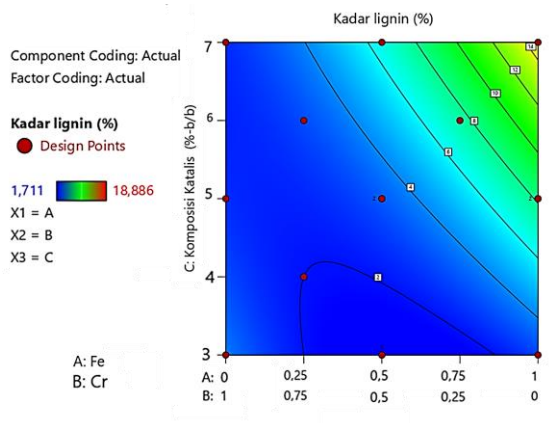
Fig. 3: (a) Contour, (b) Plot surface plot of catalyst composition and catalyst weight ratio of $Fe(NO_3)_3.9H_2O$ and $Cr(NO_3)_3 9H_2O$ to nanocellulose yields.

Figures 3a and 3b show that an increase in the weight ratio of Fe: Cr corresponds to a decrease in the yield value of nanocellulose. The weight ratio of the Fe catalyst influences the yield value of nanocellulose. Based on the findings derived from this investigation, it was seen that using a 3%-w/w catalyst with a weight ratio of Fe: Cr catalyst 0:1 resulted in much higher production of nanocellulose compared to other weight ratios examined. The catalyst composition of Fe: Cr at a ratio of 1:0 produces nanocellulose with a yield of less than 10% by weight. The reduction in nanocellulose production observed in Fe catalysts with excessive weight ratios can be attributed to $Fe(NO_3)_3.9H_2O$. This compound can cleave lignin links and hemicellulose and cellulose bonds, affecting the overall yield of nanocellulose. Additionally, Fe(NO3) in the catalyst may contribute to this decrease in nanocellulose yield. The Fe catalyst caused a Fenton reaction that speeds up the breakdown of lignocellulosic bonds. During hydrolysis, iron triggers hydrogen peroxide decomposition, creating OH radicals. More iron increases radical production, which could speed up the breakdown of lignocellulose, which includes cellulose, lignin and hemicellulose (Wu *et al.*, 2019). Moreover, the Fe Catalyst does not directly interact with OPEFB but with H_2O_2. This contact leads to foaming under operating conditions of 75 °C, which in turn causes the dissolution of OPEFB into the solution. Therefore, the yield of nanocellulose is reduced.

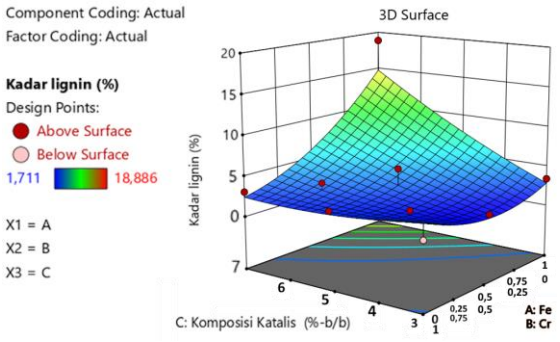
Effect of Catalyst Concentration and Catalyst Ratio $Fe(NO_3)_3.9H_2O$ $Cr(NO_3)_3.9H_2O$ on Lignin Content

Table 1 shows the lowest lignin content was attained in Run 9 with a catalyst composition of 3%-w/w and a ratio of Fe: Cr of 0.5:0.5. The lignin concentration obtained

after treatment was lower than the lignin concentration obtained before the treatment. This decrease in lignin content is because the longer the delignification and bleaching period, the more lignin is degraded, causing the production of more cellulose. By research by Chen *et al.*, 2017, lignin removal occurs predominantly during the delignification and bleaching procedures. In addition, the bleaching agent H_2O_2 would degrade lignin and hemicellulose into water-soluble oligomers, making cellulose simple to obtain because lignin would have been degraded. Anova analysis is also performed for lignin composition response. The result shows the significance of the model, and the lack of fit was established at a p-value < 0.05.



(a)



(b)

Fig. 4: (a) Contour, (b) plot surface plot of catalyst composition and catalyst weight ratio of $Fe(NO_3)_3.9H_2O$ and $Cr(NO_3)_39H_2O$ to nanocellulose lignin contents

Figures 4a and 4.b show that the lowest lignin content is in the blue area with a value of lignin content <2%-w obtained at 3%-w/w catalyst composition with a weight ratio of Fe: Cr catalyst 0.5:0.5, and a catalyst composition of 3%-w/w with a catalyst weight ratio of Fe: Cr 0:1, while for the highest lignin content value, namely > 18%-w, it was obtained in a catalyst composition of 7%-w with a weight ratio of Fe: Cr catalyst is 1:0. This indicates that the composition of the Fe catalyst influences the value of the lignin content, since the higher the composition of the Fe catalyst used, the higher the value of the lignin content produced. This is possible because the catalyst $Cr(NO_3)_3.9H_2O$ can effectively dissolve lignin bonds with a 3% or 7% w/w catalyst composition. This is consistent with the findings of Chen *et al.* (2017), who utilized a $Cr(NO_3)_3.H_2O$ catalyst in which the lignin produced was 2% due to the delignification and bleaching processes degrading the lignin. In addition, the acid hydrolysis process aids in the degradation of the lignin after delignification and bleaching.

Optimization

Based on ANOVA analysis, the optimal operating condition is achieved at a Cr:Fe catalyst weight ratio of 1:0, with a catalyst composition of 3% w/w, resulting in a nanocellulose yield of 30.146% w/w and the lowest lignin content of 1.610% w/w. Compared with experimentation conducted in run 5 using the Cr:Fe catalyst ratio of 0:1 and a 3% w/w catalyst composition, it yielded a nanocellulose yield of 29.616% w/w with a lignin content of 1.799% w/w. The percent error between the model and the experiment is 4.7% and 5.4% for yield and lignin, respectively, as shown in Table 4. This indicates that the model used is satisfactory. In addition, the optimal condition in this

experiment solely involves using the Cr catalyst, excluding the Fe catalyst, allowing the Cr catalyst to penetrate the amorphous zone of cellulose. This penetration facilitates the cleavage of cellulose glycosidic bonds, resulting in easy splitting and formation of nanocellulose, aligning with Chen *et al.*'s research (2017).

Table 4. Percentage error between model and experiment

	Experiment (%-w)	Model (%-w)	Error (%)
Yield	29.616	30.146	4.7
Lignin	1.799	1.610	5.4

Characterization

Fourier-Transform Infrared (FTIR)

The analysis of the FTIR spectrum indicates that the nanocellulose obtained shares similar functional groups with commercial cellulose (microfiber cellulose), as shown in Figure 5. Common peaks include the O-H group at 3200-3550 cm^{-1}, the C-H group at 2840-3000 cm^{-1}, and the C-C group at 1705-1725 cm^{-1}, the predominant cellulose functional group. The presence of -OH groups in this range signifies intramolecular hydrogen bonds, which are crucial in cellulose's long β-glucose chain structure.

Additionally, the -CH stretching (carboxyl) group at 2908.75 cm^{-1} originates from the methyl group and forms the cellulose framework (Ayu *et al.*, 2021). However, there was no detection of the C=O stretching (carbonyl) group in the 1690-1760 cm^{-1} range, indicating a minimal presence of lignin in the tested samples (Chieng *et al.*, 2017; Hidayatulloh *et al.*, 2021).

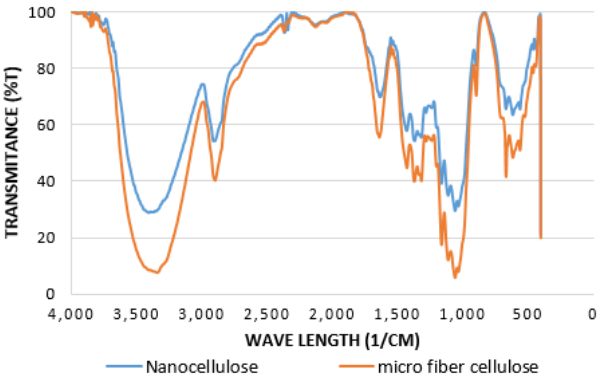


Fig. 5: FTIR spectrum of Sample Run 15

X-Ray Diffraction (XRD) Analysis

Figure 6 shows the X-Ray Diffraction (XRD) analysis of nanocellulose. It revealed a diffractogram with three peaks at 2θ values of 11°, 22°, and 34°.

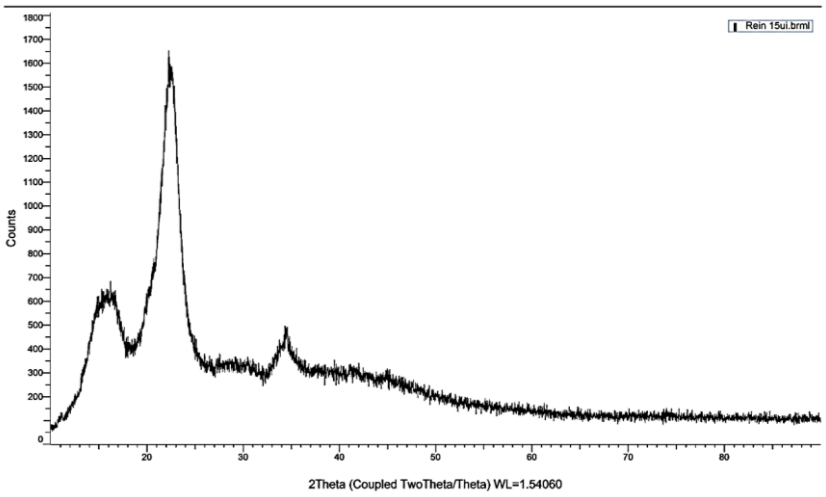
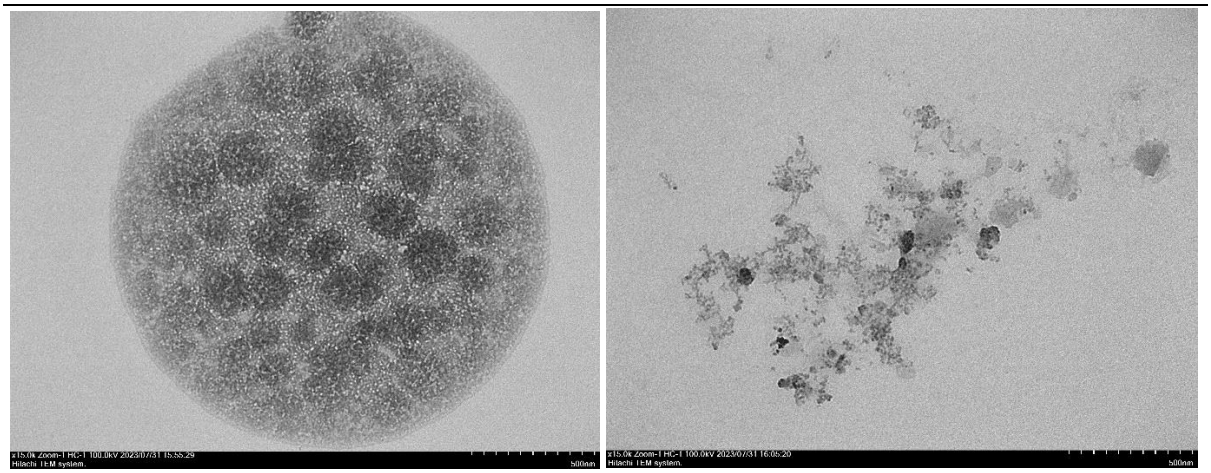
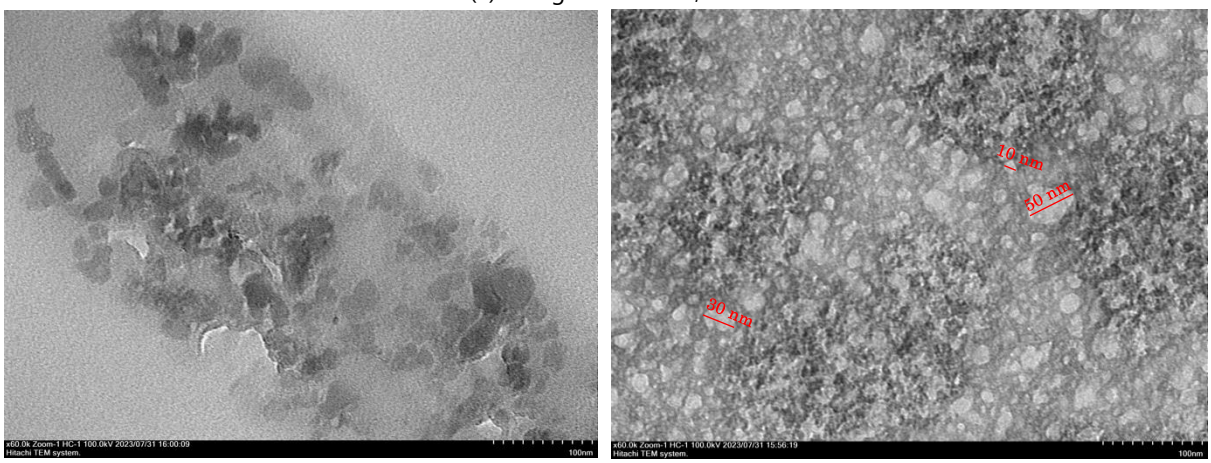


Fig. 6: XRD diffractogram on Sample Run 15



(a) Magnification 15,000x



(b) Magnification 60,000x

Fig. 7: Image of Transmission Electron Microscopy (TEM) analysis for Sample Run 15 with 60,000x

The analysis indicated a crystallinity value of 34.9%, suggesting that the produced nanocellulose remains in the amorphous phase. The crystallinity of cellulose is typically affected by the concentration of acid used during hydrolysis, with higher acid concentrations leading to reduced crystallinity. Excessive acid can damage both amorphous and crystalline cellulose structures. Additionally, the low crystallinity values obtained may be attributed to using Fe as a catalyst, which is a strong oxidizing agent, and the relatively long hydrolysis time. Further research is needed to confirm the influence of these factors. The crystallinity index of nanocellulose is crucial in its isolation process, as it significantly impacts the mechanical strength of nanocomposite applications. High crystallinity nanocellulose is expected to enhance the stiffness of nanocomposite materials.

Transmission Electron Microscope (TEM) Analysis

TEM analyzed the morphology and dimensions of the samples. The results of the analysis are presented in Figure 7. TEM analysis revealed that individual cellulose fibers had sizes below 100 nm. However, there was a tendency for agglomeration among the nanocellulose fibers obtained. This agglomeration could be attributed to inadequate drying of the nanocellulose. Additionally, the morphology of the nanocellulose did not exhibit the typical spherical structure resembling corn grains

commonly found in nanocellulose. This spherical structure is typically associated with nanocellulose with a crystallinity index above 70% (Doan and Chiang, 2022; Wulandari *et al.*, 2016). The absence of this structure suggests that the tested samples contained more amorphous regions than crystalline areas, aligning with the previously discussed low crystallinity index of the nanocellulose.

Particle Size Analyzer (PSA)

The analysis of particle size distribution using PSA reveals that the tested sample has an average diameter of 670.9 nm and a maximum diameter of 1505.6 nm. This finding is consistent with TEM analysis, which indicates agglomeration and nanocellulose diameters exceeding 100 nm.

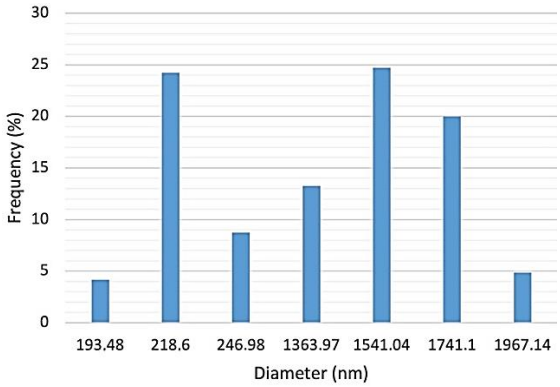


Fig. 8: Particle size distribution in Sample Run 15

The particle size distribution analysis is presented in Figure 8. The results of PSA analysis with a mole ratio of Cr to Fe of 1: 1 show that nanocellulose measuring <300 nm, namely 193.49 nm, 218.6 nm, and 246.98 nm. This shows that the size of nanocellulose has been achieved. This is even supported by TEM analysis in Figure 7, where nanocellulose is already below 100 nm in the 10-50 nm range. Meanwhile, the PSA results show that there are still micro sizes, namely> 300 nm, starting from 1363.97 nm to 1967.14 nm. This can be caused by the low crystallinity of nanocellulose (34.9%), so some are amorphous. This triggers agglomeration, which causes nanocellulose particles to unite and become larger (micro).

CONCLUSION

It was concluded that RSM was effectively optimizes the process parameters to produce nanocellulose. The best yield was achieved without any Fe catalyst. The model gives satisfactory results compared with the experiment data. Moreover, the characteristics of the synthesized nanocellulose are influenced by the catalyst used. The lower the Fe content on the catalyst, the greater the yield value of nanocellulose and the lower the lignin content. The obtained results were a yield content of 29.616%-w and lignin content of 1.799%-w on a catalyst composition of 3%-w/w with a catalyst ratio of Fe: Cr of 0:1. The greater the weight ratio of $Cr(NO_3)_3.9H_2O$ catalyst added, the lower the lignin content and the greater the yield produced in the one-pot method. Conversely, the higher weight ratio of $Fe(NO_3)_39H_2O$ catalyst added, the greater the lignin content and the lower the yield produced in the one-pot method.

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