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Isolation and Characterization of Nanocellulose from Rice Husk *(Oryza sativa L.)* Waste Through Chemical and Ultrasonication Treatment

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Abstract: Rice husk waste is one of the abundant lignocellulosic biomass sources in Indonesia and holds significant potential as a raw material for nanocellulose production. This study aims to isolate nanocellulose from rice husk through a combination of chemical and mechanical treatments to obtain a product with high crystallinity and nanoscale particle size. The isolation process involved several steps: alkali treatment using NaOH to remove hemicellulose, bleaching with H_2O_2 to degrade lignin, and acid hydrolysis using H_2SO_4 to eliminate the amorphous regions of cellulose. The resulting cellulose suspension was then subjected to ultrasonic treatment to further reduce particle size and achieve a more homogeneous dispersion, followed by freeze drying to obtain stable dry nanocellulose powder. Characterization using Fourier Transform Infrared Spectroscopy (FTIR) revealed changes in chemical structure, marked by the disappearance of lignin and hemicellulose peaks and the dominance of hydroxyl (-OH) and C-O functional groups, indicating increased cellulose purity. The nanocellulose yield obtained from this process was 38.15%, calculated based on the dry weight of nanocellulose relative to the initial dry weight of rice husk used. This value falls within the typical range reported for nanocellulose isolation using sulfuric acid hydrolysis and suggests that the applied method was effective in converting rice husk biomass into nanocellulose. The relatively high yield also supports the viability of rice husk as a sustainable and cost-efficient raw material for nanocellulose production. These findings demonstrate that rice husk waste can be effectively converted into high-quality nanocellulose through combined chemical and mechanical approaches, offering a sustainable and value-added solution for various functional materials, biodegradable packaging, and polymer composite applications.

Keywords: Chemical Treatment; Nanocellulose; Rice Husk; Ultrasonication; Yield.

Introduction

Cellulose is the most common natural polymer on Earth and is largely present in plant cell walls. Its primary sources include woody plants such as teak, meranti, belian, and bengkirai, as well as non-woody materials like rice straw, wheat, corn, and various grass fibers [1]. Cellulose is widely utilized as an alternative raw material in various industries, including textiles, construction materials, natural polymer-based products, paper, and pharmaceutical formulations. The increasing use of cellulose is driven by growing industrial demand and the limited availability of raw materials from non-renewable resources [2].

One of the most widely developed cellulose derivatives today is nanocellulose. Nanocellulose is a nano-sized material derived from cellulose, typically with a diameter of 1–100 nm and a length ranging from 500–2000 nm, featuring a high surface area and a large number of hydroxyl groups [3]. These particles offer advantages such as a high aspect ratio, increased crystallinity, large specific surface area, excellent dispersibility, and biodegradability. In addition, nanocellulose has a low density and high chemical reactivity, making it highly promising as a reinforcing agent (filler) in polymer composites [4].

The primary source of nanocellulose is lignocellulosic biomass, one of which is rice husk. Rice husk is an abundant agricultural waste in Indonesia that has not yet been optimally utilized. According to data from Statistics Indonesia (BPS, 2024), national rice production in 2024 reached 53.63 million tons of milled dry grain, generating a large amount of husk waste. Rice husk primarily contains approximately 50% cellulose, 25–30% hemicellulose, and 15–20% lignin, making it a promising raw material for nanocellulose production [5]. However, to obtain high-purity cellulose, the lignin and hemicellulose content must first be removed through a pretreatment process.

The isolation of nanocellulose from rice husk is generally carried out through a combination of chemical and mechanical treatments. The chemical steps include alkalization with NaOH solution to remove hemicellulose, bleaching using H_2O_2 to degrade lignin, and acid hydrolysis with H_2SO_4 to separate the amorphous regions of cellulose [6]. Subsequently, mechanical treatment such as ultrasonication is applied to break down the cellulose fibers to the nanoscale and produce a more homogeneous dispersion [7]. The final step involves freeze drying, which is used to convert the nanocellulose suspension into a stable dry powder ready for various advanced applications [8].

Previous studies on the isolation of nanocellulose from rice husk have mainly employed single-step chemical treatments, such as alkali treatment alone or direct acid hydrolysis, which often result in lower yield

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or incomplete removal of lignin and hemicellulose [9]. Other methods, such as enzymatic hydrolysis or mediated oxidation, have also been reported; however, these approaches require longer reaction times, higher costs, and involve more complex procedures [10]. In this study, the combined NaOH, H_2O_2, H_2SO_4, and ultrasonication method was selected to enhance the efficiency of lignin and hemicellulose removal while producing nanocellulose with higher crystallinity and uniform particle size. Furthermore, freeze drying was applied as the final step to preserve the porous structure and morphology of nanocellulose, which might otherwise collapse under conventional oven drying [11]. This combination of chemical and mechanical processes provides a more practical, cost-effective, and scalable route for nanocellulose production from rice husk compared to other existing methods.

Based on this background, utilizing rice husk as a source of nanocellulose presents a strategic solution to reduce agricultural waste while providing a sustainable raw material for green-material-based industries. Therefore, this study was conducted to isolate nanocellulose from rice husk through a combination of chemical and mechanical methods and to evaluate its characteristics using spectroscopic and crystallinity analyses. The results of this study are expected to support the development of high-value, eco-friendly materials applicable across various sectors.

Research Methods

This research was conducted in the Chemistry Laboratory of Universitas Negeri Surabaya. The study is a laboratory-based experimental research aimed at isolating nanocellulose from rice husk waste using a combination of chemical and mechanical treatments. There are three variables in this study: independent, dependent, and controlled variables. The independent variable is the type of treatment used in the isolation process, which includes alkalization (NaOH), bleaching (H_2O_2), and acid hydrolysis (H_2SO_4). The dependent variable is the quality of the resulting nanocellulose, as evaluated by its degree of crystallinity and functional group structure. The controlled variables include temperature, reaction time, and the concentration of chemical solutions used during the process.

Isolation of Nanocellulose from Rice Husk Waste

Preparation of Rice Husk

Rice husk was collected, washed thoroughly with distilled water to remove dirt and impurities, and then oven-dried at 80°C for 6 hours. The dried husk was ground and sieved using an 80 mesh sieve to obtain fine powder. Subsequently, the obtained powder was processed to nanocellulose through a combination of chemical treatment and mechanical process (ultrasonication) [12].

Alkali Treatment

A total of 50 grams of rice husk powder was mixed with 500 mL of 10% NaOH solution (w/v) and heated at 80°C for 2 hours while being stirred using a magnetic stirrer. This process was repeated twice. The residue was then washed with distilled water until neutral pH was achieved and oven-dried at 50°C for 12 hours.

Bleaching Process

The alkali-treated powder was mixed with 35% hydrogen peroxide (H_2O_2) solution and stirred at 70°C for 12 hours. The resulting sample was washed again, dried at 50°C, and stored at room temperature.

Acid Hydrolysis

Two grams of bleached cellulose were reacted with 100 mL of 40% sulfuric acid (H_2SO_4) at 45°C for 30 minutes under continuous stirring. After hydrolysis, the solution was quenched with cold distilled water and centrifuged at 11,000 rpm for 10 minutes at room temperature. The sediment was repeatedly washed until a neutral pH was reached to obtain a cellulose slurry.

Ultrasonication and Freeze Drying

The obtained cellulose slurry was sonicated in an ultrasonic bath for 30 minutes to further reduce the fiber size. The suspension was then frozen at -40°C for 48 hours and subsequently freeze-dried for another 48 hours to obtain dry nanocellulose powder.

Characterization

The obtained nanocellulose was characterized using FTIR (Fourier Transform Infrared Spectroscopy) to observe functional group changes.

Data Analysis Techniques

The characterization data were processed and analyzed using OriginPro 2018 software. This software was used to generate graphs and analyze FTIR spectra.

Results and Discussion

Isolation of Nanocellulose from Rice Husk Waste

The rice husk used in this study was first washed, dried at 80°C for 6 hours, ground, and sieved using an 80-mesh screen. The isolation of nanocellulose was carried out through a combination of chemical and mechanical methods, including alkalization, bleaching, and acid hydrolysis.

The alkalization step using 10% NaOH aims to remove lignin and hemicellulose without damaging the cellulose structure. This process breaks ether and ester bonds between lignin and cellulose, which is indicated by a darker color change. Next, the bleaching step using 35% H_2O_2 functions to oxidize residual lignin and whiten the cellulose. H_2O_2 was chosen because it is more environmentally friendly and selectively removes lignin without damaging the essential –OH groups in cellulose [13].

The acid hydrolysis process using 40% H_2SO_4 aims to break down the amorphous regions of cellulose while preserving the crystalline regions, resulting in the formation of crystalline nanocellulose (CNC). The chemical reaction produces particles with more uniform and stable sizes [14]. Subsequently, ultrasonication is applied to further reduce particle size and improve the homogeneity of the nanocellulose suspension through cavitation effects [15]. The ultrasonicated suspension is then dried using the freeze drying method for 12 hours to preserve the porous structure of the nanocellulose and obtain dry nanocellulose powder derived from rice husk.

The nanocellulose yield is calculated based on the ratio between the final dry mass of nanocellulose and the initial mass of rice husk used, using the following equation:

$$\text{Yield (\%)} = \frac{\text{dry weight of nanocellulose}}{\text{initial weight of rice husk powder}} \times 100\%$$

Based on the calculation, a yield of 38.15% was obtained, as shown in Table 1.

Table 1. Nanocellulose Yield

Sample	Mass Sample (g)	Yield (%)
Rice husk	50.04	-
Alkali treatment (1)	44.80	89.52
Alkali treatment (2)	40.50	80.91
Bleaching treatment	36.20	72.32
Acid hydrolysis	21.30	42.56
Ultrasonication	20.10	40.15
Freeze dry	19.10	38.15

Based on Table 1, the nanocellulose yield obtained from rice husk reached 38.15%, indicating that the isolation process was fairly efficient. This value falls within the general range of nanocellulose yields. As a comparison, a study on nanocellulose isolation from rice straw using the $NaOH$–H_2O_2– H_2SO_4 method reported a final yield of 37.4%, suggesting that the result obtained in this study can be considered good [16].

The yield is influenced by several factors, such as the type and structure of the biomass, pretreatment conditions (including the concentration and soaking time in NaOH and H_2O_2), as well as the efficiency of the hydrolysis and ultrasonication processes. Nevertheless, this result indicates that rice husk has great potential as a source of nanocellulose with a competitive final yield compared to other widely studied sources.

The success of the nanocellulose purification process from rice husk is reflected not only in the yield but also visually through the color change of the powder. The rice husk powder, originally dark brown, turned yellowish white after undergoing the complete chemical and mechanical treatment stages. This color transformation is shown in Figure 1.

Figure 1 shows the color change of rice husk powder before and after the nanocellulose isolation process. The initial dark brown color reflects the high content of lignin and hemicellulose. After undergoing alkalization, bleaching, and acid hydrolysis, the powder's color changes to yellowish white, indicating a significant removal of non-cellulosic components. This yellowish-white appearance serves as a visual indicator of the successful pretreatment in eliminating lignin and hemicellulose from the lignocellulosic biomass. These visual observations also support the effectiveness of the chemical processes applied in this study [17].

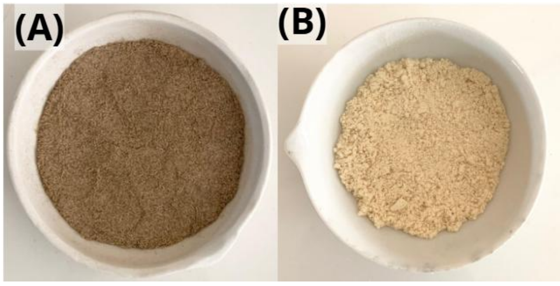

Figure 1. Physical appearance of (A) rice husk powder and (B) nanocellulose from rice husk

FTIR (Fourier-Transform Infrared Spectrometer) Analysis

The infrared (IR) absorption spectrum identifies functional groups based on the observed absorption bands, which reflect the chemical structure of a molecule. The purpose of the FTIR study is to verify that lignin and hemicellulose have been successfully removed during the chemical modification process. In the initial stage, bleaching treatment was used to isolate cellulose in the production of nanocellulose (NC). The FTIR spectra of rice husk and rice husk-derived nanocellulose are shown in Table 2 and Figure 2.

Based on the FTIR spectrum in Figure 2 and the data in Table 2, several characteristic absorption bands indicate the presence of key functional groups in the cellulose structure. A strong absorption band around 3420 cm^{-1} corresponds to O-H stretching from hydroxyl groups, which appears more intense in nanocellulose than in raw rice husk, indicating an increased number of hydroxyl groups due to the removal of lignin and hemicellulose during the isolation process [18]. Furthermore, the band around 2900 cm^{-1}, representing C–H stretching of methyl and methylene groups, is weaker in nanocellulose, suggesting a reduction in alkyl structures. The band at approximately 1635 cm^{-1} indicates the presence of carbonyl (C=O) groups from lignin or aromatic C=C stretching, which diminishes or disappears after bleaching and hydrolysis, indicating that lignin has been significantly removed [19]. In the 1400–1000 cm^{-1} region, absorption bands associated with C–O and C–C stretching in the cellulose polysaccharide structure appear. These bands are sharper and more distinct in nanocellulose, suggesting an increase in cellulose purity and the removal of non-cellulosic components [20-22].

Table 2. FTIR Transmittance Peaks Corresponding to Functional Groups

Functional Groups	Wave Number (cm^{-1})
O-H (hidroxyl)	3420
C-H (aliphatic)	2900
C=O aromatic	1635
C=C aromatic	1515
C-O-C	1205
C-O (glycosidic bond)	1050-1030

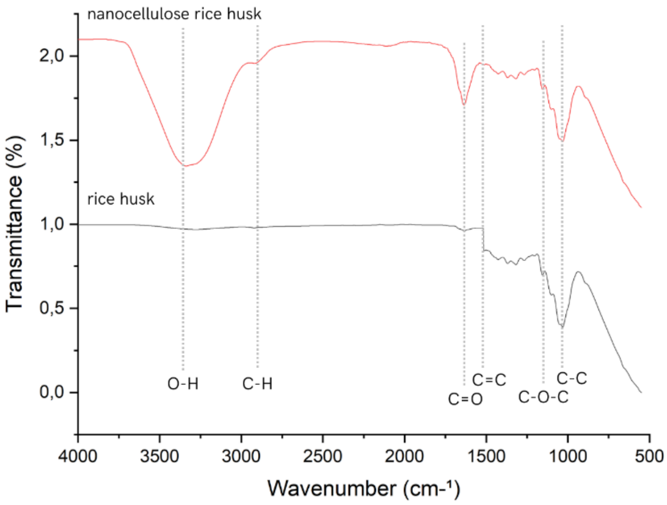

Figure 2. FTIR Spectrum

Overall, the changes in intensity and position of the absorption bands between the rice husk and nanocellulose samples support the success of the isolation process, particularly in the removal of lignin and hemicellulose and the enrichment of pure cellulose content. The main finding of this study is that nanocellulose can be successfully isolated from rice husk waste with a yield of 38.15% through a combination of chemical and mechanical treatments. FTIR analysis confirmed the effective removal of hemicellulose and lignin, resulting in higher cellulose purity. Compared to other lignocellulosic sources, rice husk offers several advantages, including its abundance, low cost, and limited prior utilization, making it a practical and sustainable raw material for nanocellulose production. In addition to yield and FTIR confirmation, further characterization, such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and particle size distribution analysis could provide a deeper understanding of the morphology and crystallinity of the isolated nanocellulose. Such studies would strengthen the conclusions regarding the structural integrity and nanoscale properties of the obtained material.

Conclusion

This study demonstrates that rice husk waste can be effectively utilized as a raw material for nanocellulose production through a combination of chemical treatments (alkalization, bleaching, and acid hydrolysis) and mechanical treatment (ultrasonication). The isolation process yielded nanocellulose with a yield of 38.15%. FTIR analysis confirmed the successful removal of most hemicellulose and lignin. These findings highlight the great potential of rice husk as an eco-friendly source of nanocellulose for use in various biopolymer-based material applications. These findings highlight the potential of rice husk as an eco-friendly source of nanocellulose for various biopolymer-based material applications. Future studies are recommended to explore the morphology, crystallinity, and thermal stability of the isolated nanocellulose, as well as its application in biopolymer composites and biodegradable packaging, to provide broader insights into its functionality and industrial relevance.

Author's Contribution

Viona Witya Paramitha: Collecting research data and preparing articles; and Dina Kartika Maharani: Responsible person and article compiler.

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References

[1] P. Y. Nor, Sunardi, and I. Utami, "Synthesis and characterization of alginate-based bioplastic with the addition of nanocellulose from sago frond as filler," *Jurnal Sains dan Teknologi*, vol. 4, no. 1, pp. 30–39, 2021.

[2] I. Mulyadi, "Isolasi dan karakteristik selulosa," *Jurnal Saintika Unpam*, vol. 1, no. 2, pp. 177–180, 2019.

[3] N. E. Fitriana, A. Suwanto, T. H. Jatmiko, S. Mursiti, and D. J. Prasetyo, "Cellulose extraction from sugar palm (Arenga pinnata) fibre by alkaline and peroxide treatments," in *IOP Conf. Ser.: Earth Environ. Sci.*, vol. 462, no. 1, p. 012053, 2020, doi: 10.1088/1755-1315/462/1/012053.

[4] A. Hayatun, M. Jannah, A. Ahmad, and P. Taba, "Synthetic bioplastic film from rice husk cellulose," in *J. Phys.: Conf. Ser.*, vol. 1463, no. 1, p. 012009, 2020, doi: 10.1088/1742-6596/1463/1/012009.

[5] A. R. Al Mofty, M. A. Wahid, and G. El Sayed, "Isolation and characterization of nanocellulose from rice husk waste: Physicochemical properties and potential applications," *Journal of Biopolymer Research*, vol. 17, no. 3, 2023.

[6] R. Isroi, "Isolasi nanoselulosa dari sekam padi menggunakan metode alkalisasi, bleaching, dan hidrolisis asam," *Jurnal Teknologi Polimer & Biokomposit*, vol. 4, no. 2, pp. 85-94, 2016.
[7] S. Habibi and M. Jamshidi, "Synthesis of TiO_2 nanoparticles coated on cellulose nanofibers with different morphologies: Effect of the template and sol-gel parameters," *Mater. Sci. Semicond. Process.*, vol. 109, p. 104927, 2020, doi: 10.1016/j.mssp.2020.104927.
[8] M. Waje and P. Kumar, "Preparation of cellulose nanocrystals from rice husk using ultrasonic-assisted acid hydrolysis," *Carbohydr. Polym.*, vol. 156, pp. 435-442, 2017.
[9] K. Vasić, M. Dokl, Ž. Knez, and M. Leitgeb, "Isolation and characterization of nanocellulose from *Polypodiophyta* fern using chemo-mechanical method," *Biomimetics*, vol. 9, no. 10, p. 624, Oct. 2024, doi: 10.3390/biomimetics9100624.
[10] P. Phanthong, P. Reubroycharoen, X. Hao, G. Xu, A. Abudula, and G. Guan, "Nanocellulose: Extraction and application," *Carbon Resour. Convers.*, vol. 1, no. 1, pp. 32-43, 2018, doi: 10.1016/j.crcon.2018.05.004.
[11] F. Baraka, K. Ganesan, B. Milow, et al., "Cellulose nanofiber aerogels: Effect of the composition and the drying method," *Cellulose*, vol. 31, pp. 9699–9713, 2024, doi: 10.1007/s10570-024-06191-2.
[12] N. Samsalee, J. Meerasri, and R. Sothornvit, "Rice husk nanocellulose: Extraction by high-pressure homogenization, chemical treatments and characterization," *Ind. Crops Prod.*, vol. 201, p. 116939, 2023, doi: 10.1016/j.indcrop.2023.116939.
[13] Z. Amrillah, F. F. Hanum, and A. Rahayu, "Studi efektivitas metode ekstraksi selulosa dari agricultural waste," in *Seminar Nasional Penelitian LPPM UMJ*, vol. 8, 2022. [Online]. Available: http://jurnal.umj.ac.id/index.php/semnaslit
[14] L. Zhang and Z. Zhang, "Preparation and characterization of cellulose nanocrystals from different sources using sulfuric acid hydrolysis," *Bioresour. Technol.*, vol. 201, pp. 122–129, 2016.
[15] A. B. D. Nandiyanto, "Cost analysis and economic evaluation for the fabrication of activated carbon and silica particles from rice straw waste," *J. Eng. Sci. Technol.*, vol. 13, no. 6, pp. 1523–1539, 2018.
[16] K. R. Ningtyas, M. Muslihudin, and I. N. Sari, "Sintesis nanoselulosa dari limbah hasil pertanian dengan menggunakan variasi konsentrasi asam," *J. Penelitian Pertanian Terapan*, vol. 20, no. 2, pp. 142–147, 2020.
[17] A. S. Zulkifli, N. A. Abd Halim, S. Ibrahim, N. Hamzah, and S. H. Saleh, "Effect of sulphuric acid concentration on nanocellulose extraction from rice husk," *Malays. J. Chem.*, vol. 26, no. 5, pp. 562–571, 2024. [Special Issue: 4th IKMPB Online Symposium 2024 – Development of Chemistry to Accomplish a Sustainable World].
[18] X. Feng, Y. Li, and Z. Chen, "Fourier-transform infrared analysis of nanocellulose from bamboo pulp," *Carbohydr. Polym.*, vol. 255, p. 117482, 2021.
[19] M. A. Pradana, H. Ardhyananta, and M. Farid, "Pemisahan Selulosa dari Lignin Serat Tandan Kosong Kelapa Sawit dengan Proses Alkalisasi untuk Penguat Bahan Komposit Penyerap Suara," *Jurnal Teknik ITS*, 2017.
[20] F. Widyawati, S. Hidayat, A. Wiradana, A. K. Setyaningtyas, S. Bahtiar, and E. Yanuar, "Synthesis of Cellulose from Decorticated Sisal Plants (Agave sisalana) using the Acid Hydrolysis Method," *Jurnal Pijar Mipa*, vol. 20, no. 3, pp. 559–563, 2025, doi: 10.29303/jpm.v20i3.8099
[21] S. Widyawati, A. Hidayat, A. K. Wiradana, S. Setyaningtyas, S. Bahtiar, and E. Yanuar, "Synthesis of cellulose from decorticated sisal plants (Agave sisalana) using the acid hydrolysis method," *J. Pijar MIPA*, vol. 20, no. 3, pp. 559–563, May 2025, doi: 10.29303/jpm.v20i3.8099.
[22] M. R. Afifi, Z. Aini, T. T. Irawadi, and H. Purwaningsih, "Isolation and Characterization of Cellulose Whiskers from Lampung Sugarcane Bagasse, Indonesia," *Jurnal Pijar Mipa*, vol. 20, no. 5, pp. 870–875, 2025, doi: 10.29303/jpm.v20i5.9371