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Utilization of Empty Oil Palm Fruit Bunches as a Source of Nanocrystalline Cellulose through the Acetic Acid Hydrolysis Method

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Abstract

This study investigates the production of nanocrystalline cellulose (CNC) derived from oil palm empty fruit bunches (OPEFB) using acetic acid hydrolysis as a greener alternative to conventional mineral-acid-based hydrolysis. The effects of acetic acid concentration (2–10%), hydrolysis time (2–6 h), and temperature (160–200°C) were examined regarding CNC yield, particle size, zeta potential, chemical structure, crystallinity, and morphology. The result shows the CNC yield increased from 78.17% to 87.39% at 8% acid concentration, then decreased at 10% due to over-hydrolysis. With the variation of acetic acid concentration from 2 to 10%, the particle size was reduced from 1200 nm to 639 nm, while zeta potential values ranged from –18.62 to –22.80 mV indicating moderate to good colloidal stability. Fourier Transform Infra-Red (FTIR) confirmed the removal of lignin and hemicellulose. XRD analysis showed sharper peaks at 2θ ≈ 22.6°, signifying increased crystallinity, and SEM revealed rod-like CNC structures with smoother surfaces at higher acid concentrations. The results demonstrate that OPEFB is a promising raw material for environmentally friendly CNC production, with optimal performance obtained at 8–10% acetic acid.

Keywords: *Acetic Acid Hydrolysis, Cellulose Nanocrystals, Empty Fruit Bunches, Palm Oil Waste, XRD, SEM.*

1. Introduction

Indonesia is the world's leading producer of crude palm oil, generating more than 40 million tons of palm oil annually. Alongside this production, large quantities of lignocellulosic residues are generated, with oil palm empty fruit bunches (OPEFB) representing approximately 22–24% of the total weight of fresh fruit bunches processed in mills [1][2]. OPEFB is recognized as one of the most abundant biomasses wastes in Indonesia and contains a high cellulose fraction (~46%), making it a promising renewable feedstock for the synthesis of value-added bioproducts, including nanocellulose.

Nanocrystalline cellulose (CNC), also known as cellulose nanocrystals, is a rod-shaped, highly crystalline nanomaterial extracted from natural cellulose. CNC exhibits exceptional mechanical strength, high surface area, biodegradability, non-toxicity, and strong reinforcement capability in composite systems [3][4]. As a result, CNC has gained wide application in polymer nanocomposites, biomedical delivery systems, barrier coatings, electronics, and membrane technology. With Indonesia's abundant biomass resources, the development of CNC aligns well with national strategies for sustainable materials development and circular bioeconomy.

Conventional CNC extraction typically uses strong mineral acids such as sulfuric acid (H_2SO_4) or hydrochloric acid (HCl), which selectively hydrolyze amorphous cellulose regions to liberate crystalline nanofibers [5]. Although effective, these acids present several drawbacks, including corrosive waste generation, equipment degradation, and, in the case of sulfuric acid, reduced CNC thermal stability

due to the formation of sulfate ester groups [6][7]. These limitations have encouraged the exploration of greener hydrolysis alternatives based on organic acids, which offer lower environmental impact, easier handling, and reduced corrosivity.

Acetic acid is a promising candidate for green hydrolysis due to its mild acidity, biodegradability, and ability to partially hydrolyze lignocellulosic structures [8]. However, compared with mineral acids, the use of acetic acid for CNC production remains significantly underexplored, especially for OPEFB biomass. Existing studies primarily focus on mineral-acid-based CNC, sonication-hydrothermal treatments, or oxidative pathways such as TEMPO-mediated oxidation [9][10]. Systematic investigations evaluating the combined effects of acetic acid concentration, hydrolysis temperature, and reaction duration on CNC structure and surface characteristics are limited.

Based on this gap, the present study aims to investigate the synthesis of CNC from OPEFB using acetic acid hydrolysis as an environmentally friendly alternative, while evaluating its efficiency and structural performance in comparison with conventional methods. The novelty of this research lies in (1) the application of acetic acid under controlled high-temperature autoclave conditions, (2) the integration of multiple characterization techniques—FTIR, PSA, Zeta Potential, XRD, and SEM—to comprehensively assess CNC quality, and (3) the use of local biomass waste (OPEFB) to support sustainable material development in Indonesia. The objectives of this study are to determine the influence of acetic acid concentration, reaction temperature, and hydrolysis duration on CNC yield and physicochemical properties and to evaluate the structural, chemical, and morphological characteristics of CNC produced under these conditions.

2. Literature Review

OPEFB is a lignocellulosic material containing cellulose (≈46%), hemicellulose (≈23%), and lignin (≈16%). Pretreatment involving delignification and bleaching is essential to remove non-cellulosic components before acid hydrolysis. Previous studies using hydrochloric or sulfuric acid on OPEFB have achieved CNC yields of 60–75%, with particle diameters between 50–200 nm and crystallinity indices up to 80%.

However, limited studies have explored organic acid hydrolysis. Li et al. reported that formic acid hydrolysis followed by TEMPO oxidation could produce CNC with improved environmental performance [8]. Beck-Candanedo et al. [5] and Trache et al. [4] emphasized that controlling acid concentration and temperature is crucial to prevent over-hydrolysis and maintain nanocrystal integrity.

Table 1. Previous Studies of Organic Acid Hydrolysis

No	Authors / Year	Hydrolysis Method	Acid Concentration / Conditions	Time & Temperature	Main CNC Results
1	Zulnazri et al., 2024 [9]	HCl + Hydrothermal	HCl 3 M	1 h	Yield 74.82%; CrI 78.59%; Tmax 339.8°C
2	Hastuti et al., 2018 [6]	HCl Hydrolysis	Various HCl levels	≈3 h; 70–80°C	Aspect ratio 23–29; Tmax 347–359°C
3	Hidayatulloh et al., 2021 [7]	H_2SO_4 Hydrolysis	64% H_2SO_4	45 min; 40–50°C	Diameter 86–109 nm
4	Mohamad Haafiz et al., 2013 [11]	H_2SO_4 Hydrolysis	40–60% H_2SO_4	60 min; 40–50°C	Yield ≈ 62%
5	Soetaredjo et al., 2022 [12]	Low-Acid H_2SO_4	~30% H_2SO_4	<80°C	Diameter 1.66 nm; length ≈ 9.85 nm
6	Dyah Kencana Wulan et al., 2024 [10]	H_2SO_4 / TEMPO / HCl	Low-acid hydrolysis	30–180 min; 40–80°C	Diameter 2–20 nm; length 100–500 nm
7	Li et al., 2015 [8]	Formic Acid + TEMPO	Formic acid hydrolysis	—	High crystallinity CNC

3. Methods

The raw material used in this study was oil palm empty fruit bunch (OPEFB) powder with a particle size of 100 mesh, obtained from PT Polytech Indonesia, Indonesia. All chemicals employed were analytical grade, including sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), and acetic acid (CH_3COOH), purchased from Sigma Aldrich to ensure high purity during pretreatment and hydrolysis. Distilled water was used in every washing step to avoid inorganic contaminants that could interfere with cellulose purity. The pretreatment process consisted of two main steps: delignification and bleaching. In the delignification stage, 250 g of OPEFB powder were treated with 5% NaOH solution at 100°C for 2 hours under atmospheric pressure to remove lignin, hemicellulose, waxes, and extractive components. This alkaline treatment disrupts ester–ether linkages between lignin and carbohydrates, enhancing cellulose accessibility. The residue was washed repeatedly until pH became neutral. The bleaching process was then conducted using a mixture of 1% H_2O_2 and 1% acetic acid at 90°C for 1.5 hours at pH 9. Hydrogen peroxide acted as an oxidative bleaching agent to eliminate the remaining lignin chromophores, while acetic acid provided buffering capacity to support the bleaching reaction, resulting in purified α-cellulose with a whiter appearance and reduced amorphous impurities.

The acid hydrolysis stage aimed to convert α-cellulose into nanocrystalline cellulose (CNC) by selectively cleaving the amorphous regions while preserving the crystalline microfibril domains. Five grams of bleached α-cellulose were hydrolyzed with 50 mL of acetic acid at concentrations of 2%, 4%, 6%, 8%, and 10% (v/v). Hydrolysis was conducted inside a sealed stainless-steel autoclave reactor, where the base temperature of 160°C was subsequently varied to higher levels, and the initial reaction duration of 2 hours was also varied to assess

the impact of hydrolysis severity on cellulose depolymerization, crystallinity development, and CNC particle size formation. These variations were implemented to systematically examine how increasing thermal and temporal intensity influences the breakdown of amorphous cellulose and the exposure of crystalline nanofibrils. After hydrolysis, the suspension was cooled to room temperature and washed with distilled water before centrifugation at 1500 rpm for 15 minutes to remove excess acid and solubilized fragments. The partially hydrolyzed suspension was then subjected to ultrasonic treatment using a probe sonicator for 30 minutes to break residual agglomerates, reduce microfibril bundling, and enhance CNC dispersion. The resulting suspension was neutralized to pH 7 to prevent acid-induced degradation and freeze-dried to obtain CNC in powder form.

The final CNC products were comprehensively characterized. Particle size distribution and zeta potential were measured using dynamic light scattering (DLS) to determine hydrodynamic diameter and colloidal stability. Fourier-transform infrared spectroscopy (FTIR) was employed to identify functional groups and confirm lignin and hemicellulose removal. X-ray diffraction (XRD) analysis was conducted to assess crystallinity and verify the presence of cellulose Iβ peaks. Scanning electron microscopy (SEM) was used to visualize morphological changes, including fibril shape, rod-like crystal formation, and surface texture after hydrolysis.

4. Result and Discussion

The production of nanocrystalline cellulose (CNC) from OPEFB through acetic acid hydrolysis displayed a strong dependence on variations in acid concentration, reaction temperature, and hydrolysis duration. CNC yield increased steadily from 78.17% at 2% acetic acid to 87.39% at 8%, reflecting efficient penetration of acetic acid into amorphous cellulose domains. This behavior aligns with the well-established mechanism in which amorphous cellulose is selectively hydrolyzed, while crystalline regions remain intact [5]. However, when the acid concentration reached 10%, the yield declined to 83.02%, indicating over-hydrolysis, where prolonged or severe acid exposure begins degrading crystalline segments—a phenomenon also reported in other organic and mineral-acid hydrolysis studies [7][8].

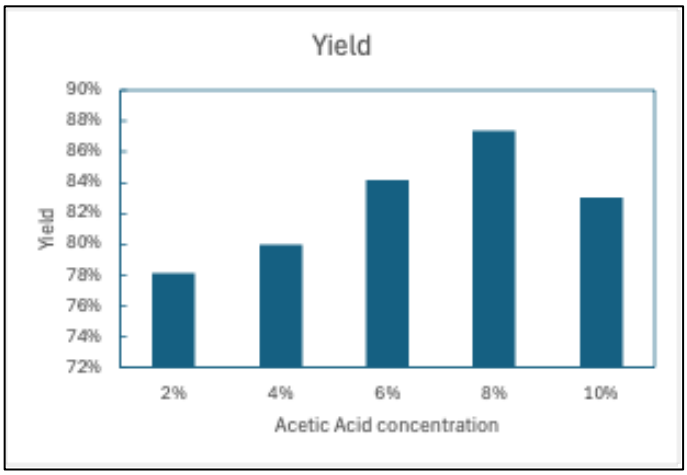

Fig 1. CNC yield at various acetic acid concentrations (2–10%).

Particle size analysis (PSA) demonstrated substantial reductions as acid concentration increased. At lower concentrations (2–4%), CNC particles exhibited relatively large sizes (≈1200–900 nm), indicating incomplete fibrillation and the presence of residual fiber bundles. This is consistent with previous observations that mild hydrolysis is insufficient to fully isolate nanocrystals from cellulose microfibrils [3][4]. As the acid concentration increased to 6–8%, particle size decreased dramatically to 639 nm, suggesting more effective cleavage of glycosidic linkages and enhanced liberation of crystalline nanodomains. A slight increase to 654 nm at 10% acid may be attributed to re-agglomeration of excessively fragmented nanocrystals, a well-documented effect in CNC suspensions subjected to high hydrolysis severity. Similar agglomeration behavior was also reported in CNC derived from OPEFB via sonication–hydrothermal methods [9].

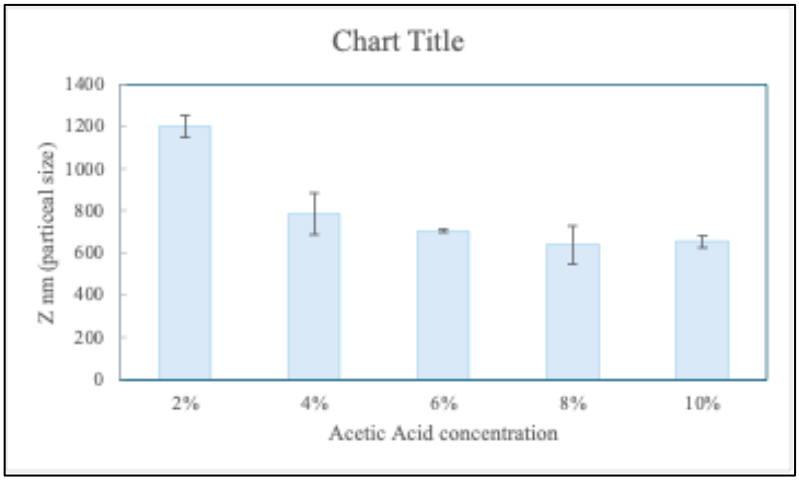

Fig 2. Particle size distribution of CNC at different acetic acid concentrations.

The zeta potential values ranged from –18.62 mV to –22.80 mV, indicating moderate to good colloidal stability. More negative values at higher acid concentrations may be attributed to increased exposure of carboxyl and hydroxyl groups after partial depolymerization. Although acetic acid does not generate sulfate ester groups like sulfuric acid hydrolysis, its hydrolytic fragmentation can still increase negative surface charge, yielding CNC with acceptable dispersion stability[6][13]. The stability values observed in this study fall within typical ranges for CNC produced using organic acids, supporting the suitability of acetic acid as a green hydrolysis agent.

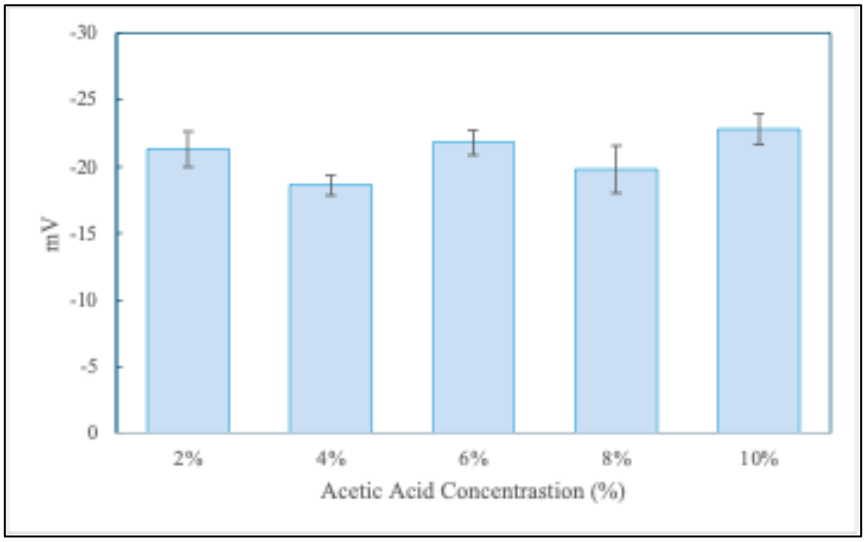

Fig 3. Zeta potential values of CNC as influenced by acetic acid concentration.

FTIR analysis confirmed the successful removal of lignin and hemicellulose during pretreatment. The disappearance of the carbonyl band at 1735 cm^{-1}, associated with hemicellulose ester bonds, and the reduction of aromatic lignin peaks around 1505 cm^{-1} indicate effective delignification [14]. The presence of characteristic cellulose peaks—such as the broad O–H stretching at 3330 cm^{-1}, C–H stretching at 2890 cm^{-1}, and the glycosidic C–O–C vibrations between 1052–1161 cm^{-1}—further supports successful purification [3][12]. The persistence of a weak band near 896 cm^{-1} indicates residual amorphous cellulose, which is typical in CNC structures even after optimized hydrolysis [4].

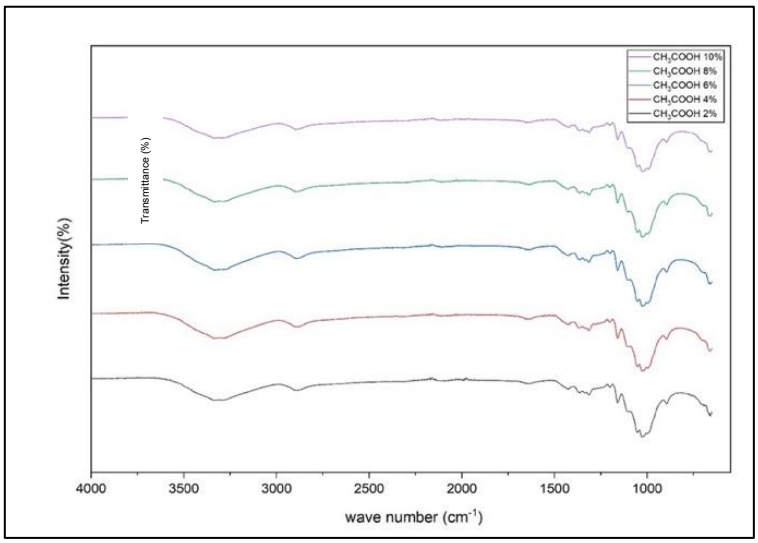

Fig 4. FTIR spectra of cellulose before and after acetic acid hydrolysis.

XRD patterns revealed dominant diffraction peaks at $2\theta \approx 16°$ and 22.6°, corresponding to the (110) and (200) planes of cellulose Iβ. These peaks are hallmarks of native cellulose and indicate that acetic acid hydrolysis preserved the cellulose Iβ polymorph [7]. The sharpness of the 22.6° peak increased with increasing acid concentration, reflecting enhanced crystallinity due to the preferential removal of amorphous domains. This finding is consistent with trends observed in CNC derived using HCl hydrolysis [6] and organic acid treatments [8]. The diminishing amorphous halo at approximately 18° confirms that acetic acid effectively reduces disordered cellulose content, resulting in higher crystalline purity. Importantly, no shift toward peaks characteristic of cellulose II was observed, indicating the absence of polymorphic transformation typically associated with alkaline mercerization [4].

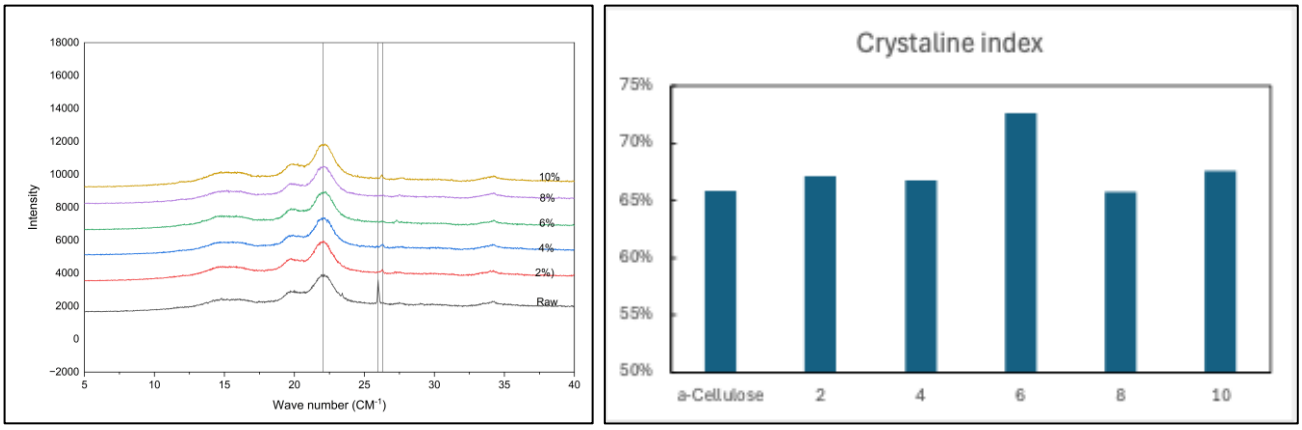

Fig 5. XRD diffraction patterns of CNC at different acetic acid concentrations showing cellulose Iβ structure.

SEM images visually confirmed the structural changes inferred from PSA and FTIR. At low acid concentrations, the samples showed irregular, coarse fibrillar aggregates, illustrating incomplete separation of cellulose bundles. Similar morphologies have been reported in early-stage hydrolysis of OPEFB cellulose [12]. As the acid concentration increased to 6–10%, the morphology transitioned to rod-like nanocrystals with smoother surfaces, consistent with the typical appearance of CNC produced through controlled hydrolysis [3][9]. The improved uniformity and reduced dimensions at higher acid concentrations further confirm CNC formation and support the optimal hydrolysis condition at 8–10%.

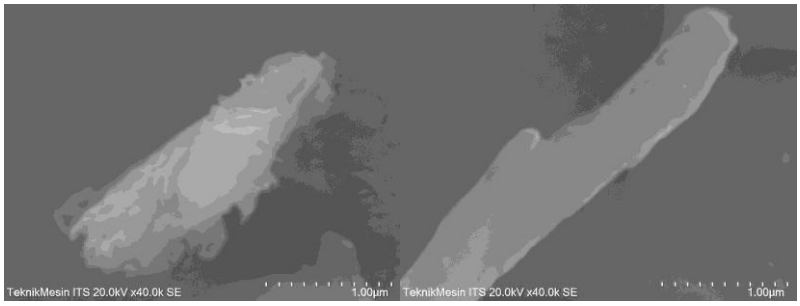

Fig 6. SEM micrographs of CNC showing morphological transformation at increasing acetic acid concentration.

Overall, the combined results from yield, particle size, zeta potential, FTIR, XRD, and SEM demonstrate that acetic acid hydrolysis is capable of producing CNC with competitive structural integrity compared to conventional mineral acid methods. The optimal condition was achieved at 8% acetic acid, where high crystallinity, small particle size, good dispersion stability, and rod-like morphology were obtained. These findings show that OPEFB is a promising feedstock for sustainable CNC production and highlight acetic acid as a viable green solvent for nanocellulose synthesis.

5. Conclusion

This study successfully demonstrated the production of nanocrystalline cellulose (CNC) from oil palm empty fruit bunches (OPEFB) using acetic acid hydrolysis as an environmentally friendly alternative to conventional mineral-acid methods. The results showed that acetic acid concentration strongly influenced CNC yield, particle size, crystallinity, and morphological transitions. The highest performance was obtained at 8% acetic acid, which provided the highest CNC yield (87.39%), significant particle size reduction, moderate-to-good dispersion stability, confirmed lignin–hemicellulose removal, and the emergence of rod-like crystalline structures. XRD analysis revealed sharper diffraction peaks characteristic of cellulose Iβ, indicating enhanced crystallinity under optimal hydrolysis conditions.

Although the hydrolysis process produced material with nanoscale characteristics, particularly in terms of fibril morphology and crystalline domain formation, the particle size distribution obtained in this work indicates that the resulting CNC has not yet fully reached the optimal nanometer scale expected for highly uniform CNC suspensions. This suggests that the hydrolysis conditions used—especially acid concentration, temperature, and time—have not completely optimized the breakdown of the amorphous matrix, and further refinement is still required to achieve consistently smaller and more homogeneous CNC nanoparticles.

Therefore, future research should explore a broader range of variables to enhance CNC quality, including the use of higher temperatures, extended reaction durations, controlled pressure variations, sequential or combined acid treatments, and more advanced mechanical disintegration techniques such as ultrasonication optimization, high-shear homogenization, or microfluidization. Additional parameters such as solid-to-liquid ratio, catalyst addition, and post-treatment fractionation may also significantly influence CNC uniformity. These

improvements are expected to produce CNC with smaller particle sizes, narrower distribution ranges, and properties that better match industrial and high-performance material applications.

Acknowledgement

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