Characterization of Hydrochloric Acid Hydrolysis to Sawdust Waste for Nanocellulose Extraction

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ABSTRACT

Sawdust waste (SDW) is an abundant lignocellulosic residue that can serve as a precursor for cellulose-based nanomaterials. This study investigated the effects of hydrochloric acid (HCl) concentration and hydrolysis duration on the structural and morphological characteristics of cellulose-rich material obtained from sawdust waste. A two-stage treatment consisting of alkaline pretreatment followed by HCl hydrolysis was employed. Sawdust was first treated with 5 wt.% NaOH at 80 °C for 2 h, followed by acid hydrolysis using 60 and 65 wt.% HCl for 30 and 60 min at 45 °C. The resulting materials were characterized using X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The crystallinity index increased from 51% for untreated sawdust to 68% after alkaline treatment and reached 66–71% after acid hydrolysis. The Scherrer-derived crystallite size of the hydrolyzed samples ranged from 2.27 to 2.85 nm. The highest crystallinity index of 71% was obtained for both 60 wt.% HCl for 30 min and 65 wt.% HCl for 60 min. FTIR spectra indicated the retention of characteristic cellulose functional groups following treatment, while SEM showed changes in surface morphology and some particle aggregation after hydrolysis. Because the experimental design consisted of only four treatment combinations without replication, the results are considered a preliminary assessment of the effects of acid concentration and hydrolysis time rather than a statistically validated optimization. Further work involving replicated experiments, quantitative yield and purity measurements, and direct nanoscale dimensional analysis is required to establish the suitability of the material for specific applications.

Keywords: Sawdust waste, Nanocellulose, Acid hydrolysis, Hydrochloric acid, Crystallinity index, Sustainable materials

INTRODUCTION

The global transition toward a circular economy has highlight the urgent need for sustainable, bio-based alternatives to synthetic materials [1]. Nanotechnology has come as a one of the cornerstones of this transition, offering reliable materials like nanocellulose that address critical needs in energy, environmental remediation, and structural engineering [2]. Derived from abundant biomass, nanocellulose provides a biodegradable and mechanically robust alternative to the waste management issues for example a petroleum-based plastics that currently dominate industrial landscapes. However, the scalability of nanocellulose production remains constrained by energy-intensive extraction processes and the difficulty of isolating high-purity cellulose from lignocellulosic matrices without compromising its inherent crystalline structure.

Sawdust waste, an abundant byproduct of the timber industry, especially in asean regions, represents a

compelling but technically challenging feedstock for nanocellulose synthesis [3]. Current extraction methodologies often suffer from inconsistent quality, primarily due to the recalcitrant nature of lignin and hemicellulose. Achieving high-performance nanocellulose requires precise control over chemical hydrolysis, as parameters such as acid concentration and reaction duration exert a deterministic influence on the resulting crystallinity index and morphology. While hydrochloric acid is a common reagent, there is a critical requirement for a refined, data-driven approach to optimize its application to ensure both structural integrity and process efficiency [4]. Additionally, the composition of high carbon content of sawdust makes it a suitable feedstock for applications such as activated carbon production and energy generation. Its structural and chemical characteristics also support its use in a range of industrial and environmental applications. The composition of meranti wood sawdust is presented in Table 1 [5].

Table 1: The composition of Meranti wood sawdust

Analysis	**Composition (%, w/w)**
Extractives	3.08
Ash	0.64
Lignin	33.56
Cellulose	41.58
Hemicellulose	32.81

The significance of this research lies in establishing preliminary parameter of technical foundation of framework for the valorization of sawdust waste. By systematically modulating hydrochloric acid concentrations and hydrolysis durations, this study aims to delineate the precise processing window required to maximize the crystallinity index and crystallite dimensions of cellulose nanocrystals. Moreover, this work offers a comprehensive structural analysis utilizing Fourier Transform Infrared Spectroscopy (FTIR) and X-ray Diffraction (XRD). Furthermore, this research validates the role of alkaline pretreatment in conditioning the feedstock for effective hydrolysis, providing a morphological assessment via Scanning Electron Microscopy (SEM) that correlates process efficiency with material performance. Ultimately, this study provides a fundamental insight of potential for future scale-up and facile pathway for converting industrial wood waste into high-value functional materials, thereby advancing the frontiers of sustainable materials science.

MATERIALS AND METHODS

Experimental Material and Equipment

SWD serving as the primary lignocellulosic precursor, was procured from a local wood processing sector. To ensure experimental consistency, the raw biomass was subjected to systematic purification, including thorough washing with deionized water to remove extraneous debris, followed by mechanical grinding and sieving to achieve a uniform particle size distribution. Analytical-grade sodium hydroxide (NaOH) was employed for the alkaline delignification process, while hydrochloric acid (HCl) was utilized as the hydrolytic agent for cellulose nanocrystal isolation. All chemical reagents were utilized as received without further purification. Throughout the experimental procedure, deionized water (resistivity ≥ 18.2 MΩ·cm) was used exclusively for washing and sample preparation to minimize potential ionic contamination and ensure the reproducibility of the structural characterization [6].

Preparation of Sawdust

To ensure experimental consistency and minimize extraneous contamination, the raw sawdust waste was

initially subjected to a rigorous purification process, involving multiple rinses with deionized water to eliminate particulate impurities, soil, and foreign debris. Following cleaning, the biomass was dehydrated in a forced-convection oven at 80 °C for 12 to 24 h to ensure the complete removal of moisture, which is critical for maintaining accurate stoichiometric ratios during subsequent chemical treatments. The dried sawdust was then been grinded using a high-energy laboratory grinder and passed through a standardized sieve to achieve a uniform particle size distribution, thereby optimizing the surface area-to-volume ratio for efficient alkaline pretreatment and subsequent hydrolysis.

Alkaline Treatment of Sawdust

To facilitate the effective isolation of cellulose, the processed sawdust was subjected to an alkaline delignification protocol designed to remove recalcitrant non-cellulosic components, including lignin and hemicellulose [7]. In this procedure, approximately 10 g of the dried sawdust substrate was dispersed in a 5 wt.% NaOH solution. The reaction was maintained at 80 °C for 2 h under continuous, vigorous stirring to ensure uniform chemical penetration and optimal dissolution of the matrix. Following the treatment, the resultant cellulose pulp was subjected to extensive washing cycles with deionized water until the filtrate reached a neutral pH, ensuring the complete removal of residual alkali and solubilized impurities. Finally, the purified cellulose samples were dehydrated in a convection oven at 60 °C for 12 h to ensure stability before proceeding to the subsequent acid hydrolysis extraction process.

Extraction and Isolation of Cellulose by Acid Hydrolysis

The isolation of CNCs was done through controlled acid hydrolysis, a process designed to selectively remove the amorphous regions of the cellulose fibers [8]. In this study, the alkaline-treated sawdust pulp was subjected to HCl hydrolysis under rigorously maintained conditions. The experiment focused on preliminary parameter study on two critical parameters: the acid concentration (60–65 wt.%) and the hydrolysis duration (30–60 minutes), both executed at a constant temperature of 45 °C to ensure reaction stability. The specific experimental parameters utilized for the hydrolysis procedures are recorded in Table 2. Upon completion of the designated reaction interval, the suspension was immediately quenched with chilled deionized water to terminate further hydrolytic degradation. The recovered nanocellulose was subsequently subjected to repeated centrifugation and washing cycles until a neutral pH was attained, followed by dehydration to yield a stable powder for subsequent analytical characterization. The resulting suspension was subjected to repeated high-speed centrifugation and washing cycles until the supernatant achieved a near-neutral pH, signifying the complete removal of residual acid and reaction byproducts. The purified nanocellulose was subsequently recovered and dehydrated, ensuring it remained in a stable state for subsequent structural and morphological characterization.

Table 2. Experimental parameters for the acid hydrolysis of sawdust waste.

Sample	HCl concentration (wt.%)	Hydrolysis time (min)	Temperature (°C)
H1	60	30	45
H2	60	60	45
H3	65	30	45
H4	65	60	45

Material Characterization

The extracted nanocellulose samples were subjected to a comprehensive analytical regime to evaluate their chemical structure, crystalline integrity, and morphological characteristics. FTIR was utilized to verify the successful removal of lignin and hemicellulose during the pretreatment and hydrolysis phases. Spectra were

recorded in the range of 4000–400 cm^{-1} to detect shifts in the O-H stretching and C-H vibrations, which are indicative of cellulose purification.

X-ray diffraction was used to examine the crystalline structure of the untreated, alkaline-treated, and acid-hydrolyzed materials. The crystallinity index (CrI) was calculated using the Segal method:

$$\mathrm{CrI}(\%) = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \qquad \text{Equation 1}$$

where I_{002} represents the intensity of the main crystalline cellulose reflection and I_{am}represents the intensity associated with the amorphous region. The apparent crystallite size was estimated using the Scherrer equation:

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad \text{Equation 2}$$

where Dis the apparent crystallite size, Kis the shape factor, λis the X-ray wavelength, βis the full width at half maximum (FWHM), and θis the diffraction angle. It should be emphasized that the Scherrer-derived value represents the apparent coherent crystallite dimension and should not be interpreted as the physical length or width of individual CNC particles.

To quantify the structural order of the nanocellulose, XRD analysis was performed using a Rigaku Table top Diffractometer, with Cu Kα radiation. The crystallinity index (CrI) was calculated based on the intensities of the crystalline and amorphous peaks. This metric is crucial for evaluating the effectiveness of the hydrolysis parameters in isolating highly ordered crystalline domains.

Finally, the surface morphology and dimensions of the resulting nanocellulose were examined using SEM machines provided at Universiti Teknikal Malaysia Melaka. Samples were sputter-coated with a thin, conductive layer of gold to prevent charging and to provide high-resolution images of the nanofibrillar network [9]. This imaging was essential for confirming the successful disintegration of the sawdust matrix into nanoscopic structures under the optimized hydrolysis conditions. SEM observations were not used to quantitatively determine CNC dimensions because SEM alone does not provide sufficient evidence for confirming nanoscale particle dimensions.

RESULT AND DISCUSSION

Effect of Alkaline Treatment on the Structural Properties of Sawdust Waste

The alkaline treatment was performed to remove non-cellulosic components, primarily hemicellulose and lignin, from the sawdust waste prior to acid hydrolysis. This pretreatment step is important because the presence of amorphous region can affect the structural characteristics of nanocellulose [10]. The changes in crystallinity, chemical functional groups, and surface morphology before and after alkaline treatment provide insight into the structural changes of the sawdust fibers during the purification process.

X-ray Diffraction Analysis of Alkaline-Treated Sawdust

The XRD patterns of untreated and alkaline-treated sawdust waste are presented in Figure 1, while the corresponding crystallinity indices are summarized in Table 3. Both samples exhibited characteristic diffraction peaks at approximately 2θ = 22°, which are associated with the crystalline regions of cellulose I, whereas the broad diffraction peak around 2θ = 18° represents the amorphous phase. The preservation of the characteristic cellulose peak after alkaline treatment suggests that the crystalline cellulose structure remained intact throughout the treatment process.

The crystallinity index increased from 51% for untreated sawdust to 68% after alkaline treatment. This change is consistent with the removal of part of the non-cellulosic and less ordered components during alkaline pretreatment. The increase in CrI should therefore be interpreted as an increase in the relative proportion of crystalline cellulose rather than the formation of new crystalline cellulose [11]. Similar observations have been reported in previous studies on lignocellulosic biomass treatment, where alkaline pretreatment selectively removes non-cellulosic constituents while preserving the cellulose crystalline domains. The increase in crystallinity provides evidence that the alkaline pretreatment altered the composition of the sawdust matrix. However, quantitative cellulose, hemicellulose, and lignin analyses would be required to directly determine the extent of chemical removal.

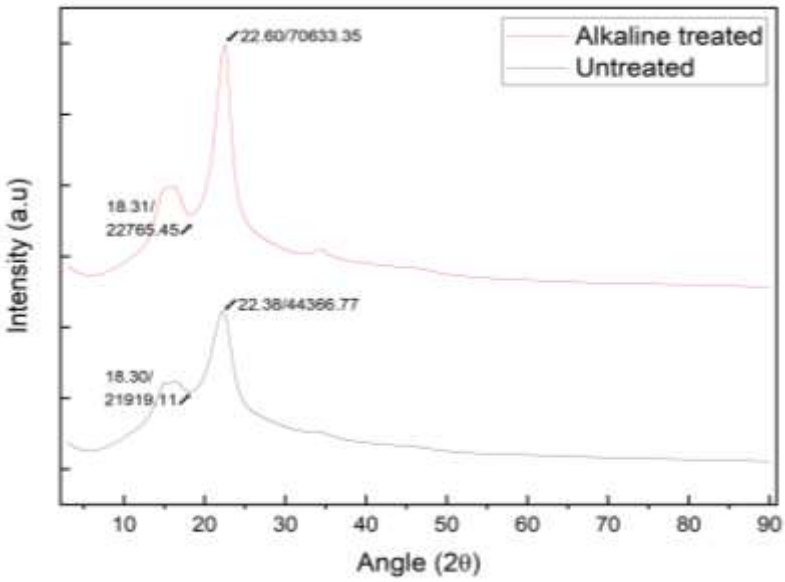

Figure 1: X-ray diffraction patterns of untreated and alkaline treated SDW

Table 3: Crystallinity Index (CrI) of untreated and alkaline treated SDW

Sample	**2θ(Amorphous)**		**2θ(Crystalline)**		**Crystallinity Index %**
	Degree	Intensity (Iam)	Degree	Intensity (I002)	
Untreated Sawdust Waste	18.3	21919	22.38	44366	51%
Alkaline Treated Sawdust Waste	18.31	22765	22.60	70633	68%

Fourier Transform Infrared Spectroscopy Analysis

The FTIR spectra of untreated and alkaline-treated sawdust waste are shown in Figure 2. The spectra revealed several characteristic absorption bands associated with cellulose and other lignocellulosic components [12]. The broad absorption band observed at approximately 3325–3337 cm^{-1} corresponds to the stretching vibration of hydroxyl groups involved in intermolecular hydrogen bonding. Meanwhile, the peaks located in the region of 812–831 cm^{-1} are attributed to C–H vibrations, whereas the absorption bands near 1024–1032 cm^{-1} correspond to C–O stretching vibrations.

Following alkaline treatment, slight shifts and changes in peak intensity were observed. These variations may indicate changes in the hydrogen bonding environment and partial removal of hemicellulose and lignin components from the sawdust structure. The changes in band position and intensity indicate that the chemical environment of the lignocellulosic matrix was modified during alkaline treatment. The observations are consistent with partial removal of non-cellulosic components; however, FTIR alone does not provide quantitative cellulose purity. Therefore, given the preliminary nature of this study, further quantitative analyses are recommended to verify the removal of non-cellulosic components and to provide definitive evidence of high cellulose purity.

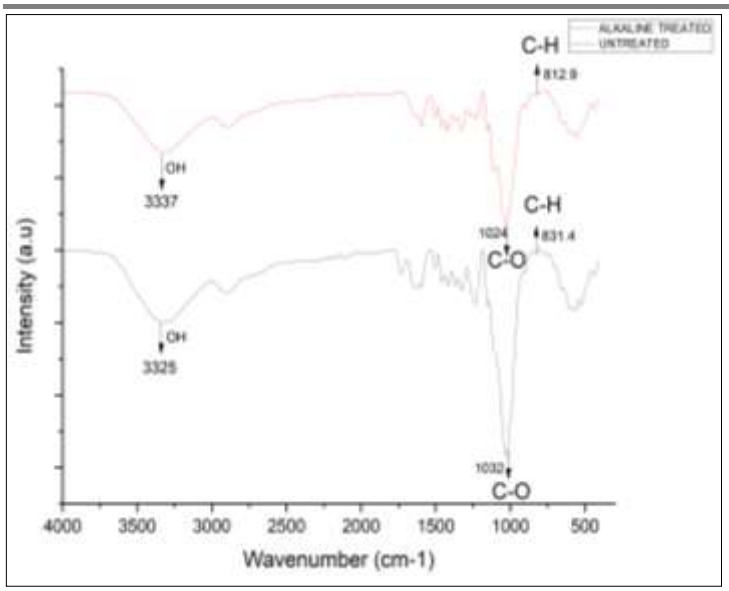

Figure 2: FTIR For Untreated and alkaline treated SDW

. Morphological Analysis of Alkaline-Treated Sawdust

The surface morphology of untreated and alkaline-treated sawdust was examined using SEM, and the corresponding images are presented in Figures 3 and 4. Visual observation of the samples showed slight differences in color and texture after alkaline treatment, indicating structural modifications induced by the removal of non-cellulosic materials [13].

The untreated sawdust exhibited an irregular and relatively rough surface morphology with the presence of surface impurities and agglomerated structures. These features are commonly associated with lignin, hemicellulose, waxes, and other extractive compounds that naturally exist in lignocellulosic biomass. After alkaline treatment, the surface morphology appeared cleaner and relatively rougher, suggesting the removal of surface impurities and partial exposure of cellulose fibrils. The observed morphological changes indicate that the alkaline treatment effectively altered the surface structure of the sawdust, thereby facilitating subsequent acid hydrolysis.

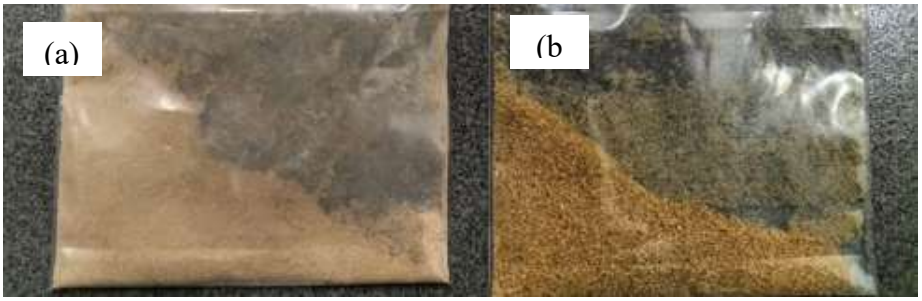

Figure 3: Image of SDW fiber (a) before alkaline treatment and (b) after alkaline treatment

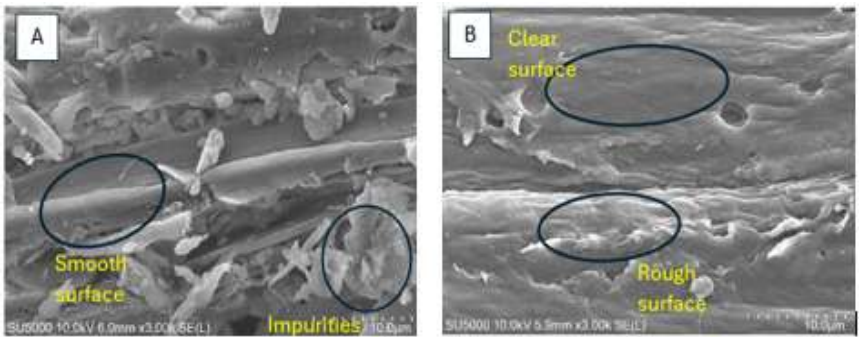

Figure 4: Surface morphology of (A) untreated SDW and (B) Alkaline treated SDW

Effect of Acid Hydrolysis Parameters on Nanocellulose Formation

Acid hydrolysis was performed to isolate nanocellulose from the alkaline-treated sawdust fibers by selectively removing the amorphous cellulose regions. In this study, the effects of acid concentration and hydrolysis duration were investigated while maintaining a constant reaction temperature. The structural evolution of the hydrolyzed samples was evaluated using XRD, FTIR, and SEM analyses to determine the influence of processing parameters on the characteristics of the resulting nanocellulose.

X-ray Diffraction Analysis of Acid-Hydrolyzed Samples

The XRD patterns of nanocellulose obtained under different hydrolysis conditions are presented in Figure 5, while the corresponding crystallinity indices and crystallite sizes are summarized in Tables 4 and 5, respectively. All samples exhibited diffraction peaks near 2θ values of approximately 18°, 22°, and 23°, which are characteristic of cellulose I. The retention of these diffraction peaks indicates that the native crystalline structure of cellulose was preserved during acid hydrolysis. The crystallinity index of the samples ranged from 66% to 71%, which was higher than that of untreated and alkaline-treated sawdust. This increase in crystallinity may be attributed to the preferential removal of amorphous cellulose regions during acid hydrolysis. Among the investigated conditions, the samples prepared using 60 wt.% acid for 30 min and 65 wt.% acid for 60 min exhibited the highest crystallinity values of 71%. However, the differences observed among the investigated conditions were relatively small, suggesting that both acid concentration and hydrolysis duration influenced the structural properties of the nanocellulose.

The calculated crystallite sizes ranged from 2.27 to 2.85 nm. The largest crystallite size was observed for the sample hydrolyzed using 60 wt.% acid for 30 min, whereas the smallest crystallite size was obtained at 65 wt.% acid for 30 min. These findings suggest that acid concentration and hydrolysis duration affected the degree of crystalline domain preservation during the hydrolysis process.

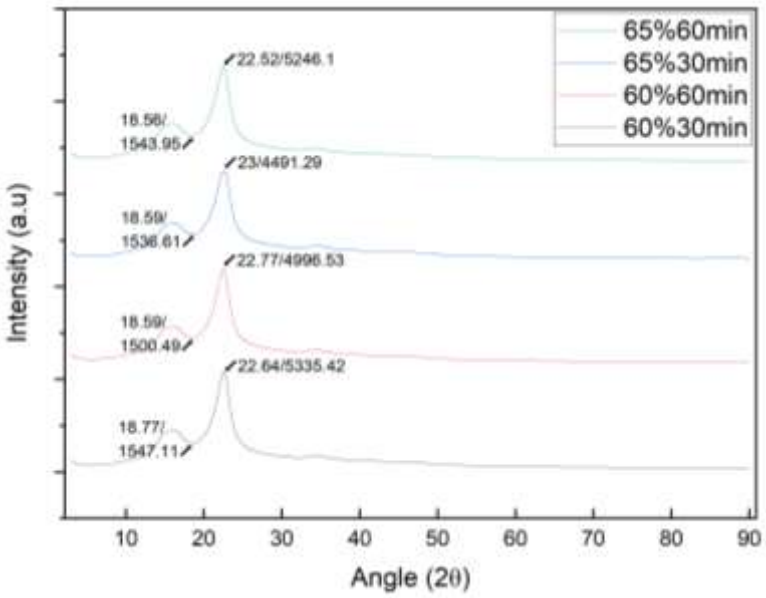

Figure 5: XRD analysis of HCl hydrolysis for 60wt% for 30min, 60wt% for 60min, 65wt% for 30min and 65wt% for 60min

Table 4: Cellulose intensity peak and the respective CrI

Sample	2θ(Amorphous)		2θ(Crystalline)		Crystallinity Index %
	Degree	Intensity (Iam)	Degree	Intensity (I002)	
Untreated Sawdust Waste	18.3	21919	22.38	44366	51%
Alkaline Treated Sawdust Waste	18.31	22765	22.6	70633	68%

HCl 60wt%30min	18.77	1547.11	22.64	5335.42	71%
HCl 60wt%60min	18.59	1500.49	22.77	4996.53	70%
HCl 65wt%30min	18.59	1536.61	23	4491.29	66%
HCl 65wt%60min	18.56	1543.95	22.52	5246.1	71%

Table 5: Crystallite size of cellulose nanocrystal (CNC) at different parameters

Sample	Half Intensity (I002)	b	a	FWHM	(θ), rad	D,Crystallite Size (nm)
Untreated Sawdust Waste	22183	24.27	18.72	0.0969	0.195302	1.4591
Alkaline Treated Sawdust Waste	35316.5	23.73	20.62	0.0543	0.195302	2.6039
HCl 60wt%30min	2667.71	23.73	20.89	0.0496	0.195302	2.8515
HCl 60wt%60min	2498.265	23.74	20.73	0.0525	0.195302	2.6904
HCl 65wt%30min	2245.645	24.02	20.46	0.0621	0.195302	2.2748
HCl 65wt%60min	2623.05	23.69	20.77	0.0510	0.195302	2.7734

Fourier Transform Infrared Spectroscopy Analysis of Nanocellulose

The FTIR spectra of nanocellulose samples prepared under different hydrolysis conditions are shown in Figure 6. The spectra exhibited several characteristic absorption bands corresponding to cellulose structures [14]. The absorption band around 3331 cm^{-1} is associated with O–H stretching vibrations, whereas the peaks near 1148 cm^{-1} and 1026 cm^{-1} correspond to cellulose I structures and C–O stretching vibrations, respectively.

The similarity of the FTIR spectra across all hydrolysis conditions suggests that the chemical structure of cellulose was largely preserved following acid hydrolysis. Minor differences in peak intensity and position may be attributed to variations in the removal of amorphous components and changes in intermolecular hydrogen bonding. The absence or reduction of certain bands associated with non-cellulosic constituents further supports the effectiveness of the purification process. However, additional characterization techniques would be required to quantitatively determine the purity of the isolated nanocellulose.

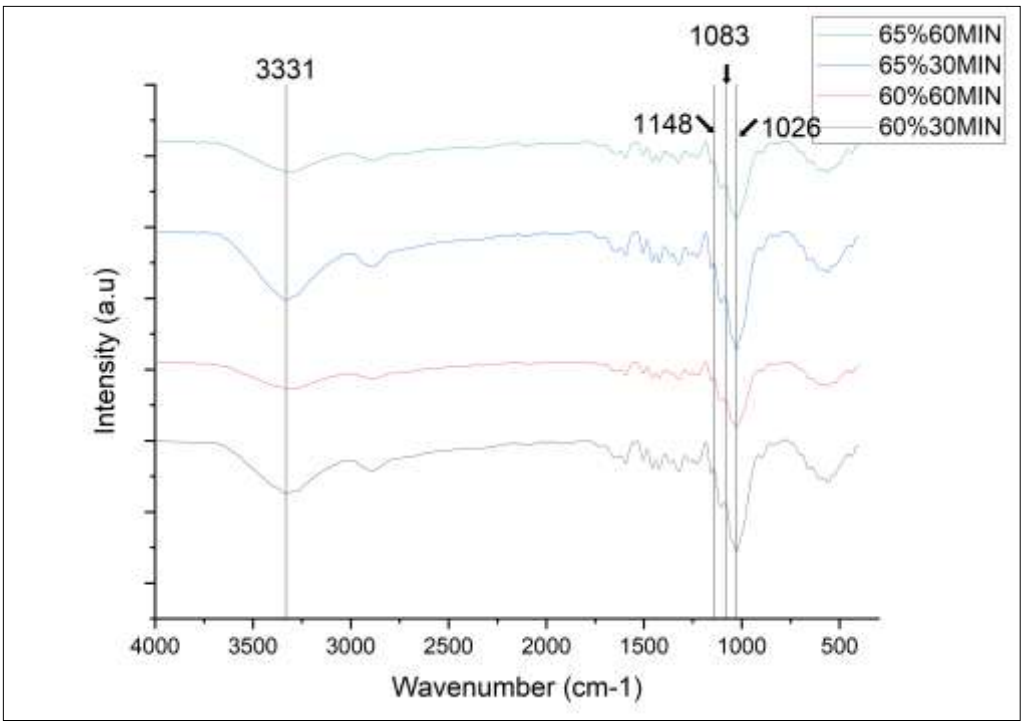

Figure 6: FTIR Spectra of HCl hydrolysis for 60wt% for 30 min, 60wt% for 60 min, 65wt% for 30 min and 65wt% for 60 min

Morphological Analysis of Nanocellulose

The morphology of nanocellulose obtained under different acid hydrolysis conditions was investigated using SEM, and the results are presented in Figures 7. Visual inspection of the samples indicated slight differences in powder appearance after hydrolysis. SEM observations revealed the presence of fragmented cellulose structures with varying degrees of particle aggregation [15].

The occurrence of aggregation may be attributed to intermolecular hydrogen bonding during the drying process, as well as incomplete removal of residual impurities during pretreatment. The morphology observed in the present study is comparable to previous reports on acid-hydrolyzed cellulose materials. However, it should be noted that SEM analysis alone cannot conclusively confirm nanoscale dimensions, and additional analyses such as transmission electron microscopy (TEM) or atomic force microscopy (AFM) would be required to further characterize the dimensions and morphology of the isolated nanocellulose.

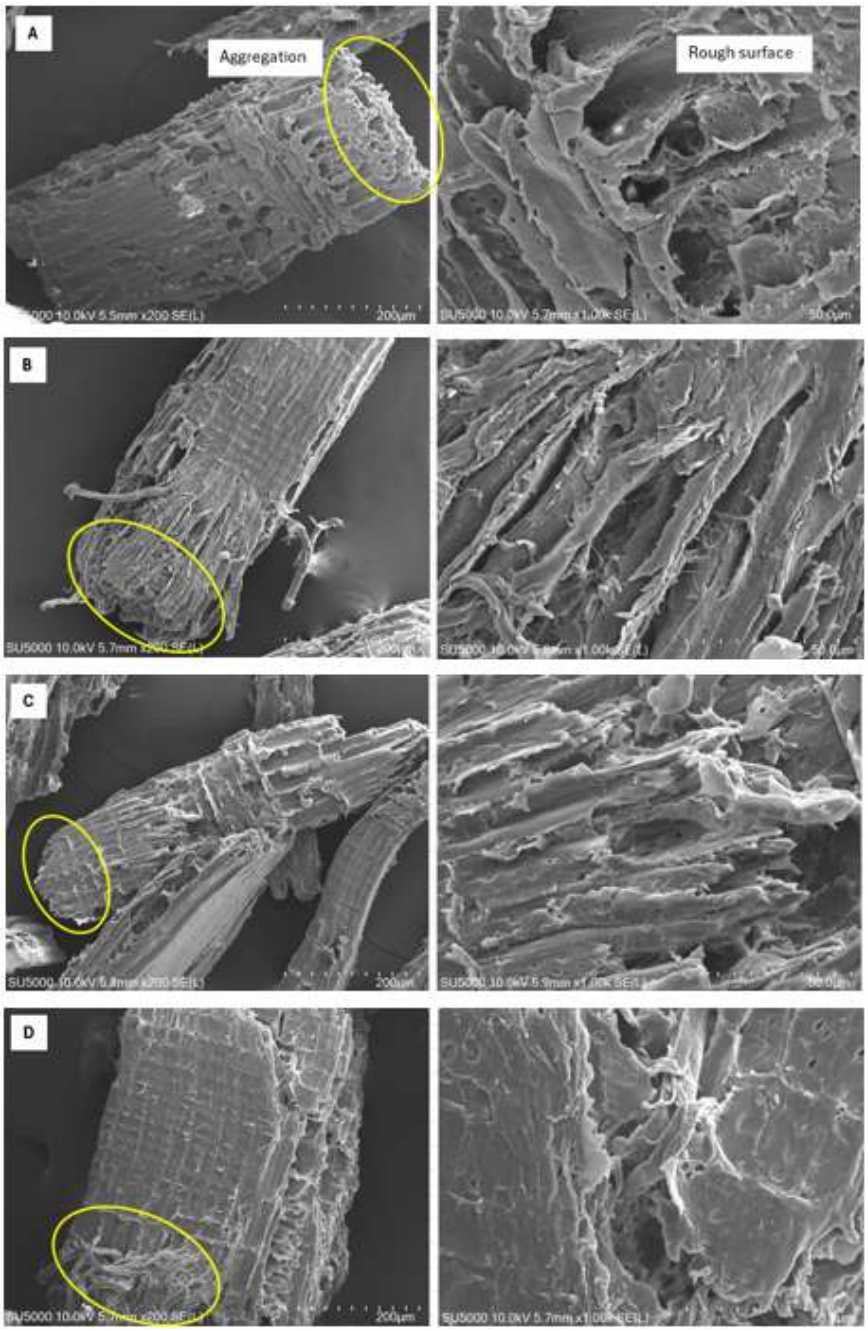

Figure 7: Surface morphology of HCl acid hydrolysis at (A) 60wt% for 30min, (B)60wt% for 60min, (C)65wt% for 30min and (D)65wt% for 60min

CONCLUSION

This study demonstrated the successful isolation of nanocellulose from sawdust waste through a two-stage process combining alkaline treatment with 5 wt.% NaOH and subsequent hydrochloric acid hydrolysis. The transformation from raw sawdust to nanocellulose was confirmed by structural and morphological analyses. Specifically, XRD results indicated that the crystallinity index improved from 51% in the untreated biomass to 71% under optimized hydrolysis conditions, with crystallite sizes ranging from 2.27 to 2.85 nm. FTIR and SEM analyses corroborated these findings, confirming the effective, albeit partial, removal of lignin and hemicellulose, which facilitated the preservation of the cellulose structure. Among the tested parameters, hydrolysis at 65 wt.% HCl for 60 minutes at 45 °C emerged as an effective approach for enhancing both crystallinity and crystallite size. These results underscore the viability of sawdust waste as a lignocellulosic feedstock for nanocellulose production. While this study establishes a foundational processing framework, rather than a statistically validated optimization, future work should include replicated experiments, additional concentration and time levels, quantitative extraction yield and cellulose purity, and direct nanoscale characterization using TEM or AFM. These additions would provide a stronger basis for assessing the material's suitability for specific applications.

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