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Green Extraction of Microcrystalline Cellulose from Cabbage Waste *(Brassica Oleracea L.)* via Steam Explosion Under Pressurized and Non-Pressurized Nitrogen (N_2)

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ABSTRACT: Agricultural residues such as cabbage waste (Brassica oleracea L.) are rich in cellulose and offer promising potential for sustainable microcrystalline cellulose (MCC) production. This study aims to extract and characterize MCC from cabbage waste using an environmentally friendly approach that combines high-speed blending, low-concentration oxalic acid hydrolysis (0–2% w/v), and steam explosion at 130 °C for 15 minutes, under both pressurized and non-pressurized nitrogen (N_2) atmospheres. The application of pressurized N_2 significantly improved delignification efficiency and preserved cellulose crystallinity. The optimal treatment (2% oxalic acid with N_2) yielded a cellulose content of 79.18%, with hemicellulose and lignin contents reduced to 15.28% and 0.10%, respectively. FTIR analysis confirmed the effective removal of non-cellulosic components, while XRD analysis revealed a crystallinity index 66%, which is high compared to typical MCC values from other biowastes (~50–60%). SEM revealed clean and well-dispersed fiber morphology. These results indicate that oxalic acid combined with N_2-assisted steam explosion is an effective and eco-friendly method for producing MCC. This approach minimizes chemical use and oxidation, making it suitable for pharmaceutical excipients, biodegradable composites, and other green material applications. Overall, the process aligns with circular economy principles and contributes to the Sustainable Development Goals (SDGs).

Keywords: Microcrystalline cellulose (MCC); Cabbage waste; Steam explosion; Green extraction; Nitrogen atmosphere

1. Introduction

Agricultural activities generate a substantial amount of biomass waste, much of which remains underutilized and contributes to environmental burdens if improperly managed. One of the major contributors to this waste is vegetable residue, especially from cabbage *(Brassica oleracea L.)*, where up to 30% of post-harvest biomass primarily outer leaves is typically discarded. In Indonesia alone, cabbage waste is estimated to reach nearly 33,000 tons annually (Statistics Indonesia, 2022). This biomass is rich in cellulose (31–67%), a biodegradable and renewable biopolymer widely used in pharmaceutical, packaging, and biomedical applications (Khukutapan et al., 2018; Ciolacu et al., 2020). Conventional methods like acid hydrolysis, while effective, often involve the use of strong acids (e.g., sulfuric or hydrochloric acid), which can result in low yield and generate hazardous waste (Song et al., 2018; Jiang et al., 2021). Consequently, more sustainable and efficient extraction approaches are urgently needed.

Recent studies have explored greener alternatives, including mechanical and thermochemical methods such as high-speed blending and steam explosion. These methods effectively disrupt the cell wall and reduce chemical use, thereby enhancing environmental compatibility (Sofiah et al., 2023; Ventura-Cruz & Tecante, 2021). However, the main drawback of purely mechanical processes is their high energy consumption. Therefore, mechanical methods are often integrated with pretreatment techniques to reduce energy demands (Phanthong et al., 2018). Steam explosion utilizes high-pressure, high-temperature steam to break down the lignocellulosic matrix, increasing cellulose accessibility. When combined with light acid hydrolysis such as using oxalic acid this technique improves delignification and defibrillation while minimizing environmental impact. In this study, we address this limitation by integrating oxalic acid pretreatment and nitrogen-assisted steam explosion, aiming to achieve high extraction efficiency with reduced energy input and minimal chemical usage. Moreover, nitrogen (N_2) pressure serves as an inert medium that facilitates reagent penetration, minimizes oxidation, and preserves the crystalline integrity

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of cellulose (Raud et al., 2019; Neenu et al., 2022; Vishnoi et al., 2024) .

This study aims to extract microcrystalline cellulose (MCC) from cabbage waste through a green method combining high-speed blending, low-concentration oxalic acid treatment, and steam explosion under both pressurized and non-pressurized nitrogen atmospheres. Unlike previous studies that rely solely on mechanical or chemical treatments, this research introduces nitrogen pressurization as a novel factor to enhance extraction efficiency, reduce thermal degradation, and improve crystallinity. The proposed method contributes to the field of sustainable materials by minimizing chemical inputs and valorizing agricultural waste, in alignment with green chemistry and circular economy principles. The extracted MCC is expected to exhibit suitable properties for application in pharmaceutical excipients, bio-based packaging, and biodegradable composite materials.

2. Materials and Methods

2.1 Materials

All chemicals used in this study were of analytical grade and used without further purification. The raw material consisted of the outer leaves of cabbage waste (*Brassica oleracea* L.), an agricultural by-product collected from local markets in Pasuruan, East Java, Indonesia. Sodium hydroxide (NaOH, CAS No. 1310-73-2; Merck) and sodium hypochlorite (NaOCl; PT Chemical Indonesia Multi Sentosa) were used for delignification and bleaching, respectively. Oxalic acid (CAS No. 6153-56-6; Merck) was used as the organic acid for hydrolysis.

2.2 Method

2.2.1 Pre treatment

The cabbage waste was first chopped into pieces approximately 5 cm in size and subjected to high-speed blending in a cabbage-to-water ratio of 1:2 (w/v) using a commercial blender. The slurry was then filtered, washed thoroughly with deionized water, and oven-dried at 60 °C for 8 hours. The dried material was ground into a fine powder to improve its reactivity during the delignification process.

2.2.2 Delignification and Bleaching

50 g of the dried powder was treated with 500 mL of 1% (w/v) NaOH solution at 80 °C for 120 minutes. The residue was filtered, washed until neutral pH was reached, and bleached using 2% (w/v) NaOCl at 70 °C for 30 minutes. The bleaching process continued until the sample turned white, after which it was washed again until neutral pH was reached.

2.2.3 Steam Explosion

3 g of the sample were subjected to steam explosion at 130 °C for 15 minutes using varying concentrations of oxalic acid (OA) 0%, 0.5%, 1.0%, 1.5%, and 2.0% (w/v) under two different conditions: with and without nitrogen (N_2) pressurization. For the nitrogen-assisted condition, compressed N_2 gas was introduced into the steam chamber to a pressure of 50 bar before the heating stage. After treatment, the samples were filtered, rinsed with deionized water until neutral pH was achieved, and dried in an oven at 60 °C.

2.3 Characterization

Characterization of the resulting microcrystalline cellulose was performed using FTIR, XRD, and SEM analyses. FTIR spectra were obtained using The Agilent Cary 630 over a range of 4000–500 cm^{-1} to identify molecular vibrations associated with cellulose and lignocellulosic residues. Crystallinity analysis was carried out using X-ray Diffraction (XRD) on a Rigaku Miniflex 600 diffractometer equipped with Cu-Kα radiation (λ = 1.5406 Å), operating at 40 kV and 15 mA. Scans were conducted from 5° to 50° (2θ) at a rate of 2°/min. The crystallinity index (CrI) was calculated using the Segal method, based on the following equation:

$$CrI\ (\%) = \frac{I_{002} - I_{am}}{I_{002}} x100 \quad (1)$$

where I_{002} represents the peak intensity at $2\theta \approx 22.5°$ corresponding to the crystalline region, and I_{am} represents the intensity at $2\theta \approx 18°$, indicating the amorphous portion (Segal et al., 1959). Surface morphology was observed using a Hitachi FlexSEM 1000 (version 01-10) at an accelerating voltage of 15 kV. Prior to imaging, samples were gold-coated, and micrographs were taken at magnifications at 500×.

3. Results and Discussion

3.1 Composition Analysis

The cabbage waste used in this study was primarily composed of cellulose, with additional components including hemicellulose and lignin. To enhance the yield of cellulose extraction, non-cellulosic components such as lignin, hemicellulose, were removed through sequential pretreatment, delignification, and bleaching processes. The composition and content of these three components are not fixed but vary depending on the species, type, and source of the lignocellulosic biomass. (Phanthong et al., 2018).

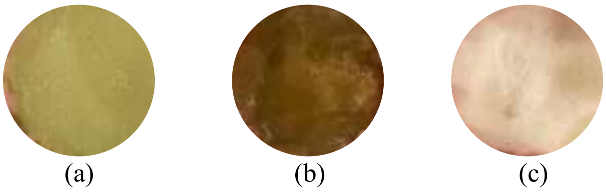
(a) (b) (c)

Figure 1. Visual observation of cabbage fiber samples at different processing stages: (a) fibers after pretreatment, (b) fibers after delignification, and (c) fibers after bleaching

The color changes observed in the material during the processing of cabbage waste reflect the underlying chemical transformations, particularly the removal of lignin, pigments, and other non-cellulosic compounds. In its native state, the cabbage waste fibers appeared leaf-green, indicating the presence of natural pigments such as chlorophyll, along with lignin and phenolic compounds.

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This green hue gradually faded to a pale green after the pretreatment stage, suggesting the initial degradation of pigments and partial removal of non-cellulosic components. These color changes were closely associated with alterations in the fiber's morphological structure, where friction and collision forces between fibrils occurred during the blending process. The strong agitation induced orientational stress on the fibrils, allowing water to penetrate the microfibril structure and create balloon-like expansions. As these balloon-like structures extended toward the fibril edges, pulp fragments with torn cell walls disintegrated into finer particles. This transformation indicates that the mechanical process not only caused physical disintegration but also accelerated the breakdown of colored non-cellulosic components (Uetani & Yano, 2011).

In accordance with the research of Zulkania et al. (2024), during the delignification stage, the material exhibited a color change to brown. This change was attributed to the breakdown of the lignocellulosic structure, leading to the formation of compounds containing aryl-ester, carbon–carbon, and alkyl–alkyl bonds. This decomposition process signified the release of lignin fractions from the cellulose–hemicellulose matrix, which was visually indicated by the appearance of a dark brown solution (Cahyani et al., 2023).

The bleaching process represents the final stage in cellulose purification, aimed at removing residual lignin, pigments, and other colored compounds that were not degraded in the previous steps. Bleaching resulted in a color change of the material from brown to white, indicating the successful elimination of chromophoric components and an increase in cellulose purity. In this study, bleaching was performed using a sodium hypochlorite (NaOCl) solution, which is widely recognized as an effective oxidative bleaching agent for the degradation of lignin and aromatic phenolic compounds. According to Juwita et al. (2018), when sodium hypochlorite is dissolved in water, it releases chlorine (Cl_2) into the solution. Chlorine acts as a strong oxidizing agent capable of breaking double bonds in organic molecules, including lignin and complex pigments. This oxidative reaction produces a significant whitening effect, confirming the efficiency of the bleaching treatment.

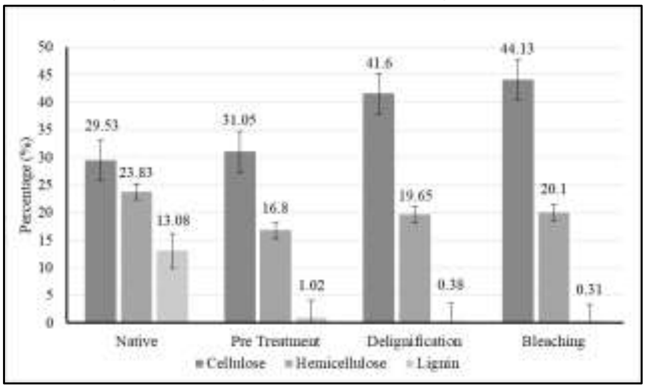

Figure 2. Chemical composition (cellulose, hemicellulose, lignin) of cabbage waste fibers at different processing stages: pre-treatment, delignification, and bleaching

Based on **Figure 2**, the contents of cellulose, hemicellulose, and lignin in cabbage waste underwent significant changes following each treatment stage. In its native state, cabbage waste contained 29.53% cellulose, 23.83% hemicellulose, and 13.08% lignin. After the pretreatment stage, cellulose content increased to 31.05%, while lignin content drastically decreased to 1.02%, indicating the process's effectiveness in removing the majority of lignin

Subsequent delignification further increased cellulose content to 41.60% and reduced lignin to 0.38%, while hemicellulose content slightly increased to 19.65%. At the final stage, bleaching, the cellulose content reached 44.13%, with a residual lignin content of only 0.31%. These results demonstrate that the series of treatments significantly improved cellulose purity, which is crucial to produce high-quality microcrystalline cellulose.

The delignification stage notably enhanced cellulose content to 41.60%, with only 0.38% lignin remaining. This aligns with findings by Rahayu et al. (2022), who reported that delignification processes effectively reduce lignin levels while increasing cellulose concentrations in biomass. Chemical treatment with dilute alkali solutions has proven effective in altering the structure of lignocellulosic materials. Alkali solutions disrupt the lignocellulosic cell wall by dissolving lignin and hemicellulose and hydrolyzing uronic acids. This occurs as alkaline conditions break the structural bonds between lignin and carbohydrates through saponification of inter-molecular ester linkages and cleavage of ether glycosidic bonds within the lignin structure. In addition, hemicellulose undergoes depolymerization into water-soluble monosaccharides and oligosaccharides. Overall, this alkali treatment facilitates the removal of non-cellulosic components and eases the isolation of cellulose from lignocellulosic biomass (Jongaroontaprangsee et al., 2018).

Furthermore, the bleaching process increased cellulose purity to 44.13%, confirming its effectiveness in removing residual lignin. During bleaching, hypochlorite anions (ClO^-) derived from sodium hypochlorite (NaOCl) react with nearly all carbonyl groups in the remaining lignin, resulting in its degradation. Lignin also reacts with sodium chlorite ($NaClO_2$) to form lignin chlorides, which are soluble and removed through repeated washing (Raju et al., 2023). In addition to lignin, residual hemicellulose is also removed during this process. Following bleaching, individual fiber cell walls become separated, although nanofibrillation has not yet occurred. The reduction in lignin content post-bleaching was also demonstrated by Wang & Zhao (2021), who used a similar method on vegetable waste for microcrystalline cellulose production. In conclusion, these results reinforce that cabbage waste has strong potential as a raw material for microcrystalline cellulose production. The applied pretreatment sequence effectively enhances cellulose purity and reduces interfering components such as lignin and hemicellulose.

Table 1. Chemical composition of fibers after steam explosion with various concentrations of oxalic acid under non-nitrogen (OA) and nitrogen-assisted (N_2–OA) conditions

	Non-Pressurized Nitrogen			**Pressurized Nitrogen (N_2-)**			**Relative Difference (Δ)**		
	Cellulose (%)	**Hemi cellulose (%)**	**Lignin (%)**	**Cellulo se (%)**	**Hemi cellulose (%)**	**Lignin (%)**	**ΔCellulose (%)**	**ΔHemi cellulose (%)**	**ΔLignin (%)**
OA 0%	45.65	19.85	0.28	55.00	19.50	0.24	+20.98	-1.76	-14.29
OA 0,5%	48.10	19.10	0.26	61.50	18.38	0.19	+27.86	-3.77	-26.92
OA 1%	56.95	18.38	0.18	68.90	17.10	0.13	+20.98	-6.96	-27.78
OA 1,5%	67.90	17.95	0.15	75.35	16.25	0.12	+10.97	-9.47	-20.00
OA 2%	69.88	16.58	0.11	79.18	15.28	0.10	+13.31	-7.84	-9.09

Notes: Δ values represent relative difference pressurized nitrogen to the non-pressurized N_2 counterpart at each acid concentration

Table 1. shows that the use of nitrogen gas (N_2) during the steam explosion (SE) process had a significant impact on the chemical composition of cabbage waste, particularly in enhancing cellulose content and reducing lignin. The data indicate that the presence of N_2 consistently improved the effectiveness of fractionation, as evidenced by higher cellulose content and lower lignin levels compared to treatments without N_2.

Under conditions with OA 0%, the cellulose content was 45.65%, hemicellulose 19.85%, and lignin 0.28%. Nitrogen pressurization enhanced cellulose content across all oxalic acid concentrations, with the largest gain (+27.86%) at N_2-OA 0.5%. It also consistently reduced hemicellulose and lignin content, especially at N_2-OA 1%, where lignin decreased by −27.78%. This confirms the synergistic effect of N_2 in improving delignification and cellulose enrichment efficiency (Real Pérez et al., 2024). This trend indicates that the use of N_2 not only enhances lignin removal but also helps preserve cellulose integrity during processing. Raud et al. (2019) reported that introducing nitrogen gas into wet biomass under high pressure allows the gas to penetrate the biomass structure. Subsequent heating initiates autohydrolysis, during which acetyl groups in hemicellulose are hydrolyzed, releasing acetic acid that acts as a catalyst to accelerate polysaccharide hydrolysis, especially hemicellulose. A sudden release of pressure then causes the nitrogen gas to expand within the biomass matrix, triggering micro-explosions that break the material into smaller fragments, release fibers, and disrupt the lignocellulosic structure.

Mechanistically, nitrogen gas also provides an inert atmosphere that prevents the formation of free radicals typically generated at high temperatures under oxidative conditions. This helps to protect cellulose from oxidative degradation and inhibits the re-condensation of fragmented lignin into undesirable compounds. These findings are supported by previous studies, including Ricciulli et al. (2024), who reported that using an inert atmosphere during thermochemical biomass processing improves the selectivity of lignin solubilization and reduces the formation of unwanted humin-like substances.

3.2 Fuctional Group Analysis

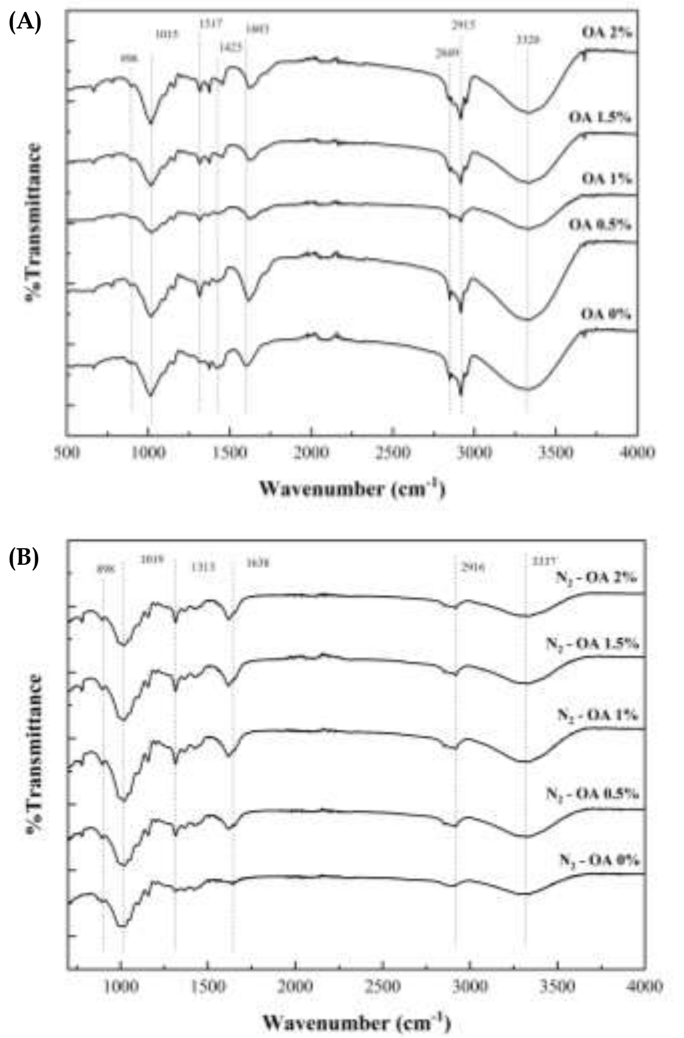

Figure 3. FTIR spectra of MCC after steam explosion treatment **(A)** non-pressurized nitrogen and **(B)** pressurized nitrogen conditions.

In general, both spectra exhibit characteristic cellulose absorption bands. The strong absorption band appearing in the region of 3320–3337 cm⁻¹ is attributed to the stretching vibrations of hydroxyl (–OH) groups, which are the primary features of the cellulose chain (Raju et al., 2023; Thomas et al., 2015). The intensity of this band increases with rising oxalic acid concentration, indicating enhanced exposure of hydroxyl groups due to the removal of amorphous components such as lignin and hemicellulose (Abu Bakar et al., 2022).

The absorption band around 2900–2916 cm⁻¹, related to the stretching vibrations of aliphatic C–H bonds (Chavez et al., 2023; Rana & Gupta, 2020), shows similar intensity across all samples but is more defined at higher acid concentrations. This indicates that the fundamental structure of the cellulose carbon chain remains intact despite partial hydrolysis (Deepa et al., 2015).

Significant changes are observed in the band at 1603–1638 cm⁻¹, associated with C=O vibrations of aromatic lignin groups or O–H bending vibrations of bound water (Lu et al., 2014). In samples without nitrogen, this band remains prominent at low acid concentrations (0–1%), indicating residual lignin presence. Conversely, in nitrogen-treated samples, the intensity of this band drastically decreases or shifts at acid concentrations ≥1%, indicating more efficient lignin removal. This suggests that the use of an inert nitrogen atmosphere helps suppress oxidative side reactions that could impede cellulose purification (Ricciulli et al., 2024).

The strong absorption band in the region of 1015–1020 cm⁻¹ is characteristic of ether C–O–C (glycosidic bond) and alcohol C–O groups (Chavez et al., 2023). The presence of a sharp band at 1015–1019 cm⁻¹ in all samples, especially those treated under nitrogen, indicates that the primary glucan chains of cellulose remain preserved without excessive degradation. The absorption band at 896–898 cm⁻¹, indicative of β-(1→4)-glycosidic linkages, serves as a specific marker for cellulose structure (Thomas et al., 2015). This is consistent with prior findings that associate this band with the ordered glucan chains in cellulose nanofibrils (Deepa et al., 2015), and aligns with the crystallinity trends observed in XRD analysis. The intensity of this band increases with acid concentration and is most pronounced in nitrogen-treated samples at 2% concentration (N_2-OA 2%).

3.3 Crystallinity Analysis

The effect of steam explosion treatment and varying concentrations of oxalic acid on the crystalline structure of cellulose was evaluated using X-ray Diffraction (XRD) analysis, as shown in **Figure 4.** The diffractograms revealed two main diffraction peaks at 2θ values around 16° and 22°, which are indicative of the crystalline structure of cellulose type I (Hongrattanavichit & Aht-Ong, 2020). This is in accordance with the research of Chavez et al. (2023), the intensity of the (002) peak generally increased with higher oxalic acid concentrations, indicating an increase in the crystalline fraction because of the degradation and dissolution of amorphous components such as lignin and hemicellulose during treatment.

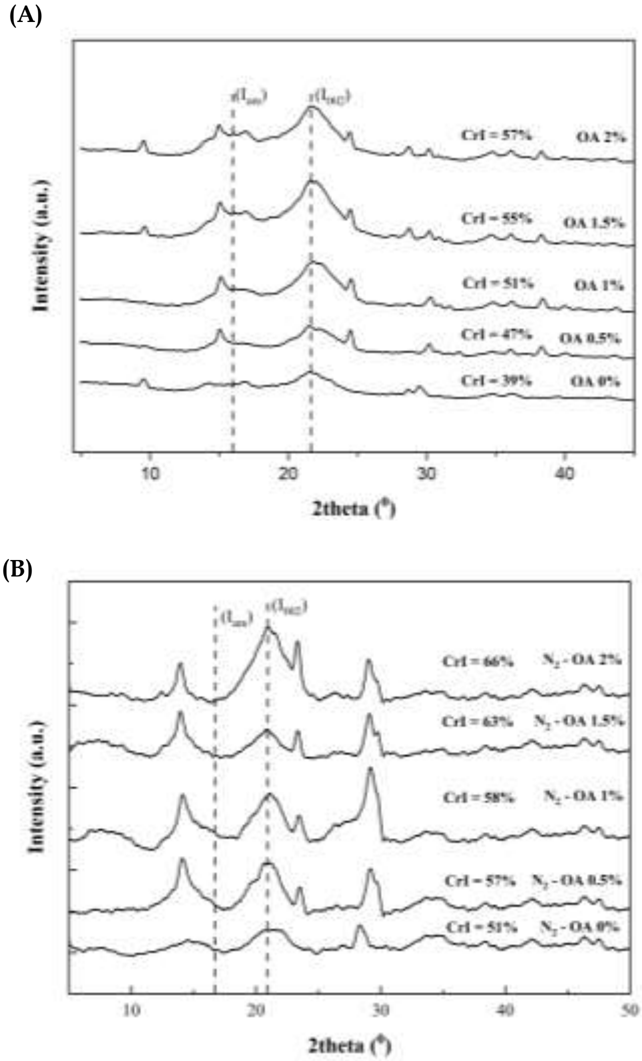

Figure 4. XRD diffractograms of MCC samples treated via steam explosion using oxalic acid under: **(A)** Non-pressurized condition, and **(B)** Pressurized nitrogen condition

These findings are consistent with literature reports stating that hemicellulose and lignin primarily constitute the amorphous regions, while cellulose dominates the crystalline regions of the cell wall. Crystalline cellulose is embedded in a complex matrix composed of lignin, hemicellulose, and pectin (Mazumder & Zhang, 2023). During acid hydrolysis and steam explosion, these amorphous components are degraded and solubilized, thereby increasing the relative crystallinity of the overall structure. Steam explosion is a thermomechanical process that involves treating biomass with high-pressure steam, followed by explosive decompression, which breaks down the biomass fibers and transforms the material into a fibrous, dispersed solid by disrupting the structural components of cellulose (Bandyopadhyay-Ghosh et al., 2015).

Additionally, steam explosion facilitates the penetration of reagents into the internal fiber matrix through defibrillation and pore expansion induced by swelling effects, thereby enlarging the reaction surface area.

Under non-nitrogen conditions (Figure 4A), the (002) peak intensity increased from OA 0% to OA 2%, indicating an increase in crystallinity due to the removal of amorphous fractions such as lignin and hemicellulose. However, the relatively broad and asymmetric peak shape suggests that thermal degradation occurring during steam explosion in the absence of an inert atmosphere may reduce crystalline order. In contrast, under pressurized nitrogen conditions (Figure 4B), the (002) peak appeared sharper and more intense, particularly at N_2-OA 1.5% and N_2-OA 2%, indicating that the presence of pressurized nitrogen acts as an inert atmosphere that suppresses oxidative degradation, thereby preserving the crystalline structure of cellulose.

The combination of 2% oxalic acid and pressurized nitrogen (N_2-OA 2%) yielded the highest and sharpest (002) peak intensity, indicating that this condition is the most optimal for producing highly crystalline and well-defined microcellulose. The role of oxalic acid in this system is to act as a catalyst that facilitates the selective hydrolysis of amorphous components (lignin and hemicellulose) while preserving the crystalline regions of cellulose, as reflected in the increased crystallinity ratio of the material (Lacerda et al., 2015).

The crystallinity index (CrI) increased relatively linearly from OA 0% to OA 2%, reflecting the effectiveness of acid hydrolysis in removing amorphous components such as lignin and hemicellulose. However, the CrI values remained lower compared to those of samples processed under a nitrogen atmosphere. This indicates that steam explosion under oxidative conditions cause partial degradation of the crystalline regions of cellulose, thereby limiting the potential increase in crystallinity. Fibers treated under a nitrogen atmosphere exhibited a steeper increase in CrI, particularly at N_2-OA 1.5% and N_2-OA 2%. This enhancement confirms that the inert atmosphere suppresses oxidative degradation reactions and improves thermal stability during the process. The oxygen-free environment allows the reagents to act more selectively on the amorphous fractions without damaging the crystalline regions of cellulose (Mamleev et al., 2007).

A crystallinity index (CrI) value of 66% is considered relatively high when compared to MCC derived from other biomass wastes. For instance, MCC extracted from banana peel showed a CrI of approximately 64.3% (Gabriel et al., 2020), while that from corn cob was reported to have a CrI of 53% (Shao et al., 2020). MCC produced from *Stipa pbtusa* fibers using similar treatment with weak acid and steam explosion exhibited a CrI of around 52.9% (Chavez et al., 2023). These results suggest that the combination of oxalic acid treatment and nitrogen-assisted steam explosion employed in this study effectively enhanced the purity of the cellulose crystalline fraction by minimizing thermal degradation and preserving the native cellulose I crystal structure.

3.4 Morphology Analysis

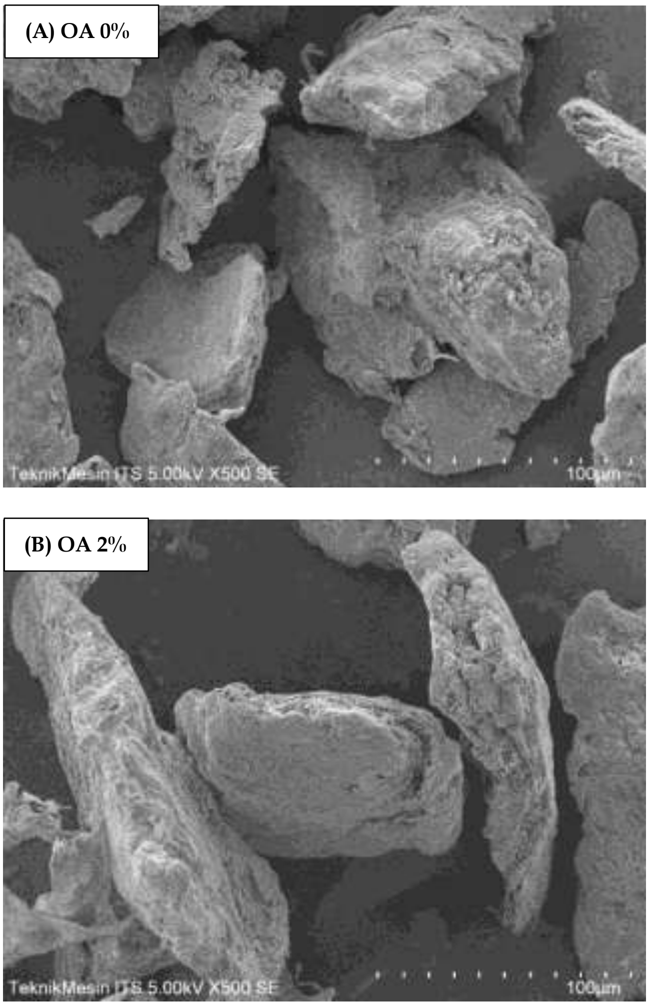

Figure 5 SEM images of cellulose fibers extracted at 130 °C under different oxalic acid concentrations: (a) OA 0% and (b) OA 2% observed at 500× magnification

In **Figure 5A**, fibers treated without oxalic acid (0%) exhibit a rough and irregular morphology, with a heterogeneous surface that retains debris and small particles adhering to it. This condition indicates suboptimal delignification, where lignin and hemicelluzlose have not been effectively separated from the cellulose structure. In contrast, **Figure 5B** shows that fibers treated with 2% oxalic acid undergo significant morphological changes, revealing a cleaner and more orderly structure. The fiber surfaces appear relatively smoother, with visibly reduced debris and impurities compared to the untreated sample. This morphological difference can be attributed to the action mechanism of oxalic acid as a delignifying agent. Through acid hydrolysis, oxalic acid breaks lignocellulosic bonds, facilitating the removal of lignin and hemicellulose from the cellulose fiber matrix and resulting in a higher cellulose content. H^+ ions easily penetrate and disrupt the amorphous regions, promoting their separation. Therefore, acid hydrolysis not only cleaves glycosidic bonds but also enhances cellulose purity by degrading the more labile amorphous domains (Neenu et al., 2022).

The cleaner and more regular fiber morphology observed with 2% oxalic acid treatment has positive implications for the mechanical properties of resulting composite materials. In a study by Kumar & Singh (2025), the researchers evaluated the effect of oxalic acid treatment on sisal fibers at various concentrations, including 2%. They found that this treatment improved fiber morphology, enhanced tensile strength, and increased the thermal stability and mechanical properties of the resulting bio-PBS composites. A smoother fiber surface allows for better adhesion with the polymer matrix, thereby improving stress transfer between the fiber and matrix, and potentially enhancing the overall mechanical strength of the composite.

This morphological transformation indicates that oxalic acid effectively dissolves non-cellulosic fractions and breaks down the complex lignocellulosic network, thereby facilitating the release of cellulose microfibrils (Sriruangrungkamol et al., 2020). When combined with the steam explosion process, oxalic acid enhances reagent penetration into the cell wall through swelling and vapor expansion mechanisms, leading to more thorough defibrillation (Chavez et al., 2023). These results are consistent with the increased crystallinity index observed in previous XRD analysis and support the claim that this treatment successfully removes a substantial portion of amorphous domains. Overall, the fiber morphology at 2% oxalic acid concentration exhibits a more open and organized structure, which directly impacts mechanical properties, matrix-binding capacity, and the potential use of the material in bio-composite formulations. The increased degree of defibrillation is also critical for microcrystalline and nanocellulose production, as it provides well-dispersed and homogeneous precursor fibers.

Pressurized nitrogen (N_2) consistently improved MCC extraction performance across all measured parameters, including cellulose content, lignin reduction, crystallinity index, and fiber morphology. The treatment with 2% oxalic acid under N_2 yielded the best results, achieving the highest CrI (66%) and the cleanest, most defibrillated surface structure. This confirms that nitrogen pressure facilitates delignification and helps preserve cellulose crystallinity. However, a trade-off was observed while crystallinity increased with higher acid concentrations, the cellulose yield did not rise proportionallylikely due to partial degradation or solubilization of polysaccharides. These results are also in line with the findings of Chavez et al. (2023), who observed that increasing oxalic acid concentrations beyond 8% provided minimal improvements in cellulose purity or crystallinity index. Their study reported CrI values of 52.37%, 52.92%, and 56.89% at 6%, 8%, and 12% oxalic acid, respectively, indicating diminishing returns at higher concentrations. This supports the use of low-concentration oxalic acid in our study (2%), which achieved a higher CrI of 66%, reinforcing its effectiveness as a mild and green pretreatment agent.

4. Conclusions

This study demonstrated the successful extraction of microcrystalline cellulose (MCC) from cabbage waste using a combination of high-speed blending, low-concentration oxalic acid hydrolysis, and steam explosion under nitrogen pressure. Among all treatments, the sample treated with 2% oxalic acid under pressurized nitrogen yielded the highest cellulose content (79.18%) and exhibited the highest crystallinity index (66%) along with clean, defibrillated fiber morphology. Compared to treatments without nitrogen or with lower acid concentrations, this condition significantly enhanced the removal of lignin and hemicellulose while preserving the integrity of the crystalline cellulose structure.

The integrated process offers clear green chemistry advantages, including the use of a weak organic acid instead of strong mineral acids, reduced chemical input, and minimal generation of hazardous waste. The use of nitrogen as an inert pressurizing agent also helps avoid oxidative degradation, thus enhancing process efficiency and product quality. These factors support the scalability and environmental safety of the method.

The resulting MCC exhibits qualities suitable for various high-value applications. In pharmaceuticals, it can serve as an excipient with excellent compressibility and binding properties. In materials engineering, its high crystallinity and surface morphology make it an effective reinforcement agent in biodegradable composites. Additionally, its microfibrillar structure and purity are promising for controlled drug delivery systems and barrier films in sustainable packaging. By utilizing agricultural waste as raw material and applying a low-impact processing strategy, this study contributes directly to waste valorization, pollution reduction, and resource-efficient manufacturing aligning with Sustainable Development Goals (SDGs)

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Statement

During the preparation of this work the authors used ChatGPT 4o and DeepL Translate in order to improve English language and proofread the text. After using this tool/service, the authors reviewed and edited the content as needed and take full responsibility for the content of the publication.

CRediT authorship contribution statement

Nesha Permata Syafira: Writing – review & editing, Writing – original draft, Visualization, Investigation, Formal analysis.

Bramantyo Airlangga: Validation, Resources, Investigation, Conceptualization, Supervision.

Sumarno: Validation, Resources, Investigation, Conceptualization, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The data that has been used is confidential.

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