

Research Article

Effect of High-Energy Ball-Milling Duration on the Physicochemical Properties of Chemically Treated MD2 Pineapple Peel Cellulose

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ABSTRACT

MD2 pineapple peel represents an abundant agro-waste rich in cellulose but remains underutilised in high-value applications. This study investigates the influence of ball-milling duration on the physicochemical properties of cellulose extracted from MD2 pineapple peel. The peel was chemically purified via alkaline delignification, bleaching, drying and high-energy ball milling for 0, 60, 90, and 120 min. The extraction yield of 10.6% (dry basis) was obtained using fresh peel with an initial moisture content of $83.4 \pm 1.1\%$. FTIR confirmed removal of lignin and hemicellulose, while XRD showed diffraction patterns consistent with cellulose I, with crystallinity peaking at 60 min before declining with prolonged milling. SEM revealed initial fibre fragmentation and size reduction at 60 min, followed by increased particle agglomeration with prolonged milling. TGA indicated that milled-extracted cellulose exhibited high thermal stability, comparable to that of commercial microcrystalline cellulose. Among the milling durations investigated, 60 min produced the highest measured crystallinity index (66.67%) and the smallest apparent Feret diameter ($193.3 \pm 46.6 \mu\text{m}$) among the extracted cellulose samples, while thermal stability remained comparable among the milled samples ($T_{\text{max}} = 354\text{--}355^\circ\text{C}$). These results highlight the influence of milling duration in refining processing conditions for the effective valorisation of pineapple peel into functional cellulose.

Key words: Ball-milling, cellulose, chemical treatment, MD2, pineapple peel, waste valorisation

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INTRODUCTION

The rapid growth of the pineapple industry has led to a substantial increase in both production and food processing. According to FAOSTAT, global pineapple production in 2023 reached approximately 29.64 million metric tons, with major contributions from Indonesia, the Philippines, and Costa Rica (FAOSTAT, 2026). Malaysia contributes a modest but growing share of global production, with its pineapple sector playing a key role in national agricultural transformation programs, supplying nearly 70% of fresh fruit and 95% of canned pineapple products for export (Ghazi *et al.*, 2023). Among the cultivated varieties, the MD2 pineapple, also known as “Gold” or “Honey Gold”, has become the dominant cultivar due to its sweet flavour, low acidity, juiciness, golden colour, and longer shelf life (Karimi Alavijeh & Zarei, 2025; Ong *et al.*, 2025).

With the growing demand for MD2 and its widespread cultivation, processing activities have increased accordingly. However, the rise in production results in substantial agro-industrial waste. This

waste is rich in sugars, moisture, and organic matter, making it prone to rapid microbial spoilage and leading to environmental issues such as odour, resource loss, and pollution if not properly managed (Rico *et al.*, 2020; Sarangi *et al.*, 2022). Among the various waste fractions in pineapple processing, the peel constitutes roughly 29–42% (w/w) of the total fruit mass and contains valuable biopolymers such as cellulose, hemicellulose, lignin, and pectin (Soni *et al.*, 2022; Jatav & Bhattacharya, 2024). Despite being discarded, the peel contains 20–25% cellulose (dry basis), a highly renewable material with excellent mechanical strength and biocompatibility, making it suitable for a wide range of sustainable applications (Dai *et al.*, 2018; Fan *et al.*, 2020; Putro *et al.*, 2023).

Cellulose is a linear polysaccharide composed of repeating β -1,4-d-glucopyranose units, where β -1,4-glycosidic bonds link each glucose monomer to form a long, unbranched chain (Khalid *et al.*, 2023; Abolore *et al.*, 2024). The strong hydrogen bonds formed between these chains produce a crystalline structure that contributes to the mechanical strength of plant cell walls.

However, within plant cell walls, cellulose is tightly bound within a lignocellulosic network of hemicellulose and lignin, making its isolation challenging. Conventional extraction methods typically rely on strong alkaline or oxidative reagents that can purify cellulose but often produce coarse particles with irregular structure and reduced crystallinity. The harsh chemical conditions also cause uneven fibre degradation, leading to structural defects and lower-quality cellulose (Sadeghi-Shapourabadi *et al.*, 2023; Macarez *et al.*, 2025). These methods are also energy-intensive and generate significant amounts of chemical waste, thereby limiting their environmental sustainability. These limitations have prompted interest in hybrid chemical-mechanical strategies, particularly high-energy ball milling.

High-energy ball milling is an effective mechanical technique for size reduction and has become widely adopted in industrial production due to its scalability across different processing capacities. In this technique, the material and grinding media are loaded into a rotating cylindrical chamber, in which centrifugal forces drive the balls into repeated impact and attrition, reducing particle size and altering the material's properties (Telidi & Salim, 2023; Sun *et al.*, 2024). For cellulose, high-energy ball milling not only reduces particle size but also disrupts crystalline regions, thereby increasing the amorphous content and influencing properties such as chemical reactivity and thermal stability. These structural modifications are strongly time-dependent; thus, milling duration is a key parameter to control. Controlling milling duration is essential to balance the reduction in crystallinity with the preservation of cellulose structural integrity and thermal stability (Mattonai *et al.*, 2018).

For pineapple peel, previous studies have applied ball milling primarily for cellulose isolation and nanofibrillation, rather than for systematic post-extraction modification. Zhang *et al.* (2022) employed NaOH-assisted ball milling, in which milling simultaneously promoted cellulose purification and nanofibrillation, while Wang *et al.* (2025) focused on the influence of component composition on the properties of pineapple peel cellulose nanofibrils prepared by NaOH-assisted ball milling. However, the influence of milling duration on the structural, morphological, and thermal properties of chemically extracted pineapple peel cellulose remains insufficiently investigated. Addressing this gap, the present study investigates the effect of high-energy ball-milling durations (0, 60, 90, & 120 min) on the physicochemical properties of chemically extracted MD2 pineapple peel cellulose using FTIR, XRD, SEM, and TGA, with commercial microcrystalline cellulose (MCC) as a reference material. The present study was limited to the selected milling durations under fixed milling conditions. The findings provide insight into how milling duration influences the physicochemical properties of pineapple peel-derived cellulose.

MATERIALS AND METHODS

Materials

MD2 pineapple peels were collected from a local market in Kuala Nerus, Terengganu, Malaysia. The initial moisture content of the peel was determined using the Association of Official Analytical Chemists method (AOAC, 2000). The chemical reagents used in the extraction process were sodium hydroxide (NaOH, reagent grade, ≥98%, Sigma-Aldrich, USA), glacial acetic acid (100%, EMSURE®, Supelco, USA), and sodium chlorite (NaClO₂, technical grade, 80%, Sigma-Aldrich, USA). Commercial microcrystalline cellulose (MCC) was obtained from Linghu Xinwang Chemical (Huzhou, China) for comparative analysis.

Extraction of cellulose

The peels were first washed with water to remove contaminants on the surface and then cut into small pieces before drying using

freeze-drying (Labconco, United States). Then, the dried peels were subjected to alkaline treatment following the method described by Pereira *et al.* (2021). The dried peels were immersed in a sodium hydroxide (4% w/v) solution at 70°C with a solvent-to-solid ratio of 1:20 in a beaker (500 mL). It was stirred mechanically at 500 rpm for one hr. The mixture was then filtered using a Buchner funnel (28 µm pore size) after the agitation period. The retained residue was washed with distilled water until neutral, then dried at 60°C for 24 hr. Subsequently, bleaching was performed on the alkaline-treated dried peels as described by Madureira *et al.* (2018). Briefly, the dried peels were treated in an acetate buffer-sodium chlorite solution at 80°C for 6 hr, rinsed with distilled water until neutral pH, and then oven-dried at 50°C for 12 hr. Figure 1 shows a diagram of the process for cellulose extraction from pineapple peel waste.



Fig. 1. Process for cellulose extraction from pineapple peel waste.

Extraction yield determination

The cellulose extraction yield (Y_C , in %) was gravimetrically estimated based on the sample weight before and after the chemical treatment process. The yield was calculated using Equation 1.

$$\text{Yield, } Y_C(\%) = \frac{\text{weight of dried extracted cellulose (g)}}{\text{weight of dried waste (g)}} \times 100\%$$

Equation 1

High-energy ball-milling process

The dried extracted cellulose was first pulverised using a commercial blender to obtain coarse powder prior to milling. High-energy ball milling was then performed using a BM40 mill (POWTEQ, China) to obtain superfine cellulose powder. Milling was conducted at a ball-to-powder ratio set at 10:1 for durations of 0, 60, 90, and 120 min at a constant speed of 400 rpm. The 10:1 ball-to-powder ratio was selected based on previous ball-milling treatment of cellulose (Zheng *et al.*, 2018), while the milling speed was selected based on previous studies on pineapple peel-derived cellulose (Lv *et al.*, 2022; Zhang *et al.*, 2022). These parameters were kept constant to evaluate the effect of milling duration.

Characterisation of extracted MD2 cellulose

Fourier transform infrared (FTIR) spectroscopy

FTIR (Bruker Platinum ATR FTIR, Germany) was carried out to identify the functional groups present in the extracted cellulose at different ball-milling durations. The analysis was performed using an FTIR spectrophotometer, equipped with an ATR accessory. The spectra were recorded in the range of 4000 to 400 cm⁻¹, with a resolution of 4 cm⁻¹ (Tarchoun *et al.*, 2019).

X-ray diffraction (XRD)

The crystallinity of the extracted cellulose at different ball-milling durations was analysed using X-ray diffraction (XRD, Rigaku SmartLab, Japan) equipped with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$) operated at 40 kV and 30 mA. Diffraction patterns were recorded over a 2θ range of 5–40° at a scan rate of 0.02°/sec. The Crystallinity Index (CrI) was calculated using the peak-area method, in which the integrated area of the crystalline peaks is divided by the total integrated area of the diffraction pattern (Mamudu *et al.*, 2025). This approach allows differentiation between the crystalline and

amorphous regions of the cellulose. The CrI was determined using Equation 2.

$$\text{Crystallinity Index, CrI (\%)} = \frac{\text{Area under crystalline peaks}}{\text{Area under all peaks}} \times 100$$

Equation 2

Scanning electron microscopy (SEM)

The surface morphology of the extracted cellulose at different ball-milling durations was examined using a high-vacuum scanning electron microscope (Tescan Orsay Holding, Brno, Czech Republic) (Ahmima *et al.*, 2024). Before imaging, the samples were sputter-coated with a thin layer of gold to improve electrical conductivity. The coated samples were mounted on metal stubs, and the micrographs were captured under a vacuum at an accelerating voltage of 10–15 kV. Quantitative particle size analysis (Feret diameter) was performed on the acquired micrographs using the open-source software ImageJ (National Institutes of Health, Bethesda, MD, USA) (Li *et al.*, 2024).

Thermogravimetric analysis (TGA)

The thermal stability and decomposition behaviour of the cellulose samples at different ball-milling durations were evaluated using a TGA/DSC 1 STAR System (Mettler Toledo, Switzerland). Approximately 5 mg of each sample was placed in a ceramic crucible and heated from room temperature to 600°C at a heating rate of 10°C/min under a nitrogen flow of 60 mL/min (Reddy *et al.*, 2018).

RESULTS AND DISCUSSION

MD2 cellulose extraction yield

The extraction yield was evaluated based on the percentage mass retention across each treatment step, with the fresh MD2 pineapple peel obtained at an initial moisture content of $83.4 \pm 1.1\%$ (w.b.). This result is within the range reported in previous studies on pineapple peel (Mala *et al.*, 2024; Kumaresan *et al.*, 2025). After alkaline treatment, 19% of the initial dry peel mass remained, indicating substantial removal of soluble and non-cellulosic components during delignification. Subsequent bleaching further reduced the retained mass to 10.6%, representing the final yield of the extracted cellulose material. The yield obtained was within the range reported for pineapple peel cellulose, ranging from 8.4% to 23% (Dai *et al.*, 2017; Sitorus *et al.*, 2024). Compared with other lignocellulosic wastes, cellulose yields of 79–81.4% for wheat straw (Qasim *et al.*, 2020), 18.4% for sugarcane bagasse (Rezvani *et al.*, 2025), and approximately 11–18% for banana pseudostem using NaOH-based extraction methods (Ardila A. *et al.*, 2024). These variations may be attributed to differences in the initial lignocellulosic composition of the biomass and the extraction conditions employed.

FTIR analysis

The FTIR spectra of the milled cellulose samples, presented in Figure 2, show the characteristic functional group vibrations of cellulose and align closely with those observed for commercial MCC. The major absorption bands and the functional groups are listed in Table 1. Prominent peaks appeared around 3334 cm⁻¹ (O–H stretching), 2910 cm⁻¹ (C–H stretching), and 1022 cm⁻¹ (C–O–C stretching of glycosidic bonds), confirming the polysaccharide backbone typical of cellulose. Additional peaks at 1430 cm⁻¹ (CH₂ bending) and 896 cm⁻¹ (C–H deformation of β-glycosidic linkage) were observed, which are commonly associated with Cellulose I structures. The absence of a peak around 1730 cm⁻¹, typically attributed to carbonyl groups in hemicellulose and lignin, further indicated successful removal of non-cellulosic components (Vishnoi *et al.*, 2024). This signifies the efficiency of the alkaline and bleaching treatments in delignifying the sample.

Notable differences in peak sharpness and intensity were observed between the milled cellulose and commercial MCC, particularly in the regions around 1022 cm⁻¹ and 896 cm⁻¹. Commercial MCC

exhibited sharper and more defined peaks, indicative of its higher crystallinity and uniform structure, whereas milled samples showed broader peaks, reflecting increased amorphous content due to mechanical disruption during ball milling. Similar observations were reported by Zhao *et al.* (2024), where the peak at 896 cm⁻¹ became more intense after ball milling, indicating an increase in amorphous cellulose. The band is associated with C1 vibrations of the β-glycosidic linkages in cellulose. The mechanical impact and shear forces generated during ball milling can disrupt intra- and intermolecular hydrogen bonding and the ordered arrangement of cellulose chains, thereby promoting structural disorder and the formation of amorphous regions (Yang *et al.*, 2020). Additionally, the water absorption band near 1638 cm⁻¹ was more pronounced in milled samples, suggesting greater moisture retention compared to MCC (Tarchoun *et al.*, 2019). Among the milled samples, the 60 min sample exhibited an FTIR profile most similar to that of commercial MCC.

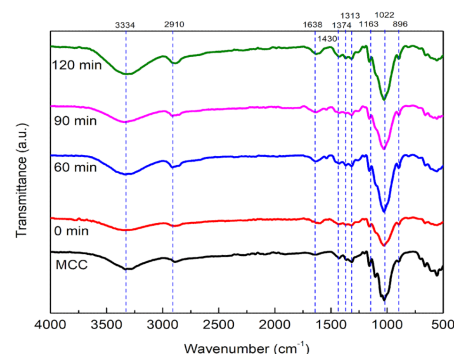


Fig. 2. Comparison of FTIR spectra for extracted cellulose at different ball milling durations and commercial MCC.

XRD analysis

The diffractograms of the cellulose milled at different durations and the commercial MCC are illustrated in Figure 3, while the corresponding crystallinity index (CrI) values are summarised in Table 2. From the results, all cellulose samples showed diffraction peaks at approximately $2\theta \approx 21^\circ$ – 22° , which correspond to the (200) plane of Cellulose Iβ, as confirmed by JCPDS card no. 00-060-1502. The presence of the (200) lattice plane is a typical feature of the native cellulose structure (Anwar *et al.*, 2024). MCC exhibited the sharpest and most intense peak at 21.52°, reflecting its high crystallinity (CrI = 74.69%). In contrast, the milled samples displayed broader diffraction features than commercial MCC, indicating differences in crystalline ordering following mechanical treatment.

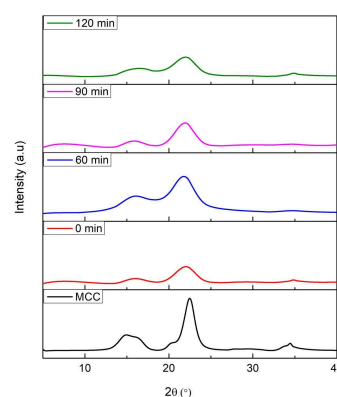


Fig. 3. XRD diffractograms of extracted cellulose at different milling durations and commercial MCC.

Table 2. XRD peak positions, maximum intensity, and plane of cellulose samples at different ball milling times.

Sample	2 θ (°)	Intensity	Plane (h k l)	Crystallinity index (%)
MCC	21.52	9323	2 0 0	74.69
0 (unmilled)	21.29	5907	2 0 0	53.33
60 min milled	21.17	15916	2 0 0	66.67
90 min milled	21.53	9780	2 0 0	64.71
120 min milled	21.24	8024	2 0 0	58.97

Table 3. Thermal degradation parameters of extracted cellulose at different milling times compared to commercial MCC.

Sample	Initial Weight Loss (%)	T _{max} (°C)	Residue at 600°C (%)
MCC	3.0	358	9.1
0 (unmilled)	5.3	356	22.9
60 min milled	6.1	355	11.0
90 min milled	6.2	355	17.5
120 min milled	6.4	354	18.8

The CrI of the unmilled cellulose was 53.33%, increasing to 66.67% at 60 min, before declining to 64.71% and 58.97% at 90 and 120 min, respectively. This pattern suggests that milling initially increased the measured CrI at 60 min, whereas prolonged milling was associated with a subsequent decrease in CrI. This trend is consistent with the studies by Mattonai *et al.* (2018), which observed a dramatic decrease in CrI after extended milling, and similar findings in MCC, showing a decline in CrI and morphological changes with increasing milling time. Additionally, minor peaks were observed around 15° and 35°, which may be attributed to the (110) and (004) planes of Cellulose I β . The occurrence of these peaks suggests partial disordering or disruption of the cellulose chains, possibly induced by the mechanical ball milling process (de Souza Fonseca *et al.*, 2019). No new reflections attributable to cellulose II were observed, and the diffraction patterns remained consistent with cellulose I. Among the milled samples, the 60 min sample exhibited the highest CrI and the diffraction profile closest to that of commercial MCC.

SEM analysis

SEM micrographs revealed clear differences in surface morphology between commercial MCC and extracted cellulose samples at different milling durations, as shown in Figure 4. The commercial MCC sample exhibited small, angular and uniformly shaped

particles with a dense, aggregated morphology, consistent with its high crystallinity and industrial processing. In contrast, the unmilled pineapple peel cellulose sample showed coarse, fibrous particles with irregular edges and loosely packed structures. As milling time increased, the cellulose surface morphology became progressively disordered. The 60 min milled samples showed clear fibre flattening and fragmentation, resulting in smaller individual fragments. However, continued milling at 90 and 120 min resulted in pronounced agglomeration, in which fragmented particles appeared to reassociate into larger, heterogeneous structures. This observation was further supported by the SEM image-based Feret-diameter distributions presented in Figure 5. Specifically, image-based measurements showed that the mean Feret diameter decreased from $246.9 \pm 84.7 \mu\text{m}$ for the unmilled sample to $193.3 \pm 46.6 \mu\text{m}$ after 60 min of milling. However, at prolonged milling durations, the apparent particle dimensions increased to $395.9 \pm 109.9 \mu\text{m}$ and $415.2 \pm 142.9 \mu\text{m}$ at 90 and 120 min, respectively, which may be attributed to the increased agglomeration observed in the SEM micrographs. In comparison, commercial MCC exhibited a considerably smaller mean Feret diameter of $74.9 \pm 30.6 \mu\text{m}$. The number of measurable particles at 90 and 120 min was limited ($n=14-15$) because pronounced agglomeration made individual particle boundaries difficult to distinguish.

Table 1. FTIR absorption bands and corresponding functional groups of extracted cellulose.

Wavenumber (cm ⁻¹)	Functional Group	Vibration Mode
3334	O-H	Hydrogen bond stretching
2910	C-H	Aliphatic C-H stretching
1638	H ₂ O (bound)	O-H bending of absorbed moisture
1430	CH ₂	Bending
1374	C-H	Symmetric bending
1313	C-C	Stretching
1163	C-O	Asymmetric stretching
1022	C-O-C	Glycosidic linkage stretching
896	β -glycosidic linkage	C-H deformation, C1 group vibration

These morphological changes indicate effective defibrillation and size reduction during the initial stage of milling, followed by secondary agglomeration at prolonged milling durations. Previous studies similarly reported that prolonged mechanical treatment disrupts fibril continuity and shortens fibre length, which may contribute to particle agglomeration (Hernández-Varela *et al.*, 2021; Valášek *et al.*, 2021). These morphological changes may influence particle-matrix interaction in applications such as bio-composites and packaging (Oesef *et al.*, 2024).

TGA analysis

The TGA curves, shown in Figure 6, illustrate differences in thermal behaviour between MCC and the extracted cellulose as a

function of milling duration. MCC exhibited the highest thermal stability, decomposing at a higher temperature with a sharper weight-loss transition, reflecting its higher crystallinity. In contrast, the extracted cellulose showed progressively broader degradation profiles as milling time increased. Table 3 summarises the corresponding initial weight loss, peak degradation temperatures and residues at 600°C for each sample. The commercial MCC showed the highest T_{max} (358°C), whereas the extracted cellulose samples exhibited slightly lower T_{max} values of 354–356°C.

When compared with cellulose isolated from other biomass sources, the T_{max} values obtained in this study are relatively high, exceeding

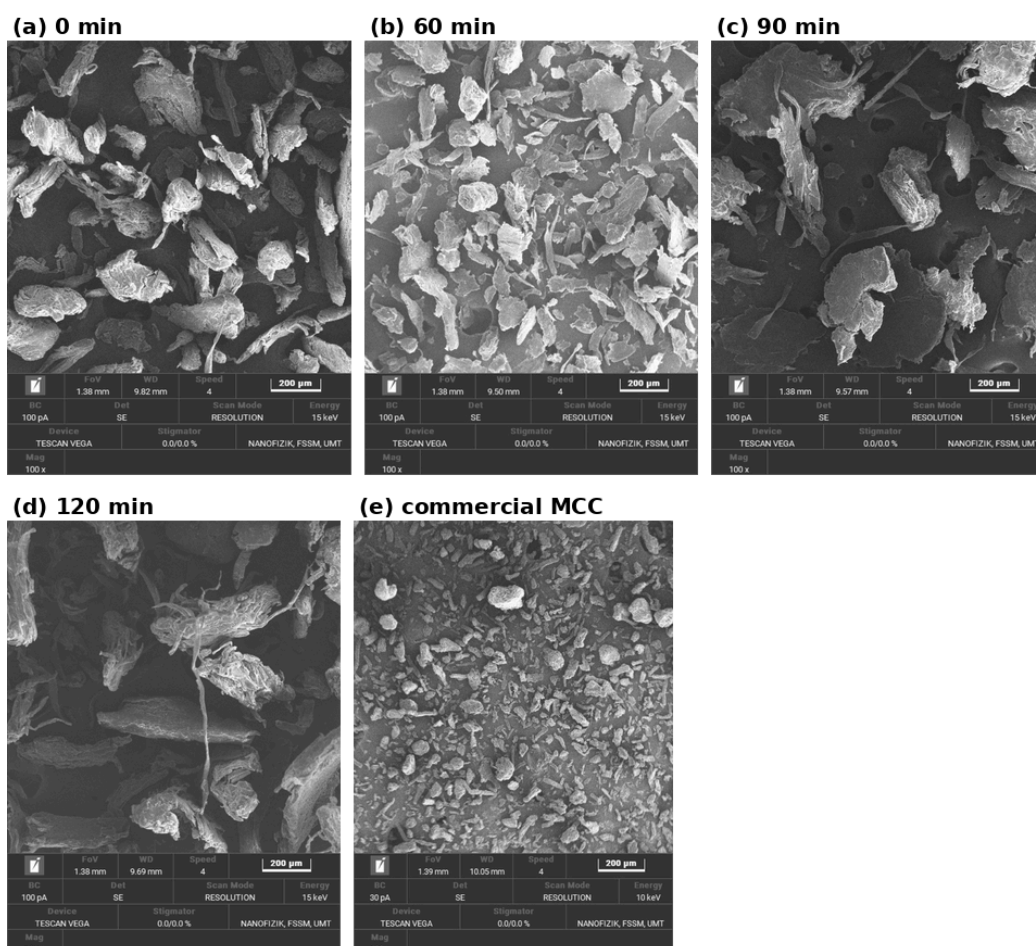


Fig. 4. SEM micrographs of cellulose samples at 100× magnification with different milling durations: (a) 0 min (unmilled); (b) 60 min; (c) 90 min; (d) 120 min; and (e) commercial MCC.

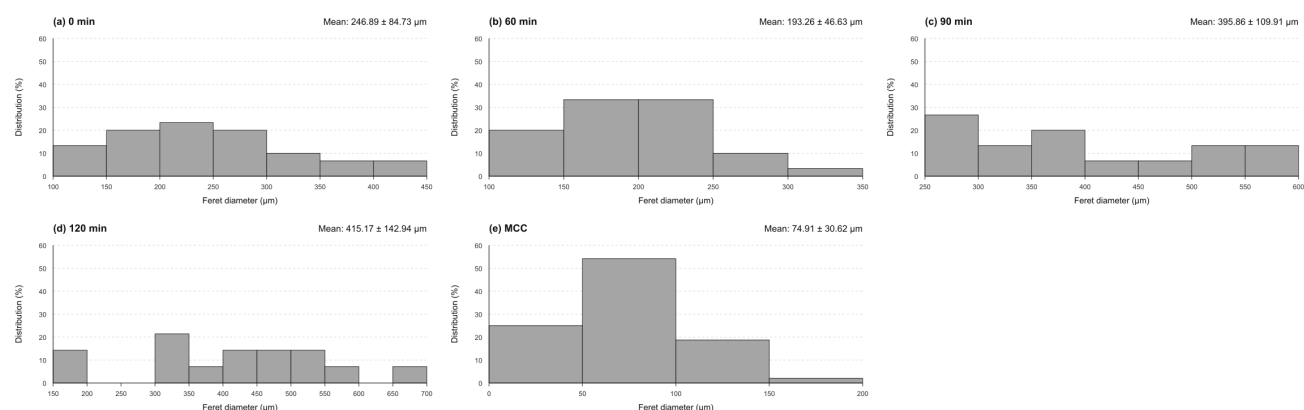


Fig. 5. Relative-frequency distributions of the Feret diameters of cellulose samples subjected to different milling durations: (a) 0 min (unmilled); (b) 60 min; (c) 90 min; (d) 120 min; and (e) commercial MCC.

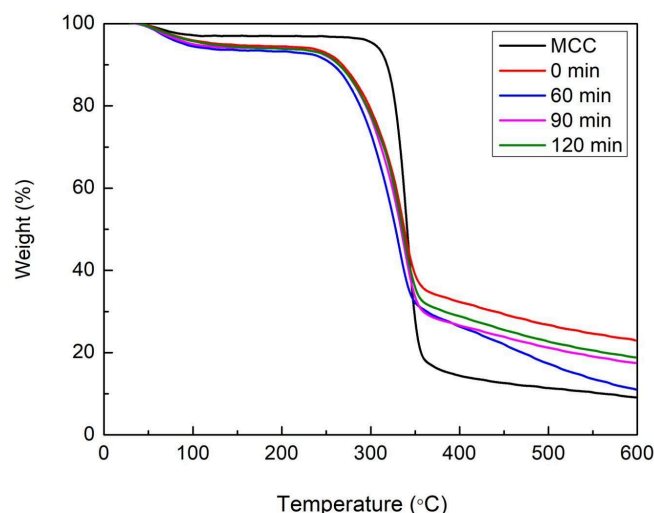


Fig. 6. TGA thermograms of cellulose samples with different milling times compared to MCC.

those reported for lotus seedpod (296.5°C) (Duc *et al.*, 2026), spent coffee grounds (315°C) (Naghdishi & Chackoshian Khorasani, 2025), kenaf stems (326°C) (Khiari *et al.*, 2020), and pepper-waste MCC (345.6°C) (Holilah *et al.*, 2021). These values remain within the characteristic decomposition range of cellulose, which typically begins around 270–300°C and peaks at 350–370°C (Kristanto *et al.*, 2021; Sygula *et al.*, 2025). This indicates that the extracted cellulose from pineapple peel retains comparatively high thermal stability.

Increasing milling time resulted in a slight decline in T_{max} from 356°C for the unmilled cellulose to 354°C after 120 min of milling, consistent with structural disruption induced by prolonged mechanical impact (Khaghani-Dehaghani *et al.*, 2011; Tejedor *et al.*, 2018). Residue at 600°C was higher in all extracted samples (11.0–22.9%) compared to MCC (9.1%), suggesting the presence of thermally resistant non-cellulosic components. Among the milled samples, cellulose milled for 60 min exhibited a T_{max} of 355°C, close to that of commercial MCC (358°C).

CONCLUSION

This work demonstrated the extraction and modification of cellulose from MD2 pineapple peel via a sequential process of alkaline delignification, bleaching, drying, and ball milling, yielding a final extraction yield of 10.6% of the initial dry peel mass. FTIR confirmed the characteristic functional groups of cellulose and supported the effective removal of lignin and hemicellulose, while XRD provided evidence of its crystalline structure. XRD showed that the diffraction patterns remained consistent with cellulose I across all milling durations, with no new reflections attributable to cellulose II. The crystallinity index increased from 53.33% in the unmilled cellulose to 66.67% after 60 min of milling, then decreased to 64.71% and 58.97% at 90 and 120 min, respectively. This decrease at prolonged milling durations indicated increasing disruption of the ordered cellulose structure due to continued mechanical treatment. SEM analysis further demonstrated the morphological effects of milling, showing that milling initially promoted fibre fragmentation and size reduction, with the mean Feret diameter decreasing from $246.9 \pm 84.7 \mu\text{m}$ to $193.3 \pm 46.6 \mu\text{m}$ at 60 min. However, prolonged milling promoted particle agglomeration, resulting in a higher measured apparent Feret diameter. TGA analysis showed comparatively high thermal stability, with T_{max} values of 354–356°C for the extracted cellulose, close to that of commercial MCC (358°C). Among the milling durations investigated, 60 min produced the highest measured

crystallinity index and the smallest apparent Feret diameter among the extracted cellulose samples, while thermal stability remained comparable across the milled samples. These findings demonstrate the potential of MD2 pineapple peel as a source of cellulose and highlight the importance of controlling milling duration to achieve the desired physicochemical properties. However, quantitative compositional analysis of cellulose, hemicellulose, and lignin was not performed on the raw and treated samples. Future research should address this limitation, further refine the extraction process to improve yield and minimise chemical consumption, and assess the performance of the resulting cellulose in composite and barrier film applications.

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ETHICAL STATEMENT

Not applicable.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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