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Article

# Cellulose Microfibres from Durian Waste Using Modified Steam Explosion as Pre-Treatment

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**Abstract:** This study presents a steam explosion (SE) system as an effective pretreatment method for efficiently extracting cellulose microfibres (CMFs) from durian rind, which makes up a significant portion of the fruit (60–75%) and is often discarded as waste. It represents a significant agricultural waste resource of lignocellulosic biomass. SE pretreatment, optimized at 145 °C for 5 minutes on dried durian rind, significantly increased CMF recovery from 34.01% to 63.6%, exceeding typical yields reported in the literature for similar biomass sources. Comprehensive analyses confirmed the quality and structural integrity of the extracted cellulose. X-ray diffraction (XRD) identified the crystalline structure as cellulose type I, while Fourier transform infrared spectroscopy (FTIR) detected characteristic cellulose peaks, affirming its chemical purity. Thermogravimetric analysis (TGA) demonstrated high thermal stability, with decomposition commencing at approximately 340 °C, consistent with pure cellulose. Field emission scanning electron microscopy (FESEM) revealed a smoother surface, refined fibre morphology, and a notable reduction in fibre diameter in SE-treated samples. These findings underscore the effectiveness of the SE system in enhancing CMF yield and purity, offering a sustainable, scalable approach for cellulose extraction from biomass. This work highlights the potential of SE technology as a transformative tool for valorising lignocellulosic waste, supporting its application in biocomposites, biofuels, and nanocellulose-based products.

**Keywords:** durian rind; biomass; steam explosion; cellulose microfibres; extraction

## 1. Introduction

The rising global demand for sustainable materials has generated considerable interest in renewable biomass resources, particularly lignocellulosic biomass, due to its availability, economic feasibility, and environmental advantages. Composed mainly of cellulose, hemicellulose, and lignin, this biomass is a promising resource for extracting cellulose microfibres (CMFs), which have a wide range of applications in industries like paper manufacturing, biocomposites, and pharmaceuticals. While various biomass sources have been explored, durian rind, a commonly discarded agricultural waste, is underutilized despite its significant cellulose content. This study is the first to apply steam explosion (SE) pretreatment to durian rind, providing new insights into the potential of this underutilized biomass as a sustainable feedstock for cellulose production (Silveira et al., 2015; Auxenfans et al., 2017; Jovičić et al., 2022). The efficient extraction of cellulose from lignocellulosic sources is indeed a complex process due to the intricate structure of plant cell walls composed of cellulose, hemicellulose, and lignin. The effective separation and extraction of each component necessitate the breakdown of all constituents, as the strong interactions between these polymers contribute to the overall recalcitrance of lignocellulosic biomass (Lee, Hamid and Zain, 2014; Deepa et al., 2015). However, extracting CMFs from lignocellulosic biomass necessitates careful optimization to ensure the effective isolation of cellulose while preserving its structural integrity for advanced applications like biocomposites and biofuels. Steam explosion (SE) has emerged as a promising technique among the various methods available. Steam explosion (SE) has emerged as a promising technique for the pretreatment of biomass. By employing elevated temperatures and pressures, SE disrupts the complex structure of lignocellulosic materials, effectively hydrolyzing hemicellulose and lignin while maintaining the integrity of cellulose (Ouyang, Chen, Zhang, Yuan, W Wang, et al., 2018). This challenge has prompted the development of innovative techniques to enhance cellulose extraction yield while preserving fibre quality. Among the various methods explored, SE has emerged as a promising pretreatment technique for improving CMF accessibility in biomass. The application of steam explosion is particularly relevant in regions with abundant lignocellulosic resources, as it provides a sustainable method for biomass valorisation (Chaib et al., 2024).

Steam explosion (SE) is a thermomechanical process that modifies the lignocellulosic structure by subjecting biomass to high-temperature steam, followed by a rapid pressure release. This method disassembles the lignocellulosic structure, facilitating the breakdown of hemicellulose while predominantly preserving cellulose, depending on the intensity of the SE pretreatment (Ziegler-Devin, Chrusciel and Brosse, 2021). The strength of the steam explosion is crucial because it helps break down the lignocellulosic structure, facilitating the removal of components, mainly cellulose, while minimizing structural changes (Pielhop et al., 2016; Liu et al., 2019; Obame et al., 2019). Studies indicate that an intense steam explosion can enhance cellulose accessibility for enzymatic hydrolysis, a crucial process for transforming biomass into fermentable sugars (Kang et al., 2013; Asada et al., 2015). Research demonstrates that optimal conditions for steam explosion can significantly enhance the yield of fermentable sugars from lignocellulosic biomass, making it an advantageous pretreatment technique in biorefineries (K. Wang et al., 2020; Nader et al., 2022). Moreover, the steam explosion is particularly relevant in regions like Southeast Asia, where abundant lignocellulosic materials, such as durian rind, remain underutilized despite their potential (Heikkinen et al., 2014; Gamay et al., 2024).

Durian rind is a commonly available but frequently discarded source of lignocellulosic biomass, as shown in Figure 1. The rind represents a significant agricultural waste stream comprising 60–75% of the fruit. The edible portion of the durian fruit constitutes only a tiny proportion, generally ranging from 15–30%, while the remainder is typically regarded as biomass waste (Ngabura et al., 2018). Known as the "King of Fruits," durian is highly popular in Southeast Asia, with approximately 2.3 million metric tonnes harvested between 2015 and 2017 (Setyaningrum et al., 2021; Thorogood et al., 2022). Disposing of durian rind poses significant challenges, particularly in landfill sites, where limited space exacerbates waste management issues (Obeng et al., 2021; Payus et al., 2021). Furthermore, the rind of the durian is rich in cellulose, containing 31.6% cellulose (Cui et al., 2021). The high cellulose content makes them suitable feedstocks for the sustainable production of value-added products. Durian rind is a notable lignocellulosic biomass resource that can improve sustainable waste management and resource utilisation, particularly in creating value-added goods (Masrol et al., 2017).


***Figure 1.*** Discarded durian rinds are thrown in a large waste bin.

Steam explosion (SE) pretreatment is an effective technique for enhancing cellulose extraction from agricultural biomass, namely durian rind. Nonetheless, the literature reveals considerable study gaps in the utilisation of SE, notably with durian rind, especially when contrasted with other pretreatment techniques. This systematic literature review seeks to identify deficiencies, concentrating on three main areas: (1) the limited application of SE on durian rind compared to other pretreatment methods, (2) the lack of quantitative comparisons between SE and alternative techniques concerning cellulose production, and (3) the scalability and industrial feasibility of SE for large-scale biomass processing.

The literature suggests that the utilisation of SE on durian rind is restricted relative to alternative pretreatment techniques. While SE has been extensively researched for various biomass types, such as corn stover and sugarcane bagasse, its application in durian rind is yet insufficiently explored (Gao et al., 2021). Halysh et al. underscore that SE is an environmentally sustainable technique that significantly reduces biomass recalcitrance, thereby enhancing cellulose accessibility. However, they do not provide specific data on durian rind, indicating a lack of targeted research. Similarly, while studies on diverse agricultural residues demonstrate the effectiveness of SE in improving cellulose extraction, the unique characteristics of durian rind, such as its high lignin content and specific structural properties, necessitate further investigation (Imlauer-Vedoya et al., 2019).

Moreover, many studies have shown how effectively steam explosion (SE) improves cellulose extraction from various lignocellulosic materials. Sharma et al. for example, compared rice straw and noted that SE produced a cellulose content of 52.7%, which is higher than the yield attained with organosolv pretreatment (46.9%). This emphasizes SE's potential as an ideal approach to increase cellulose accessibility. Likewise, D'Orsi et al. highlighted SE's adaptability across several biomass sources by demonstrating its effectiveness in separating highly digestible cellulose and pure lignin from Cynara carduncutus.

Although SE clearly demonstrates benefits in terms of cellulose output, other pretreatment techniques, such as alkaline and acid treatments, have also been closely examined. Using SE in combination with hydrotropic extraction, Olsson et al. investigated the progressive fractionation of lignocellulosic components in hardwood, demonstrating that although SE is effective as a stand-alone method, integrating it with other pretreatment techniques can enhance overall efficiency. This need for optimization is further underscored by Seidel et al., who emphasized the importance of optimizing SE parameters to maximize hydrolysis yields, highlighting the significant reliance of SE efficacy on biomass type and treatment conditions.

Despite these encouraging results, much more research is needed on the use of SE for durian rind or similar fruit waste. Further studies are necessary to ascertain the feasibility and optimization of SE for this biomass, considering the unique structural qualities and high lignin concentration of durian rind.

The scalability and practical viability of SE for large-scale biomass processing remain essential subjects for additional research. While SE is recognised for its low energy consumption and little chemical use, the shift from laboratory-scale research to commercial applications poses issues that are insufficiently addressed in the literature (Mulat et al., 2018; Steinbach et al., 2020). Steinbach et al. highlight the efficacy of SE as a pretreatment for the acid hydrolysis of lignocellulosic biomass, although they neglect to discuss the scalability of the approach. Similarly, while Abelha and Cieplik research highlights the effectiveness of SE in biomass processing, it neglects to evaluate the practical implications of deploying SE on an industrial scale The inadequate focus on scalability and practicality signifies a need for more comprehensive research to assess the economic and logistical aspects of adopting SE in durian rind and other agricultural byproducts.

Hence, steam explosion pretreatment shows potential for cellulose extraction from agricultural biomass, considerable research gaps persist regarding its applicability to durian rind. The limited research on durian rind, the absence of quantitative comparisons with other pretreatment procedures, and the challenges related to scaling and industrial applicability highlight the need for additional exploration. Alleviating these limits will be crucial for optimising SE pretreatment and enhancing the overall efficiency of cellulose extraction from durian rind and related agricultural waste.

This study systematically investigates the efficacy of SE in enhancing CMF extraction from durian rind. The primary objectives are to examine potential applications, structural and chemical alterations in CMF, and the effectiveness of SE-treated versus non-SE-treated samples. The study's findings contribute to the growing comprehension of sustainable material production and biomass valorisation. The work is organised as follows: Section 2 presents the methodology; Section 3 delineates the results; Section 4 concludes with implications and prospective avenues for research.

## 2. Materials and Methods

### *2.1. Materials*

Various varieties of Durian rinds (Figure 3) were procured from a roadside stall in Bangi, Selangor, Malaysia. Each rind was individually pulverised to achieve a particle size of less than 0.5 cm. The pulverised material was subsequently dried in an oven at 60 °C for 48 hours. The chemicals utilised in the study included 95% sulfuric acid (AR grade) sourced from Chemiz, Malaysia; 99% pure sodium hydroxide obtained from R&M Chemicals (KSFE, Malaysia); and 80% technical grade sodium chlorite acquired from Sigma-Aldrich. All chemicals were utilised as received without further purification, and distilled water was consistently employed throughout the experimental procedures.

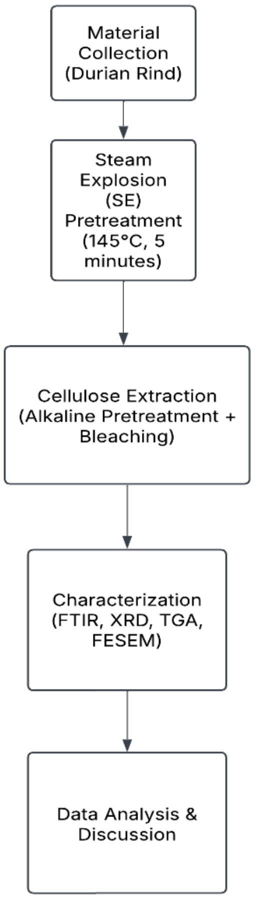


***Figure 2.*** Schematic flow diagram of the cellulose microfibres extraction process from durian rind. The process includes material collection, steam explosion (SE) pretreatment, cellulose extraction using NaOH and bleaching, and characterisation through FTIR, XRD, TGA, and FESEM analyses.

## *2.2. Research Methodology Flow Diagram*

Figure 2 presents an overview of the experimental process. The flow diagram visually summarizes the steps undertaken, including material collection, steam explosion pretreatment, cellulose extraction, and the subsequent characterization of cellulose microfibers (CMF).

## *2.3. SE Pretreatment of Durian Rinds*

The SE pretreatment was conducted in a custom-built steam reactor, designed with a 3-kW steam generator and a valve control system for rapid pressure release. The selected parameters (145 °C for 5 minutes) were based on preliminary experiments, which identified these conditions as optimal for maximizing cellulose yield while minimizing thermal degradation. The reactor's pressure capacity (up to 0.45 MPa) and operational specifications ensure precise control over the steam explosion process, which is essential for maintaining biomass quality. Approximately 40 grams of pulverised durian rind were loaded into the reactor, which was preheated until it achieved an internal pressure of 0.45 MPa. At this juncture, the residence time was established at 5 minutes. Subsequently, the valve was opened to effectuate a rapid pressure release to atmospheric levels, initiating the explosion effect. The treated samples were collected, allowed to cool to ambient temperature, and dried at 60 °C for 48 hours before further analysis. This SE pretreatment effectively disrupted the lignocellulosic matrix, enhancing cellulose accessibility and improving the efficiency of subsequent extraction processes.

## 2.4. *Extraction of CMF from Durian Rinds*

After SE pretreatment, the durian rind underwent cellulose purification to eliminate lignin and hemicellulose, following a modified method by Abdul Rahman et al. (2017). A 4% sodium hydroxide (NaOH) solution was used, with a sample-to-solution ratio of 1:25. The mixture was heated to 100 °C and stirred for 3 hours. The samples were then rinsed thoroughly with distilled water to remove any residual NaOH, and this rinsing process was repeated three times for complete removal. The subsequent bleaching step involved preparing an acetate buffer by mixing NaOH and acetic acid and adding a chlorite solution in a 1:1 ratio. The samples were immersed in the bleaching solution at a sample-to-solution ratio of 1.25:1, heated to 80 °C, and maintained at this temperature for 4 hours. After bleaching, the samples were rinsed thoroughly with distilled water to eliminate residual bleaching agents, with this rinsing step also repeated three times to ensure complete purification.

## 2.5. *Analysis of Extraction Yield*

The yield of extracted CMF was calculated by measuring the mass of dried cellulose obtained from durian rinds using the equation:

$$\text{CMF Extraction yield (\%)} = \frac{\text{Weight of extracted CMF (g)}}{\text{Weight of dried sago bark or durian rinds (g)}} \times 100 \qquad (1)$$

## 2.6. *Characterization of CMF*

### 2.6.1. Chemical and Structural Analysis (Van Soest, FTIR & XRD)

The raw durian rind and SE-treated durian rind samples were analysed for chemical composition to determine their cellulose, holocellulose, and lignin content. The standard Van Soest method was employed, involving analyses of neutral detergent fibre (NDF), acid detergent fibre (ADF), and acid detergent lignin (ADL) (Han et al., 2007).

The FTIR spectra of the extracted CMF were acquired utilising the ATR technique with a Spectrum 400 Cary 630 FTIR spectrometer (Agilent Technologies). The spectral range was established from 4000 to 650 $cm^{-1}$, with a resolution of 2 $cm^{-1}$ and 64 scans conducted. The spectra were smoothed and baseline-corrected utilising the instrument's software and subsequently exported as CSV files to OriginPRO 2024 for spectral categorisation.

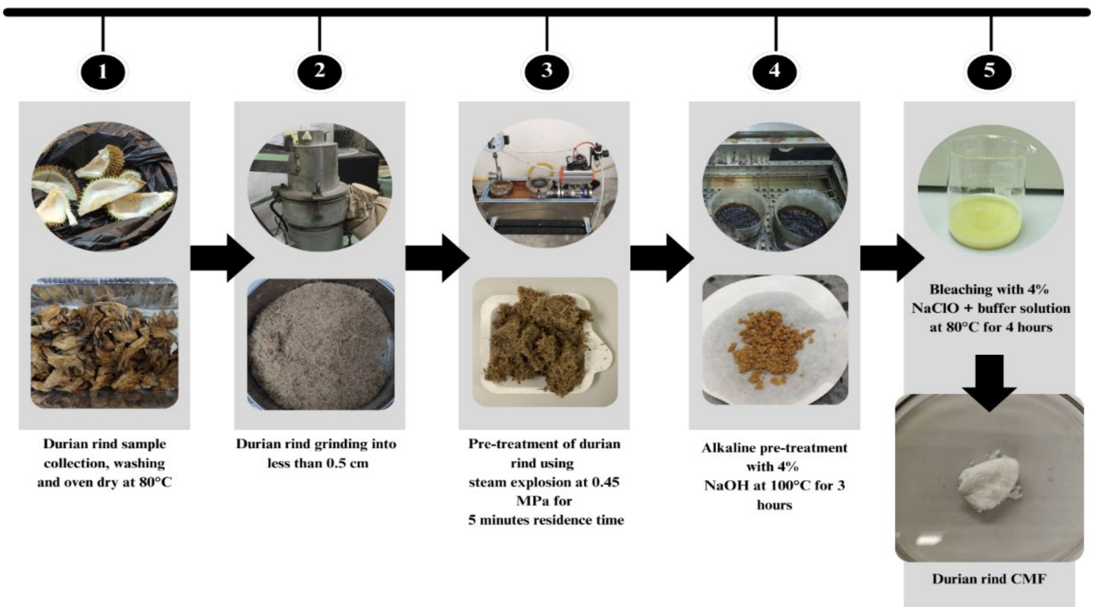


***Figure 3.*** Schematic representation of the extraction process of cellulose microfibres (CMF) from durian rind.

The X-ray diffraction pattern of CMF was characterised using a Bruker D8 Advance diffractometer from Germany. The sample was placed on a 2 × 2-inch glass slide and secured with tape on the X-ray platform. Analysis was conducted with Cu Kα radiation at a current of 30 mA and a voltage of 40 kV. The sample was scanned over a 2θ range of 4° to 35°, with each scan lasting 30 minutes. The crystallinity index (CrI) was calculated using the Segal equation (Segal et al., 1959):

$$\mathrm{CrI} = 100 \times \frac{(I_{200} - I_{am})}{I_{200}} \quad (2)$$

where I200 represents the diffraction intensity at 2θ = 22–23º and Iam is the minimum diffraction intensity at 2θ = 18–20º.

### 2.6.2. Thermal and Morphological Analysis (TGA & FESEM)

A Simultaneous Thermal Analyser (STA) assessed the thermal degradation behaviour. About 10 mg of the sample was placed in a platinum crucible. Mass changes were monitored from room temperature to 800 °C at a heating rate of 10 °C per minute. The analysis was performed in a nitrogen atmosphere with a 20 mL/min flow rate.

The surface morphology of the CMF was analysed using a field-emission scanning electron microscope (FESEM, Zeiss/Merlin Compact). The dried fibres were coated with a thin layer of gold through sputter-coating, and imaging was performed at an accelerating voltage of 3.0 kV.

## 3. Results

### *3.1. Chemical Composition of Durian Rinds*

The findings indicate that steam explosion (SE) pretreatment significantly alters the composition of durian rind biomass, increasing cellulose purity (Table 1). In SE-treated samples, cellulose content rose from 38.37% to 52.92%, a change attributed to the disruption of the lignocellulosic matrix during the SE process. This process involves exposing biomass to high-pressure steam followed by a rapid release of pressure, which causes structural breakdown and defibrillation of the plant cell wall. The mechanical disruption reduces the lignin and hemicellulose surrounding the cellulose fibres, enhancing cellulose accessibility and purity (Zeng et al., 2013; Xiang et al., 2020).

***Table 1.*** The chemical composition of non-SE treated, and SE-treated durian rind samples is presented, highlighting the weight percentages (wt%) of hemicellulose, cellulose, and lignin content.

| Sample | Hemicellulose (wt%) | Cellulose (wt%) | Lignin (wt%) |
|---|---|---|---|
| **Non-SE-treated Durian Rind** | 35.40 | 38.37 | 26.23 |
| **SE treated Durian Rind** | 29.71 | 52.92 | 17.37 |

After SE treatment, hemicellulose content decreased from 35.40% to 29.71%, while lignin content dropped from 26.23% to 17.37%. The high temperature and pressure of steam explosion promote the hydrolytic and thermal degradation of hemicellulose, an amorphous polysaccharide polymer, making it more susceptible to degradation. This degradation affects the glycosidic linkages in hemicellulose, facilitating its removal in subsequent washing stages. Additionally, the complex aromatic structure of lignin undergoes partial degradation under SE conditions, aiding its solubilization and extraction from the biomass matrix. Seidel et al. (2019) demonstrated that the steam explosion process affects the structural integrity of lignin, resulting in partial degradation. The study highlighted that the conditions during steam explosion facilitate the solubilization and extraction of lignin from biomass, supporting the assertion that steam explosion pretreatment enhances the extraction of hemicellulose and lignin. Mlauer-Vedoya et al. (2019) reported that high-pressure steam and rapid decompression during steam explosion facilitate the breakdown of the lignocellulosic structure, enhancing the solubilization of hemicellulose and lignin. This finding is supported by Kong et al. (2022), who discovered that steam explosion treatment effectively hydrolysed hemicellulose and partially degraded lignin, contributing to the overall efficiency of biomass conversion.

The increased cellulose content and reduced hemicellulose and lignin levels underscores the effectiveness of SE pretreatment in producing a high-purity cellulose product suitable for advanced applications such as biocomposites, biofuels, and nanocellulose. Removing hemicellulose and lignin exposes cellulose hydroxyl groups, increasing its reactivity and facilitating later functionalisation. This compositional change highlights the potential of SE as a sustainable and efficient strategy for valorising lignocellulosic biomass, enhancing its applicability for high-value cellulose-based materials.

### *3.2. CMF Yield from Durian Rind Extraction*

In the present study, biomass waste processing results show enhanced CMF recovery when comparing untreated and steam-exploded (SE) treated durian rind. The CMF recovery from the SE-treated durian rind is much higher, at 63.6 wt%, compared to only 34.01 wt% from the untreated rind, as presented in Figure 4 below. This demonstrates the SE treatment's efficacy in achieving better biomass recovery yields.

The increased recovery of CMF can be explained by the fact that SE treatment disrupts the lignocellulosic structure, making cellulose more accessible for conversion. The SE process aids in the breakdown of hemicellulose and partial delignification, likely playing an important role in improving the overall yield. Such structural changes are supported by previous reports on other biomass sources, where SE treatment increased cellulose accessibility and reactivity (Kang et al., 2013; Kukle et al., 2015). These comparisons underline the potential value of SE treatment in biomass valorisation strategies and further support the generalizability of this approach to all forms of lignocellulosic residues.

The novelty of this research lies in the application of SE treatment to durian rind, an agricultural waste that is typically not fully utilised. Earlier research on durian rind primarily focused on composting methods or mild chemical extraction, resulting in relatively low-value products. Through SE treatment, this study demonstrates a significant increase in the potential recovery of CMF, thereby providing a pathway for converting agricultural waste into helpful chemical feedstocks. This represents a marked improvement over previous reports where unprocessed biomass exhibited low conversion efficiency.

These developments have important implications for advancing sustainable biomass conversion technologies. The increase in CMF yield following SE treatment suggests that similar methodologies could be effectively adapted for other fruit biomass residues, contributing to a circular economy and further reducing the environmental impact of agricultural waste. This work opens possibilities for scaling up the SE process and exploring economic feasibility studies to integrate such pretreatment processes within existing biomass valorization frameworks.

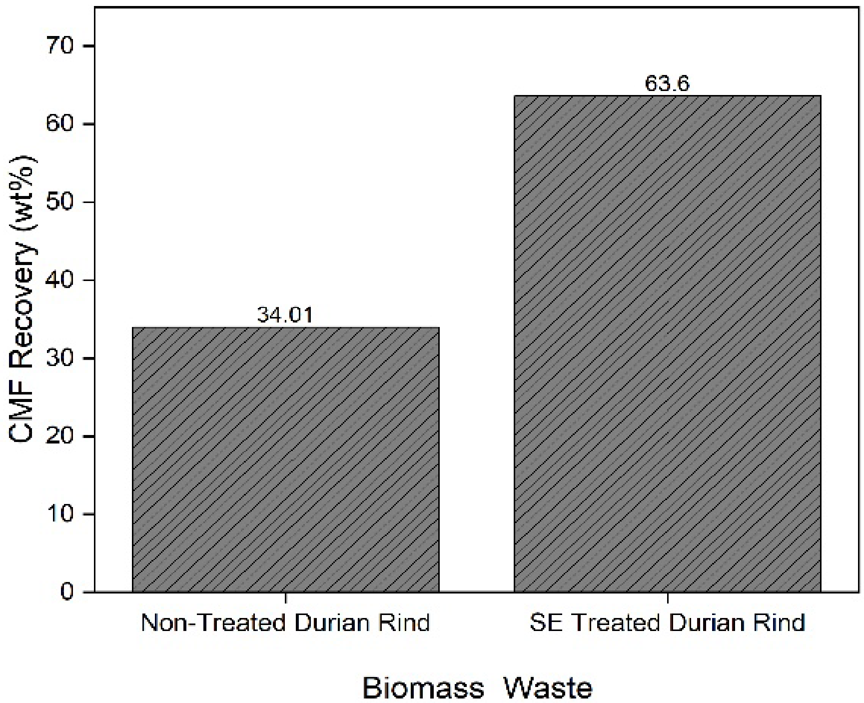


***Figure 4.*** Durian rind CMF recovery (wt.%) after SE and non-SE treatment. SE-treated durian rind recovered 63.6 percent cellulose compared to 34.01% in the non-SE sample.

## *3.3. Characterization of Extracted CMF*

### 3.3.1. Fourier Transform Infrared (FTIR) Analyses

Figure 5 displays the FTIR spectra that compare the chemical structures of CMF extracted from durian rind before and following SE treatment. Both spectra exhibit characteristic peaks that are indicative of cellulose. The spectra of all samples exhibit comparable absorption frequencies, suggesting the presence of consistent functional groups. Bands attributed to native cellulose I are observed at approximately 3400, 2900, 1430, 1370, and 890 $cm^{-1}$ (Wang et al., 2012; Veeramachineni et al., 2016). The SE-treated sample exhibits significant changes that validate the effectiveness of pretreatment in improving cellulose purity. The absorption band at 1028 $cm^{-1}$, associated with the C–O stretching of polysaccharides, indicates that both untreated and steam-exploded (SE-treated) samples possess the polysaccharide backbone of cellulose (Boukir, Fellak and Doumenq, 2019). The increase in the intensity of this band after SE treatment suggests enhanced exposure of the cellulose structure, indicating effective removal of surrounding lignin and hemicellulose that previously obscured the cellulose chains (Pérez-Aguilar, Ruzafa-Silvestre and Arán-Aís, 2022).

The peak observed at 1645 cm⁻¹ signifies the stretching vibration of C=O, which is linked to the vibrations of cellulose carbonyl groups, as well as those of the acetyl and uronic ester groups derived from pectin, hemicellulose, or the ester linkages of carboxylic groups found in ferulic and p-coumaric acids present in lignin and/ or hemicelluloses (Sain and Panthapulakkal, 2006) It also demonstrates a reduction in the intensity of the untreated sample following the SE pretreatment. This decrease in intensity indicates a reduction in lignin and suggests a partial elimination of hemicellulose (Then et al., 2014; Birnin-Yauri et al., 2016).

The band at 2896 cm⁻¹, attributed to C–H in both spectra, resembled the C-H stretching vibration of alkyl groups in aliphatic bonds of cellulose, hemicellulose and lignin (Sainorudin et al., 2021).The O–H stretching bands observed at 3289 cm⁻¹ for SE-treated CMFs and 3334 cm⁻¹ for untreated samples are attributed to hydroxyl groups that participate in hydrogen bonding within the cellulose structure (Wang et al., 2009).

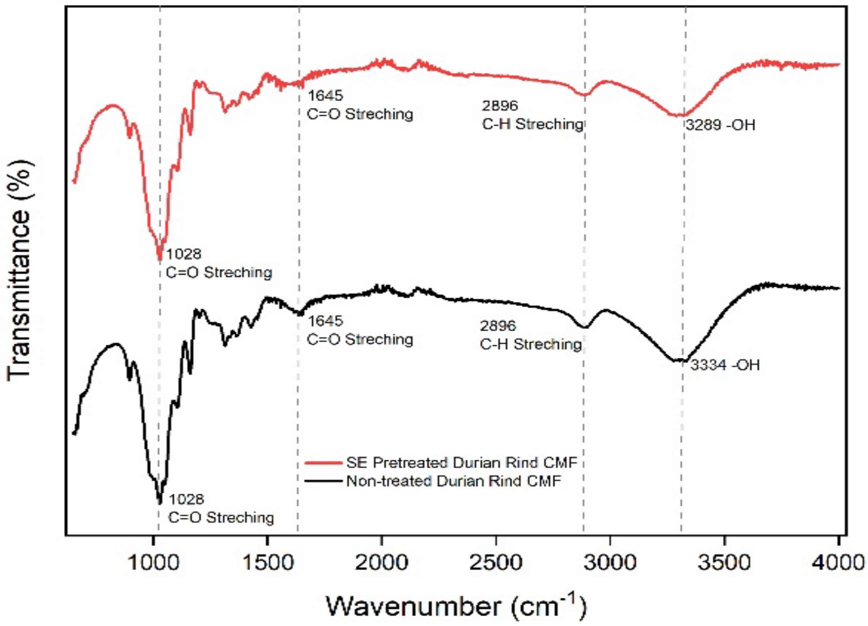


***Figure 5.*** Fourier transform infrared spectroscopy (FTIR) spectra of SE-pretreated and non-treated durian rind cellulose microfibres (CMFs), highlighting key absorption bands indicative of cellulose functional groups.

## 3.3.2. XRD Analysis of CMF Extracted from Durian Rind

X-ray diffraction (XRD) was used to determine the crystallinity and crystal structure of the cellulose samples. A crucial aspect of XRD characterisation is the preferred orientation of the crystallites, commonly referred to as texture. Various factors, such as synthesis procedures, crystallite properties, and pretreatment conditions, influence texture development. These factors also impact the relative intensities of the diffraction peaks, which, in turn, affect the crystallinity index of the samples (Flórez Pardo, Salcedo Mendoza and López Galán, 2019). Figure 6 presents the X-ray diffraction patterns of cellulose microfibres (CMFs) extracted from non-treated and SE-pretreated durian rind, elucidating their crystallinity characteristics. Three prominent peaks at 2θ values of approximately 15.80, °16.47° and 22.19° correspond to the characteristic crystalline regions of cellulose I, representing reflections from the (110) and (200) planes, respectively.

The X-ray diffraction (XRD) patterns reveal the sharp and broad intensity of the peak at approximately 22.2° for both CMF samples. This peak intensity correlates with crystallinity, suggesting high crystallinity (Zhang et al., 2013). The transmittance of SE-pretreated durian rind CMF typically shows a reduction and broadening of the intensity of crystalline peaks after treatment, indicating an increase in amorphous content. Mohamood, Halim and Zainuddin (2021) reported that cellulose post-treatment typically exhibits a reduction in the intensity of crystalline peaks in the XRD patterns, directly indicating an increase in the amorphous content. The primary diffraction peaks remain at comparable positions in treated and untreated samples, suggesting cellulose retains its type I crystalline structure despite the partial reduction in crystallinity. The diffraction peaks displayed a consistent pattern characteristic of the typical cellulose I structure. Cellulose I, the most prevalent crystalline form, consists of a series of crystallites interspersed with disordered amorphous regions. In both samples, the predominant intensity was attributed to the cellulose I crystalline structure.

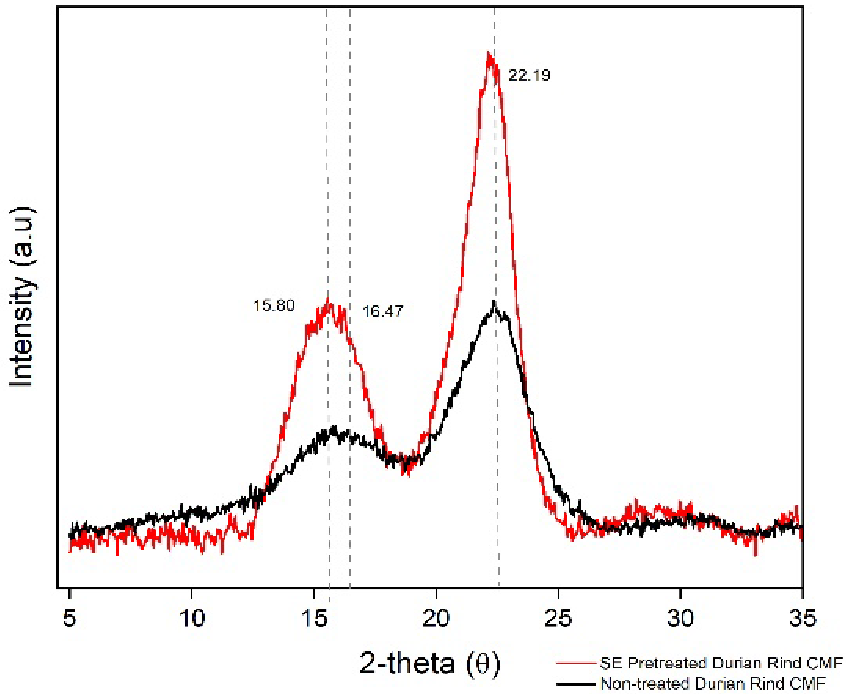


***Figure 6.*** X-ray diffraction (XRD) patterns of SE-pretreated and non-treated durian rind cellulose microfibres (CMFs), showing the characteristic crystalline peaks of cellulose.

The crystallinity index (CrI) values presented in Table 2 indicate a reduction from 62.1% in untreated durian rind CMF to 50.1% in SE-pretreated CMF. This decrease in crystallinity suggests a shift towards a more amorphous structure, which is beneficial for enhancing the cellulose's reactivity in applications such as enzymatic hydrolysis or chemical modification. The reduction in crystallinity may also improve the material's suitability for use in biocomposites and biofuels, where increased reactivity and accessibility are crucial for efficient processing. This finding adds to the growing body of knowledge on biomass valorization, highlighting SE as an effective and sustainable pretreatment for agricultural wastes like durian rind. This reduction signifies that SE pretreatment effectively modifies the crystalline regions of cellulose. The SE process encompasses the application of high-pressure steam followed by rapid depressurisation, which introduces mechanical and thermal stresses to the cellulose fibres, thereby disrupting the ordered crystalline regions and increasing the proportion of amorphous fractions. This alteration accounts for the observed decrease in CrI. This reduction is consistent with the findings of Lorenzo-Hernando, Martín-Juárez and Bolado-Rodríguez (2018), who reported that pretreatment at elevated temperatures (e.g., 150 °C) can diminish the crystalline fraction of cellulose because of the mechanical and thermal effects of steam explosion (SE).

***Table 2.*** Crystallinity index (CrI) of non-treated and SE-pretreated durian rind CMF, indicating changes in crystallinity following steam explosion pretreatment.

| **Sample** | **Crystallinity Index (CrI)** |
|---|---|
| Non-treated Durian Rind CMF | 62.1 |
| SE Pretreated Durian Rind CMF | 50.1 |

The XRD analysis and crystallinity data highlight the substantial impact of SE pretreatment on the microstructural properties of cellulose. Enhancing the amorphous fraction through SE pretreatment presents a viable approach for biomass valorisation via structural transformation, improving processability and expanding the potential applications of cellulose derived from durian rind across various industrial sectors.

### 3.3.3. TGA Analysis

The TGA thermogram shown in Figure 7 offers insights into the thermal stability and degradation behaviour of the extracted CMF. As illustrated, all samples exhibit an initial weight loss of 0.9–1.4% at temperatures above 100 °C, consistent with typical behaviour observed in lignocellulosic materials. This initial decrease in weight is mainly attributed to the evaporation of adsorbed water or bound moisture present in the samples (Narkesabad, Rafiee and Jalilnejad, 2023).

Within the temperature range of 240 to 600 °C, the samples exhibit remarkably similar decomposition patterns, which can be attributed to the degradation processes associated with cellulose, including decarboxylation, depolymerisation, and the breakdown of glycosyl units. Both durian rind cellulose microfibrils (CMF) demonstrate significant weight loss from 235 to 800 °C, indicating substantial thermal decomposition, with a notable weight loss of 75.0% occurring between 235 and 540 °C. In contrast, the pretreated durian rind CMF reveals a distinct degradation pattern, likely due to alterations in the lignocellulosic structure resulting from the pretreatment process. This pretreatment appears to enhance thermal stability, as evidenced by the modifications in the decomposition curve.

The DTG curve (Figure 8) demonstrates a significant decomposition peak within the temperature range of 300–400 °C for both samples, which corresponds to the thermal degradation of cellulose. The non-treated durian rind cellulose microfibrils (CMF) exhibit a principal decomposition peak at approximately 345 °C, indicating cellulose degradation. In contrast, the SE-pretreated durian rind CMF displays a peak at approximately 335 °C. Notably, the maximum decomposition rate for the SE-pretreated CMF occurs at a slightly elevated temperature compared to the non-treated CMF, suggesting enhanced thermal stability. This shift in the peak indicates that the SE pretreatment induces structural changes that enhance resistance to thermal degradation, likely resulting from the partial removal of hemicelluloses and other impurities (Sebestyén et al., 2013).

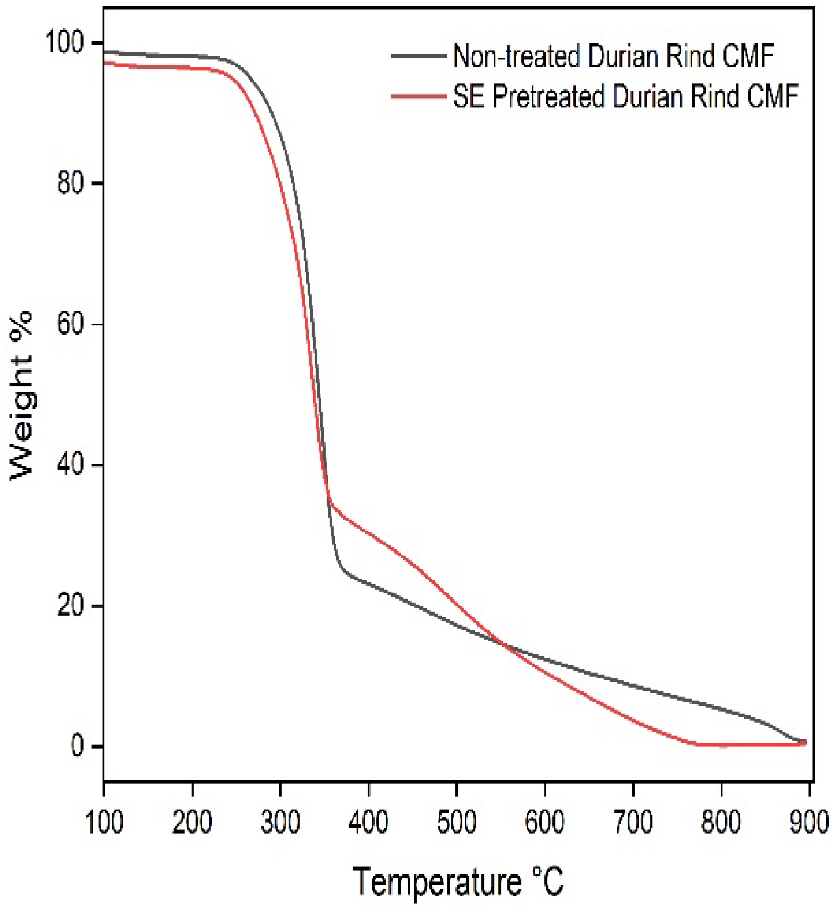


***Figure 7.*** Thermogravimetric analysis (TGA) curves of non-treated and SE pretreated durian rind cellulose microfibrils (CMF).

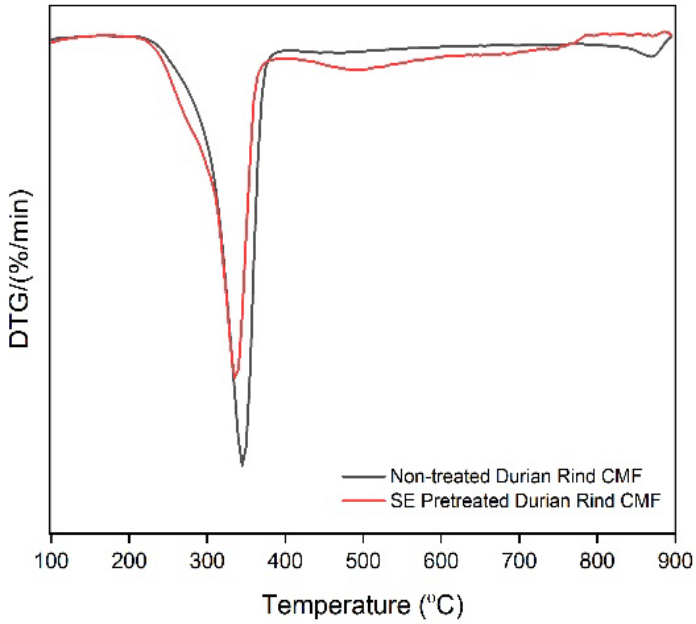


***Figure 8.*** Derivative thermogravimetric (DTG) curves of non-treated and steam explosion (SE) pretreated durian rind cellulose microfibrils (CMF).

### 3.3.4. FESEM Analysis

As shown in Figure 9a and b, Figure 9a illustrates the cellulose microfibrils (CMF) from non-treated durian rind, which display a more entangled and irregular structure. The non-treated durian rind CMF fibres appear densely packed and have a coarse surface, likely due to non-cellulosic components such as lignin and hemicellulose. This tight binding suggests that the cellulose is embedded within a complex lignocellulosic matrix, which limits the accessibility of the CMF.

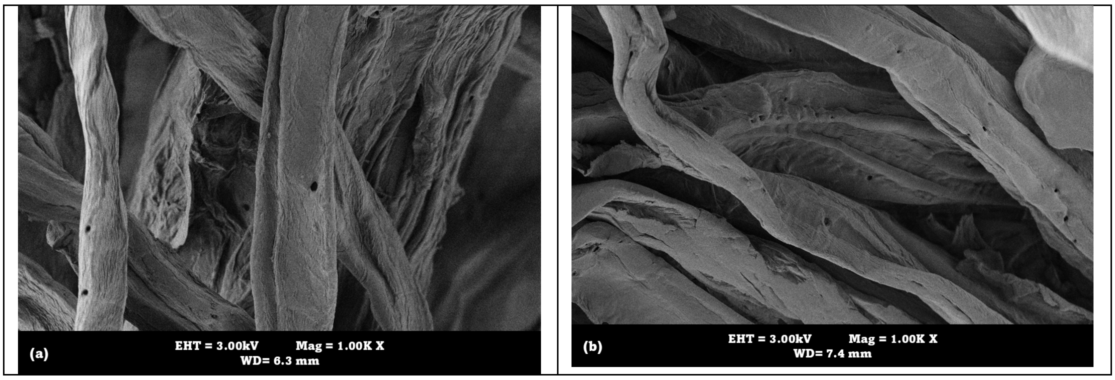


***Figure 9.*** FESEM image of extracted of CMF from (**a**) non-treated durian rind CMF, (**b**) SE pretreated durian rind CMF.

Following steam explosion (SE) pretreatment, the CMF fibres from durian rind (Figure 9b) exhibit significant improvements in fibre separation and surface morphology. The fibres have a more defined structure with reduced aggregation and a smoother surface texture. The decreased roughness and improved separation between fibres indicate that the SE pretreatment effectively disrupted the lignin-hemicellulose network, allowing for the release of cellulose microfibrils. The enhanced structure of the SE-pretreated durian rind fibres demonstrates that the pretreatment successfully removed a substantial amount of non-cellulosic materials, resulting in cleaner and more accessible cellulose fibres.

The fibres extracted from durian rind showed a significant reduction in width following steam explosion (SE) pretreatment. The raw durian rind fibres (Figure 9a) had an average width of approximately 40 micrometres, reflecting the dense and entangled structure of the raw material. After SE pretreatment (Figure 9b), the average fibre width decreased to about 30 micrometres, indicating a more refined fibre morphology. This notable reduction in fibre width is associated with disrupting and removing the lignin-hemicellulose matrix during the steam explosion process, facilitating improved separation of individual cellulose fibrils. The SE pretreatment reduced fibre width by smoothing the fibre morphology, contributing to more cellulose purification. Similar observations have been reported for cellulose fibres isolated from Phoenix canariensis palm leaves, where SE pretreatment resulted in a reduced fibre diameter and a cleaner surface (Pérez-Limiñana et al., 2022)

Steam explosion (SE) pretreatment effectively removes non-cellulosic components, reducing fibre width and a smoother surface morphology. By eliminating lignin and hemicellulose, SE treatment exposes and refines cellulose fibres, leading to narrower and more uniform fibrils. This optimised fibre morphology enhances the performance of cellulose microfibrils in applications such as composite production and bio-based materials, where finer, cleaner cellulose fibres improve mechanical properties and processing efficiency. The combination of reduced fibre width, smoother surface texture, and enhanced fibrillation in SE-pretreated samples demonstrates the effectiveness of SE pretreatment in improving the quality and functionality of cellulose fibres derived from durian rind. These enhanced fibre characteristics are expected to expand the potential applications of cellulose from these agricultural residues in sectors such as packaging, paper products, and bioplastics, where high-quality cellulose is critical for optimal material performance.

Delignification and bleaching are essential for purifying cellulose by removing lignin and other non-cellulosic compounds. The findings of Qi, Liang and Luo (2021) support that lignin removal has a strong linear correlation with enzymatic digestibility, indicating that delignification significantly enhances cellulose conversion efficiency. These processes expose the cellulose-hemicellulose matrix, often resulting in a rougher surface texture, as described by Kumar et al. (2009). However, the SE-pretreated sample exhibits a smoother surface due to several factors. The SE pretreatment hydrolyses residual hemicellulose, causing the fibres to swell or undergo gelatinisation, which results in a smoother cellulose surface (Zhao, Zhang and Liu, 2012; Liu et al., 2017; Dyartanti et al., 2019).

Additionally, SE pretreatment induces minor surface modifications, resulting in a more uniform and streamlined fibre structure. The combined effects of SE, delignification, and bleaching reveal the intrinsic structure of cellulose fibres, which are inherently smoother than untreated fibres. Smoother cellulose fibres enhance adhesion between the fibre surface and the matrix in specific applications, thereby increasing their potential as reinforcing agents, as highlighted by Yacob et al. (2018). This finding aligns with the observations made by Chandra et al. (2015), who reported that the combined effects of steam pretreatment and subsequent processing steps produced a more uniform fibre structure, crucial for improving adhesion between cellulose fibres and matrix materials.

The durian rind CMF exhibits a denser and more compact lignocellulosic structure, as illustrated in Figure 9a. The density of the raw fibres is probably attributable to an increased lignin content, which serves as a binding agent, enhancing fibre rigidity and reducing susceptibility to separation. The rigidity of non-treated durian rind CMF contributes to its coarse and irregular surface texture. The residual lignin and hemicellulose in untreated durian rind CMF result in a rougher surface.

Following SE pretreatment, the durian rind CMF (Figure 9b) exhibits notable enhancements in surface morphology, presenting a smoother and more uniform appearance. The SE pretreatment likely disrupted the lignin-hemicellulose matrix, like the effects observed in sago bark, resulting in the release of cellulose fibres and decreased surface roughness. The enhanced smoothness of SE-pretreated durian rind CMF results from the partial hydrolysis of hemicellulose and the removal of lignin, which exposes the underlying cellulose and facilitates a more streamlined fibre structure. The synergistic impact of delignification, bleaching, and SE pretreatment is expected to yield a more uniform fibre structure in durian rind. The smoother surface and decreased fibre width in SE-pretreated durian rind CMF may improve their applicability in diverse industrial contexts. Smoother fibres may enhance compatibility with polymer matrices, increasing their suitability for composite materials. Furthermore, the use of cleaner and more refined cellulose fibres is essential for paper production and other bio-based products, as surface smoothness and fibre purity significantly affect the quality of the final product (Bufalino et al., 2015; J.-X. Wang et al., 2020; Klunklin et al., 2023).

The morphological transformation of durian rind CMF following SE treatment underscores the importance of pretreatment processes in enhancing CMF quality. SE pretreatment enhances the adaptability of CMF for high-performance applications by eliminating non-cellulosic components and refining the cellulose structure. This process broadens the applicability of agricultural waste materials, such as durian rind, in sustainable industries.

The SE pretreatment technique demonstrated in this study not only improves cellulose yield and purity but also shows great potential for large-scale industrial applications. Durian rind, as an abundant waste biomass in Southeast Asia, presents a valuable source for cellulose extraction. The successful application of SE in this context opens up possibilities for industrial-scale applications, such as in biocomposites, biofuels, and nanocellulose-based products. Furthermore, SE is an environmentally friendly and cost-effective method, as it requires minimal chemical usage and energy input, making it a sustainable alternative to conventional pretreatment methods.

## 4. Discussion

This study demonstrates that steam explosion (SE) pretreatment significantly enhances the extraction of cellulose microfibers (CMFs) from durian rind. The substantial increase in cellulose yield and purity is attributed to the disruption of the lignocellulosic matrix, which reduces the hemicellulose and lignin content, thereby making the cellulose more accessible for downstream applications such as bio composites and biofuels. The marked increase in both cellulose yield and purity following SE treatment highlights its potential as a sustainable and efficient method for extracting cellulose from lignocellulosic biomass.

### *4.1. Summary of Key Findings*

This study demonstrated that steam explosion (SE) pretreatment significantly enhanced cellulose yield, with SE-treated durian rind achieving a cellulose recovery of 63.6%, compared to 34.01% from untreated samples. The SE technique resulted in a substantial reduction in the hemicellulose and lignin content of the durian rind, as verified by the Van Soest method, yielding a more refined cellulose. Furthermore, Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) measurements indicated that the SE treatment increased the accessibility of cellulose functional groups and decreased crystallinity, respectively. Thermogravimetric analysis (TGA) data demonstrated improved thermal stability of the SE-treated cellulose microfibers (CMFs), while field emission scanning electron microscopy (FESEM) imaging revealed smoother, narrower fibers with a more uniform shape. Collectively, these results indicate that SE pretreatment significantly enhances the quality and production of cellulose microfibers derived from durian rind.

### *4.2. Comparison with Existing Literature*

The findings of this study are consistent with previous research demonstrating the efficacy of steam explosions in the removal of cellulose from various lignocellulosic biomass sources. Following steam explosion pretreatment of different biomass materials, such as rice straw waste and softwoods. Serrano-Martínez et al. (2024) reported comparable increases in cellulose yield and purity. Notably, the 63.6% cellulose recovery achieved in this study suggests that steam explosion treatment may be particularly advantageous for durian rind, a biomass characterized by a high cellulose concentration, especially in comparison to the 50% recovery documented by (Chang et al., 2012; Zhang et al., 2022).

Furthermore, the findings of Szadkowski et al. (2023) conducted a comparative analysis of cellulose isolated from poplar wood which indicated that steam explosion pretreatment generally results in a reduction of crystallinity, align with the observed decrease in crystallinity noted in this study. Similarly, Ouyang, Chen, Zhang, Yuan, Wei Wang, et al. (2018) emphasized that steam explosion pretreatment effectively disrupts cellulose crystallinity, a critical factor in improving the digestibility of lignocellulosic biomass. This alteration of crystalline regions is beneficial as it enhances the accessibility of cellulose, making it more suitable for applications such as chemical modification and enzymatic hydrolysis (Zhang et al., 2022).

Additionally, previous research by Liu et al. (2017) demonstrated that steam explosion pretreatment enhances the thermal properties of cellulose by reducing the content of non-cellulosic components, such as lignin and hemicellulose, which can diminish the thermal stability of cellulose. This observation is also consistent with the enhanced thermal stability of the steam explosion-treated cellulose microfibrils observed in this study.

### *4.3. Research Limitations*

While the study demonstrated the efficacy of steam explosion (SE) pretreatment, it possesses significant limitations that warrant attention in future research. Firstly, the study was conducted under a fixed set of SE pretreatment conditions (145 °C for 5 minutes). Subsequent research should investigate the effects of varying steam explosion parameters, including temperature, pressure, and duration, on cellulose yield and purity. Additionally, this study exclusively utilized durian rind as the biomass source; thus, it would be beneficial to evaluate the applicability of SE pretreatment to other lignocellulosic materials, such as agricultural and forestry residues, to assess the generalizability of the method. Finally, although the focus of this work was on enhancing cellulose yield and purity. However, further investigation into the scalability, economic feasibility, and environmental impact of SE pretreatment for large-scale applications is necessary. Future research should focus on evaluating the cost-effectiveness of SE treatment in industrial settings and its integration with other biomass valorization strategies.

## *4.4. Future Research Directions*

Future research should focus on optimizing SE pretreatment parameters, including varying pressure, temperature, and time, to maximize cellulose yield and quality. Additionally, expanding this study to include a broader range of lignocellulosic biomass materials—such as palm oil empty fruit bunches, coconut husk, or rice straw—would provide valuable insights into the versatility and scalability of SE pretreatment for different agricultural wastes. Moreover, economic feasibility studies and life cycle assessments should be incorporated to evaluate the cost-effectiveness and environmental benefits of SE at an industrial scale. Further exploration of SE's integration with other pretreatment methods, such as enzymatic hydrolysis or alkaline treatments, could enhance its efficiency and broaden its application in biofuels, biocomposites, and other high-value products. Future research should also incorporate economic feasibility studies and life cycle assessments to determine the long-term viability and cost-effectiveness of SE pretreatment in large-scale industrial applications. Moreover, investigating the use of SE-treated cellulose in advanced applications, such as biocomposites, biofuels, and nanocellulose-based products, would help assess the commercial viability of this method.

## *4.5. Conclusion*

To sum up, this investigation shows how well steam explosion (SE) works as a pretreatment technique to improve the extraction of cellulose microfibres (CMFs) from durian rind. The findings demonstrate that SE causes structural and chemical alterations, including decreased crystallinity and improved thermal stability, while also markedly increasing cellulose yield and purity. These results demonstrate SE as a sustainable and scalable strategy for obtaining high-quality cellulose from underutilised agricultural waste, while simultaneously advancing biomass valorisation techniques. This strategy has the potential to revolutionise agricultural waste management by offering a cost-effective and sustainable substitute to produce cellulose-based goods. Comprehensive analyses using XRD, FTIR, TGA, and FESEM confirm the high quality and chemical integrity of the extracted cellulose. These findings suggest its potential for various high-value applications, such as biocomposites, biofuels, and nanocellulose-derived products.

Moreover, the study emphasizes SE technology as a scalable and sustainable approach for valorizing lignocellulosic biomass, specifically durian rind, as a renewable source of high-quality cellulose fibres. This research contributes to the growing field of biomass utilization, particularly in the sustainable production of high-quality cellulose. It underscores the potential of SE technology in valorizing agricultural waste, with applications in biocomposites, biofuels, and nanocellulose-based products. Future studies should explore the integration of SE with additional pretreatment methods to further optimize cellulose production.

Future research should focus on integrating SE with additional pretreatment methods to further improve cellulose production and quality. It is also crucial to evaluate the economic and environmental viability of scaling up SE technology for industrial applications. Exploring the uses of SE-pretreated cellulose will provide valuable insights into its commercial potential, promoting the development of innovative and sustainable materials across various industries.

**Author Contributions**

Akmal Aizuddin Zulkifli: Conceptualisation, methodology, formal analysis, writing—original draft, Masita Mohammad supervision, resources, funding, Norhasnan Sahari.; writing—review and editing. Norzita Yacob; writing—review and editing. Sabariah Kamarudin; writing—review and editing, Sakhr M. Sultan review, supervision, resources. All authors have read and agreed to the published version of the manuscript.

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**Conflicts of Interest**

The authors declare no conflict of interest.

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