

Research Article

Preparation and Characterization of Carboxymethyl-cellulose Derived from Pineapple Crown Leaves Waste

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Abstract

This research investigates the extraction and carboxymethylation of cellulose from waste pineapple crown leaves to produce carboxymethyl cellulose (CMC). Cellulose was effectively obtained by processing powdered pineapple crown leaves with sodium hydroxide (NaOH) and nitric acid (HNO₃) at 90 °C, resulting in maximum extraction yields of 51.64 ± 1.33 wt.%. The process of carboxymethylation, optimized at 60 °C for 1.5 hours with chloroacetic acid, produced CMC with a degree of substitution (DS) of 2.21. Characterization methods such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX) validated alterations in structure and composition. FTIR spectra indicated the effective elimination of hemicellulose and lignin, while the presence of significant absorption bands at 1586 cm⁻¹ and 1416 cm⁻¹ confirmed the etherification process. XRD analysis indicated a notable decrease in cellulose crystallinity due to carboxymethylation, which is linked to the addition of carboxymethyl groups. SEM imaging showed a shift from coarse raw fibers to more refined extracted cellulose, while CMC displayed a prolonged, uneven, and somewhat compressed structure. Analysis of particle size distribution revealed an average size focused around 537 µm. EDX analysis confirmed the elevated purity of the obtained cellulose. These results emphasize the capability of using agricultural waste for the eco-friendly creation of valuable biopolymers, showing the viability of transforming pineapple crown leaves fibers into functional CMC for multiple industrial uses.

Keywords

Lignocellulosic Biomass, Bio-based, Extraction, Cellulose, Carboxymethyl Cellulose, Degree of Substitution, Pineapple Crown Leaves

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1. Introduction

Pineapple crown leaves (PCL), an important byproduct produced during pineapple processing, significantly add to agricultural waste. The crown of a pineapple generally accounts for approximately 10–25% of the overall weight of the fruit [1]. Global pineapple production generates around 3 billion tons of this waste each year, creating a significant environmental issue, especially in agriculture [2]. The makeup of PCL mainly includes intricate carbohydrate polymers like cellulose, hemicellulose, and lignin, which are essential for its structural stability [1]. Along with these main ingredients, PCL includes lesser quantities of extractives, inorganic substances, and ash [1, 3, 4]. Earlier research has indicated the approximate proportions of these components, where cellulose comprises 79–83%, hemicellulose constitutes 19%, and other materials such as lignin, extractives, and ash make up the remaining 5–15% and 4–5%, respectively [3, 5-8].

Considering the high cellulose content in PCL, which represents a large part of its dry weight, this biomass can be regarded as abundant, renewable resource. Cellulose is especially significant because of its potential as a raw material for generating numerous bio-based and environmentally friendly products. These derivatives may find numerous applications, ranging from biocompatible materials to sophisticated chemical products, thereby presenting a hopeful path for minimizing the environmental impact linked to pineapple waste. The enhancement of PCL, particularly via the extraction and alteration of cellulose, symbolizes an essential move towards improving sustainable farming methods and addressing the waste issue in pineapple processing sectors.

Cellulose is a linear polysaccharide made up of repeating units of D-glucopyranose that are covalently connected through β -1, 4-glycosidic bonds [9, 10]. This biopolymer is a key structural element of plant cell walls and exists in a well-organized structure that is crucial for preserving the stiffness and integrity of plant tissues. In its natural form, cellulose creates microfibrils, which are long structures that consist of both crystalline and amorphous parts. The crystalline domains possess a high level of organization, which aids in the rigidity and insolubility of cellulose, whereas the amorphous regions display a less ordered structure, increasing its flexibility and availability for enzymatic breakdown.

The microfibrils are connected through hydrogen bonds that develop between the hydroxyl groups of neighboring cellulose chains and the oxygen atoms in the polymer structure. These inter- and intramolecular hydrogen bonds are crucial for the overall stability and strength of cellulose fibers, offering resistance to chemical degradation and mechanical stress.

All cellulose found in nature is categorized as cellulose type I, distinguished by the existence of intra- and intermolecular hydrogen bonding interactions both within and among separate cellulose chains [3]. The distinctive structural con-

figuration of cellulose type I enhances its high level of crystallinity and its inability to dissolve in water, rendering it a perfect substance for strengthening plant cell walls. Moreover, this remarkably organized structure of cellulose holds considerable importance for its possible uses in different sectors, such as biofuels, biocompatible substances, and nanomaterials. Manipulating and altering the crystalline and amorphous areas of cellulose is crucial for realizing its complete potential in sustainable and eco-friendly applications.

Carboxymethyl cellulose (CMC) is a commonly used derivative of cellulose, created by the carboxymethylation of the hydroxyl groups found in cellulose molecules. Owing to its adaptable characteristics, CMC is utilized in multiple applications, such as a thickening agent, binder, film-former, suspending aid, and in the creation of biodegradable materials [11-15]. The production of CMC requires swelling cellulose in a sodium hydroxide (NaOH) solution to enhance its reactivity, then reacting with monochloroacetic acid in an alcohol-based medium [16]. In this process, carboxymethyl groups are added to the cellulose framework by replacing the hydroxyl groups located at the C-2, C-3, and C-6 sites of the anhydroglucose units. Importantly, modification at the C-2 position is seen to be somewhat more prevalent than at the other locations [17].

The degree of substitution (DS) is an essential factor in defining the characteristics of CMC, especially its solubility in water. An increased DS usually improves the solubility of CMC, rendering it more efficient for different uses. In theory, the highest attainable DS for CMC is 3, requiring a DS over 0.4 for water solubility. Studies have generally indicated that DS values are between 0.5 and 2.0, while CMC available on the market shows DS values in the range of 0.4 to 1.4 [16-18]. In recent years, significant resources have been focused on enhancing the DS of CMC to boost the quality and effectiveness of commercial products. Research indicates that the crystalline nature and dimensions of the cellulose employed as the raw material significantly influence the attainment of a greater DS in the end product [16].

The pursuit of alternative raw materials, especially agricultural by-products, for CMC production has gained significant interest. Employing these by-products not only creates a pathway for waste valorization but also presents an economical and sustainable approach to producing CMC. For example, multiple research efforts have investigated utilizing non-wood cellulose sources like agricultural residues as substitutes for virgin softwood pulp in producing CMC [12, 16-22]. In a study conducted by Haleem *et al.* [21], it was shown that cellulose fibers obtained from cotton waste via acid hydrolysis using 10 M sulfuric acid at temperatures between 70-80 °C for 1 hour resulted in fibers measuring 15-20 μm in size. This underscores the capability of agricultural waste to act as a feasible cellulose source for CMC production.

The extraction of cellulose is a complicated procedure,

usually requiring several stages to attain a high level of substitution. Consequently, finding new, easily accessible, and affordable cellulose sources is crucial for enhancing the efficiency and sustainability of CMC manufacturing. Moreover, improvements in refining cellulose extraction techniques can boost the ability of agricultural by-products to substitute conventional cellulose sources, aiding in more sustainable production methods within the cellulose derivatives sector.

As stated by Upadhyay et al. [23], pineapple (*Ananas comosus*) ranks among the most extensively grown and economically important fruits globally. The fruit is transformed into numerous products such as jams, concentrates, juices, and canned items [23]. A significant byproduct of pineapple processing is the crown, representing about 10-25% of the fruit's overall weight and resulting in an estimated 3 billion tons of waste produced each year [24]. Despite progress in agricultural technology, a significant amount of pineapple plant components, such as the crown, are still thrown away because of insufficient knowledge about their possible inexpensive uses. Inappropriate disposal of these fruit wastes poses issues, as they have a high biological and chemical oxygen demand, leading to major environmental problems. Additionally, pineapple byproducts are largely not utilized, possessing minimal market worth [23].

Despite being low-cost, renewable, biodegradable, and widely accessible, pineapple fibers sourced from plant residues, especially crown leaves, are seldom utilized in industrial applications [25]. Nonetheless, the leaves of pineapple crowns offer a hopeful and eco-friendly source of cellulose. It is noted that these leaves have around 40-50% cellulose based on dry weight [26, 27]. The process of extracting cellulose from pineapple leaves is relatively simple [28-32], and the cellulose produced shows lower crystallinity compared to cellulose obtained from alternative sources like cotton waste [21], paper sludge [19], and rice straw [33, 34]. This comparatively low crystallinity benefits the creation of CMC with a high DS, as it makes the modification and substitution of the cellulose molecules simpler.

This research seeks to investigate and confirm the utilization of cellulose derived from pineapple crown leaves, a commonly accessible agricultural byproduct, as an eco-friendly raw material for the industrial manufacture of carboxymethyl cellulose (CMC) featuring a high degree of substitution (DS). The primary goal is to evaluate the appropriateness of the produced CMC for advanced uses like hydrogel creation and the adsorption of heavy metal ions from water solutions. Through the use of this renewable biomass, the study aims to assist in creating affordable and eco-friendly materials for wastewater treatment and associated industrial processes, hence fostering a circular economy in the agricultural and biopolymer industries [28].

2. Methods

2.1. Collection and Purification of Samples

The waste from pineapple crown leaves was collected from

Ramin Kura markets in the Sokoto metropolitan area, Sokoto State, Nigeria. The PCL was cleaned extensively with flowing water to eliminate soluble sugars, dirt, and various impurities. Following washing, the leaf samples were dried in a laboratory oven at 100 °C for 24 hours to guarantee the thorough elimination of moisture. After the drying process, the leaves were processed into a fine powder with an estimated particle size of 2 mm using a grinding machine. The powdered sample was subsequently placed in airtight polythene bags to avoid moisture uptake and contamination, maintaining the sample's integrity before further processing and utilization in cellulose extraction and subsequent analyses. This method was diligently adhered to in order to preserve the quality and uniformity of the sample for subsequent uses [32].

2.2. Extraction of Cellulose from PCL Waste

Cellulose was obtained from pineapple crown leaves via a chemical method derived from the process described by Tuyet Phan *et al.* [32], with slight alterations. A 1000-mL beaker contained 10 g of dried pineapple leaf powder, which was treated with 250 ml of 0.75 M NaOH solution, and the combination was heated to 90 °C for 2 hours while stirring. Following the treatment, the resulting dark slurry was thoroughly rinsed with 250 mL of distilled water to eliminate any soluble materials. The leftover solid residue was subsequently treated with 150 mL of 5 M HNO₃ and heated at 90 °C for 1.5 hours to enhance the purification of the cellulose. The blend was subsequently filtered through a No. 3 porous funnel and rinsed several times with distilled water until the filtrate's pH approached around 7 [32].

The resulting residue was then dried in a lab oven until its weight remained constant, signifying the total elimination of moisture. After drying, the cellulose was pulverized into a fine powder and placed into a sealed vial, which was kept in a desiccator under normal conditions to avoid moisture uptake. The cellulose yield was calculated using the equation below:

$$H (\%) = \frac{m}{m_o} \times 100 \quad (1)$$

Where m is the weight of the extracted cellulose and m_o is the initial weight of the dried pineapple leaf powder. This procedure effectively isolated cellulose from the pineapple crown leaves, ensuring its suitability for subsequent analyses and applications.

2.3. Preparation of Carboxymethyl Cellulose (CMC)

The synthesis of CMC was conducted according to the method described by Phan and Ngo [32], with minor adjustments. A 1000 mL beaker received 150 mL of isopropanol, and then 5 grams of cellulose obtained from pineapple leaf powder was added to the mixture, which was stirred for 30 minutes. Subsequently, 15 mL of sodium hydroxide (NaOH)

solution with concentrations of 8%, 12%, 16%, and 20% (w/v) was introduced into the beaker, and the blend was stirred for an extra 1.5 hours at 60 °C.

To start the carboxymethylation reaction, different amounts of monochloroacetic acid (MCA) were introduced, specifically 1.0 g, 2.0 g, 3.0 g, and 4.0 g, based on the experimental parameters. The mixture was stirred steadily for 90 minutes at 60 °C. Once the reaction finished, acetic acid was added to neutralize the mixture, bringing the solid's pH to 7 [32].

To eliminate any leftover byproducts, the product was immersed in 20 mL of ethanol for 10 minutes, after which it was washed. This cleaning procedure was conducted three times to guarantee complete purification. The filtered CMC was subsequently dried in an oven at 60 °C for 2 hours to eliminate any leftover solvent. The finished product was kept in standard conditions to avoid moisture uptake and maintain its stability. The yield of the CMC was determined using equation (1):

Where m represents the weight of the obtained CMC and m_0 is the initial weight of cellulose used in the synthesis. This procedure successfully synthesized CMC from pineapple crown leaves cellulose, providing a sustainable and efficient method for utilizing agricultural waste in the production of useful biopolymers.

2.4. Structural Description

2.4.1. Fourier Transform Infrared Spectroscopy (FTIR)

The spectra of cellulose and CMC were obtained using a CARRY 630 FTIR Agilent. All spectra (32 scans at a resolution of 8.0 cm⁻¹) were collected at 25 °C, covering a range of 4000–650 cm⁻¹.

2.4.2. X-Ray Diffraction (XRD)

An XRD-6100 model (SHIMADZU) X-ray diffractometer was utilized to capture the X-ray diffraction (XRD) patterns of the cellulose and CMC. The angle of diffraction varied from 5 ° to 80 ° (0.05 °/min). It was captured using a Cu K target at 30 kV and 15 mA.

2.4.3. Scanning Electron Microscopy (SEM)

The samples' surface morphology was analyzed with SEM. The morphologies of the synthesized hydrogel were obtained by examining the surface of the samples with a focused electron beam using the Scanning Electron Microscope Phenom proX model.

2.4.4. Assessment of Degree of Substitution (DS)

The DS of CMC was assessed based on the Vietnamese standards TCVN 11921-8:2017 (COEI-1-CMC: 2009).

(1) *Preparation of Samples.* A sample of five grams (5g) was dissolved in 350 mL of ethanol within a 500 mL conical

flask. The mixture was stirred for 30 minutes to guarantee complete blending. Afterward, the solution was passed through a porous funnel under mild vacuum to remove any solid contaminants. The filtrate was subsequently moved to a crucible, and the solvent was eliminated by heating the crucible at 100 °C for 60 minutes to evaporate the volatile solvent [32].

Once the solvent was evaporated, the crucible with the residue was positioned in an oven and dried at 110 °C until its weight plateaued, signaling the full elimination of moisture. After each drying phase, the crucible was permitted to cool in a desiccator to avoid moisture absorption from the environment. This method guaranteed that the sample was completely dried and prepared for additional analysis.

(2) *Method.* Two grams of the dry material acquired from the previously mentioned ethanol-extraction method were precisely weighed and deposited into a tared porcelain crucible. The crucible was initially charred gently with a small flame, and later exposed to a larger flame for 10 minutes to guarantee thorough charring of the sample. Following the charring process, the leftover material was combined with 3–5 mL of concentrated sulfuric acid (H₂ SO₄) and warmed gently until smoke ceased to emerge.

Subsequently, 1 gram (1g) of ammonium carbonate was introduced into the crucible, and the blend was mixed thoroughly to guarantee uniform distribution of the powder. Heating was maintained on a low flame until smoke production stopped. Following this, the crucible was permitted to cool in a desiccator to avoid moisture uptake from the atmosphere. After cooling, the crucible was weighed to find the sample's final mass. The sodium level in the sample was subsequently determined using the equation below:

$$A (\%) = \frac{a \times 32.28}{b} \quad (2)$$

Where a represents the weight of the residual sodium sulfate, and b is the weight of the alcohol-extracted dry sample.

Finally, the DS of the CMC was calculated using the formula:

$$DS = \frac{162 \times A}{2300 - 80 \times A} \quad (3)$$

These procedures enabled the assessment of the sodium level and the DS of the CMC, offering useful insights into the structural properties of the produced material [32].

3. Results and Discussion

3.1. Synthesis of CMC from Waste of Pineapple Crown Leaves

3.1.1. Yields of Cellulose

Figure 1 shows the physical properties of PCL both prior to

and following treatment. Alkali treatment of pineapple crown leaves results in a distinct colour shift, changing from brown to pale yellow. This alteration in color indicates the elimination of several elements, such as lignin, hemicelluloses, waxes, proteins, soluble mineral salts, pectin, and ash, as demonstrated by earlier research [35-37]. After the alkali treatment, the bleaching process modifies the material even more, leading to a mostly white appearance. This whitening effect sig-

nifies a notable decrease in lignin remnants and non-cellulosic materials. Sasikala and Umapathy [38], state that the bleaching procedure significantly removes the residual lignin, improving the material's purity and aiding its later use in diverse industrial and biotechnological applications. The use of alkali treatment and bleaching successfully converts the pineapple crown leaves into a cleaner, more uniform state, enhancing their suitability for subsequent processing and use [37].

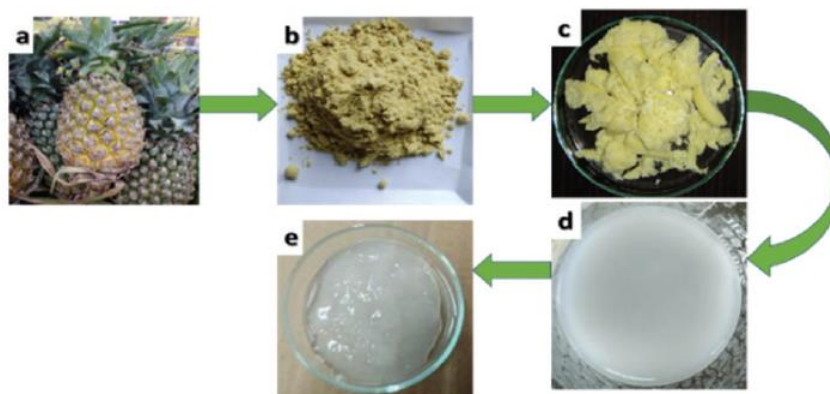


Figure 1. Images of (a) pineapple, (b) powder from pineapple crown leaves (PCL), (c) residue after treatment (TPCL), (d) residue after bleaching (BPCL), (e) cellulose obtained through extraction.

The yield of extracted cellulose was determined to be 51.64 ± 1.33 wt.%, a finding that closely aligns with earlier research. For example, Tuyet Phan and colleagues [32], found a cellulose yield of 51.13 ± 4.17 wt.% from waste of pineapple leaves, whereas Nguyen and others [39], noted a yield of 55 ± 1.75 wt.% from pineapple leaves in Vietnam. These findings are comparable and indicate uniformity across various geographical areas and processing techniques. Additionally, the cellulose yield from pineapple leaf waste is significantly greater than that from other agricultural biomasses, with figures like 37.67 wt.% from Baobab fruit shells [33], and 32 wt.% from rice straw [32, 34]. The comparatively elevated cellulose level in pineapple leaves is beneficial, as it improves the economic feasibility of cellulose extraction and guarantees reduced expenses for following cellulose derivatives. This trait is especially advantageous for industrial uses where

cost-effectiveness is vital, positioning pineapple leaf waste as a valuable resource for eco-friendly cellulose manufacturing. Furthermore, the increased cellulose yield could lead to enhanced performance and quality of the resulting cellulose-derived products, further reinforcing the promise of this biomass as a significant resource.

3.1.2. CMC Yields

(1) *Effect of NaOH Concentration on DS and Yield of CMC.* The main goal of using sodium hydroxide (NaOH) as a reagent is to cause swelling in cellulose chains, enabling the replacement of hydroxyl groups with carboxymethyl groups, leading to the production of CMC. This change is crucial for improving the solubility and functional characteristics of cellulose in multiple uses.

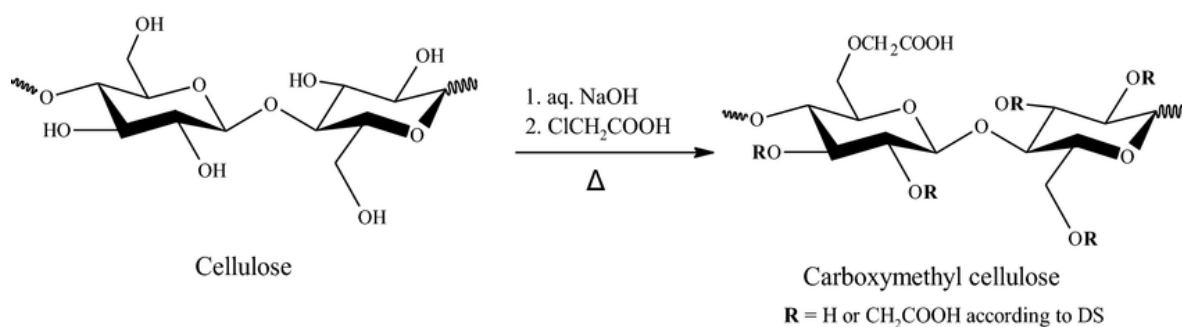


Figure 2. Reaction scheme of etherification of cellulose into carboxymethyl cellulose [40].

Table 1 shows the DS of the CMC generated at different NaOH concentrations. The DS value is an essential factor, as it indicates the degree to which carboxymethyl groups have replaced the hydroxyl groups in the cellulose, affecting the characteristics and possible applications of the resultant CMC.

Controlling the NaOH concentration allows for the optimization of reaction conditions, enabling the customization of CMC properties for particular industrial or commercial applications.

Table 1. Cellulose yield with various NaOH concentrations.

NaOH, %wt.	8	12	16	20
$H_{CMC}, \%$	163.1	172.3	178.8	172.6
DS	0.65	0.74	2.21	0.86

As shown in Table 1, the DS of CMC demonstrated a positive relationship with the NaOH concentration, attaining its highest DS value of 2.21 at a 16% (w/v) NaOH concentration. Nevertheless, additional rises in NaOH concentration past this juncture resulted in a significant drop in DS values. This trend can be explained by the interaction of various factors throughout the carboxymethylation process. The interaction of sodium hydroxide with cellulose's hydroxyl groups aids in the infiltration of NaOH into the cellulose framework, leading to changes in its morphology. This morphological change entails the alteration of crystalline areas into amorphous ones, thus improving the accessibility of cellulose for carboxymethylation processes [32, 34, 39].

Nonetheless, the degree of this morphological change is restricted and is influenced by several factors including the type of solvent, concentration of reagents, and the intrinsic characteristics of the cellulose substrate. It is noteworthy that in the carboxymethylation process, a side reaction can take place between monochloroacetate (MCA) and NaOH, especially at elevated concentrations of NaOH [32, 34]. This

competing reaction may impede the etherification of cellulose by decreasing the availability of MCA, thereby constraining the DS. At very high NaOH concentrations, the speed of this side reaction rises, altering the reaction pathway away from carboxymethylation and resulting in a lower DS value [32, 40].

The trends observed in DS align with earlier research by Chumee and Seeburin [41], Sunardi *et al.* [42], Tuyet Phan *et al.* [32] (2021), and Nguyen *et al.* [39], who noted comparable results concerning the influence of NaOH concentration on CMC characteristics. Additionally, Table 1 shows the CMC yields at different NaOH concentrations, which exhibit a trend similar to the DS values. This indicates that with an increase in NaOH concentration, the reaction efficiency first enhances, but after reaching an optimal concentration, both DS and yield start to decline, probably due to the prevalence of side reactions and the reduced efficacy of the carboxymethylation process. These findings emphasize the necessity of precisely managing NaOH concentration to enhance both the degree of substitution and yield in CMC synthesis [39].

Table 2. The yield and DS of synthesized CMC with various amount of MCA.

Amount of MCA, g	1	2	3	4
$H_{CMC}, \%$	153.7	172.3	178.8	169.1
DS	0.58	0.71	2.21	0.79

(2) *Effect of MCA Weight on DS and Yield of CMC.* The effect of monochloroacetate (MCA) mass on the DS of CMC was studied by changing the MCA quantity from 1.0 g to 4.0 g,

with the findings presented in Table 2. With the rise in MCA from 1.0–4.0 g, the DS of CMC steadily grew, peaking at 3.0 g of MCA. Nevertheless, additional rises in MCA past this

threshold led to a slight reduction in the DS. This observation indicates that the ideal quantity of MCA for attaining the maximum DS is at 3.0 g, after which the reaction effectiveness seems to decrease.

A potential reason for this reduction is the presence of an unwanted side reaction at elevated MCA concentrations. At high MCA levels, the surplus reagent may not completely engage in the carboxymethylation reaction with cellulose, resulting in by-products or unreacted MCA. This leads to a decrease in CMC yield along with a related decline in DS. This phenomenon aligns with earlier research, including studies by Tuyet Phan *et al.* [32], on pineapple leaf waste, Chumee and Seeburin [41], on pomelo peel waste, and Nguyen *et al.* [39], on pineapple leaf waste from Vietnam, which reported comparable DS values between 0.58 and 2.30 under similar experimental circumstances.

Besides DS values, Table 2, displays the CMC yields at different MCA concentrations, showing a pattern akin to that of DS. This indicates a direct correlation between the efficiency of carboxymethylation and MCA concentration, with the maximum yield and DS noted at 3.0 g of MCA. According to these results, the ideal conditions for the carboxymethylation process were found to be 5 g of cellulose, 3.0 g of chloroacetic acid (MCA), and 15 mL of a 16% (w/v) NaOH solu-

tion. In these circumstances, the resulting CMC displayed a DS value of 2.21. These findings emphasize the necessity of precisely regulating the MCA concentration to enhance the degree of substitution while reducing side reactions that might negatively impact both DS and yield throughout the carboxymethylation process.

3.2. Characterization

3.2.1. Fourier Transform Infra-Red – Analysis

FTIR spectroscopy was utilized to verify the structural properties of raw pineapple crown leaf waste, extracted cellulose, and synthesized CMC, as illustrated in Figure 3.

The FTIR spectrum of the unprocessed pineapple crown leaves waste (Figure 3a) shows various significant characteristics that reflect its compositional elements. Significantly, the absorption band at 1623 cm^{-1} is mainly linked to the C=O stretching vibration of the carbonyl group found in the acetyl groups of xylan, a component of hemicellulose, along with structural characteristics commonly associated with lignin [43]. Additional notable bands detected in the raw material spectrum align with the functional groups identified in cellulose, affirming the existence of lignocellulosic biomass.

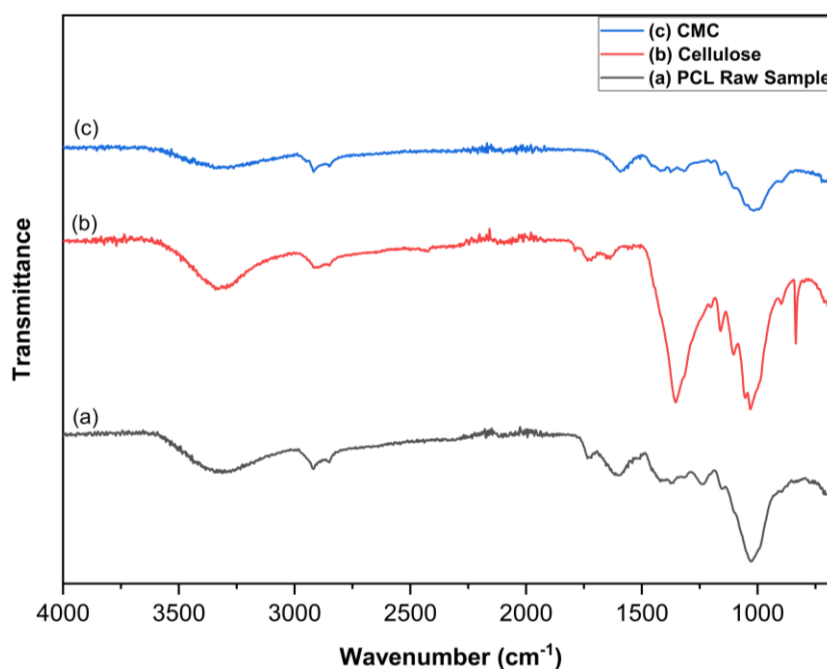


Figure 3. FTIR spectrum of unprocessed PCL sample (a), isolated cellulose (b) and produced CMC (c).

In the FTIR spectrum of the isolated cellulose (Figure 3b), clear peaks typical of cellulose are seen. The wide absorption band at 3326 cm^{-1} is associated with the O-H stretching vibration, signaling the presence of hydroxyl groups in cellulose. The peaks at 2914 cm^{-1} and 1354 cm^{-1} relate to C-H deformation vibrations, characteristic of the glucose units comprising cellulose. Moreover, the peak observed at 1159 cm^{-1}

corresponds to the stretching vibrations of the -C-O-C group, indicative of the β -(1, 4)-glycosidic linkages present in the cellulose backbone. The peak observed at 1105 cm^{-1} corresponds to the -C-O bond found in secondary alcohols and ethers present in the cellulose framework, whereas the peak at 894 cm^{-1} signifies the β -(4, 1)-glycosidic connections between glucose units in cellulose [41]. These spectral attributes

verify the existence of cellulose and its distinctive molecular arrangement.

The FTIR spectrum of cellulose obtained from Vietnamese pineapple leaf waste closely resembles that documented in the research by Bolio-López *et al.*, [44]. Crucially, the lack of absorption peaks at 1623 cm^{-1} , generally linked to the carbonyl (C=O) and aromatic ring functional groups found in hemicellulose and lignin [42, 45], indicates that these elements were successfully eliminated during the extraction process. The lack of peaks further confirms the high purity of the obtained cellulose, which was later utilized in the synthesis of CMC.

In the FTIR spectrum of synthesized CMC (Figure 3c), characteristic O-H stretching vibrations are evident at 3335 cm^{-1} , though with a broader band resulting from intermolecular and intramolecular hydrogen bonding within the cellulose framework. The peaks at 2962 cm^{-1} and 2851 cm^{-1} are associated with C-H stretching vibrations, whereas the peaks at 1052 cm^{-1} and 1026 cm^{-1} pertain to the β -(1, 4)-glycosidic bonds, indicating that the cellulose backbone remains intact throughout the carboxymethylation process. The CMC spectrum distinctly displays significant absorption bands at 1586 cm^{-1} and 1416 cm^{-1} , linked to the C=O stretching vibrations of the carboxyl group (-COO) and the sodium carboxylate group (-COONa), validating the effective etherification of cellulose. The FTIR spectrum of pure cellulose (Figure 3b) lacks these peaks, offering additional proof of the effective alteration of the cellulose structure to create CMC.

The results of the FTIR analysis provided here align with previous research on the carboxymethylation of cellulose from different plant origins. For instance, comparable spectral characteristics were observed by Chumee and Seeburin [41],

for pomelo peel waste, by Sunardi *et al.* [42], for purun tikus, and by Sophonputtanaphoca *et al.* [46], for pineapple leaves in Thailand. These results show the reliability of the carboxymethylation process and the dependability of FTIR as a method for verifying structural changes in cellulose-based materials.

3.2.2. X-ray Diffraction (XRD)

The level of crystallinity indicates the amount of crystalline content in a sample, and it is an essential attribute for comprehending the structural features of cellulose-derived materials [47]. The crystalline areas in cellulose mainly arise from hydrogen bonding among cellulose chains and Van der Waals interactions between cellulose molecules [48].

Figure 4, display the X-ray diffraction (XRD) patterns for waste from raw pineapple crown leaves, cellulose that has been extracted, and carboxymethyl cellulose (CMC). When comparing the diffractograms of untreated pineapple crown leaves waste and the cellulose obtained post-chemical treatment, it is clear that there is a rise in the intensity and a sharpening of the prominent diffraction peaks. This indicates an effective elimination of amorphous non-cellulosic elements, a conclusion that is additionally supported by the FTIR findings. These results differ from the findings of Bolio-López *et al.* [44], who observed distinct diffraction patterns for cellulose obtained from different sources. However, the present results correspond with those of Tuyet Phan *et al.* [32], Chumee and Seeburin [41], and Nguyen *et al.* [39], who likewise noted comparable trends in the crystallinity of cellulose derived from pineapple leaf waste and other organic residues like pomelo peel waste.

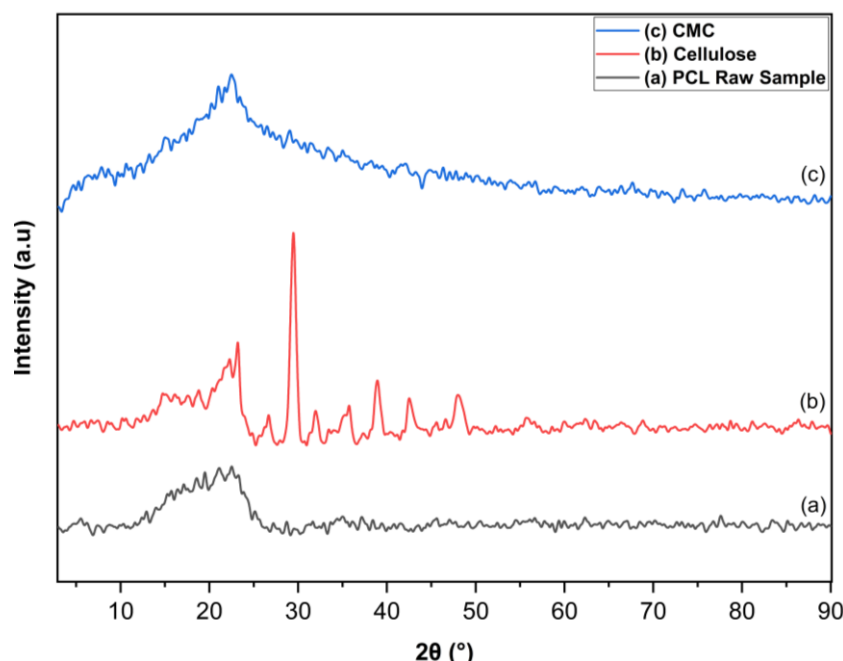


Figure 4. X-ray diffractogram of raw PCL sample (a), extracted cellulose (b) and synthesized CMC (c).

Based on the XRD analysis, it can be concluded that CMC shows a decreased level of crystallinity when contrasted with extracted cellulose. The XRD pattern of the obtained cellulose shows multiple sharp peaks ranging from 15° to 70° , signifying a well-ordered crystalline arrangement. This indicates that the extracted cellulose has a higher percentage of crystalline areas than the amorphous parts. In comparison, the diffractogram of CMC displays fewer and less pronounced peaks, indicating a more amorphous structure. The diminished intensity and decreased number of peaks in the CMC diffractogram suggest that the cellulose's crystallinity has been affected by the carboxymethylation process.

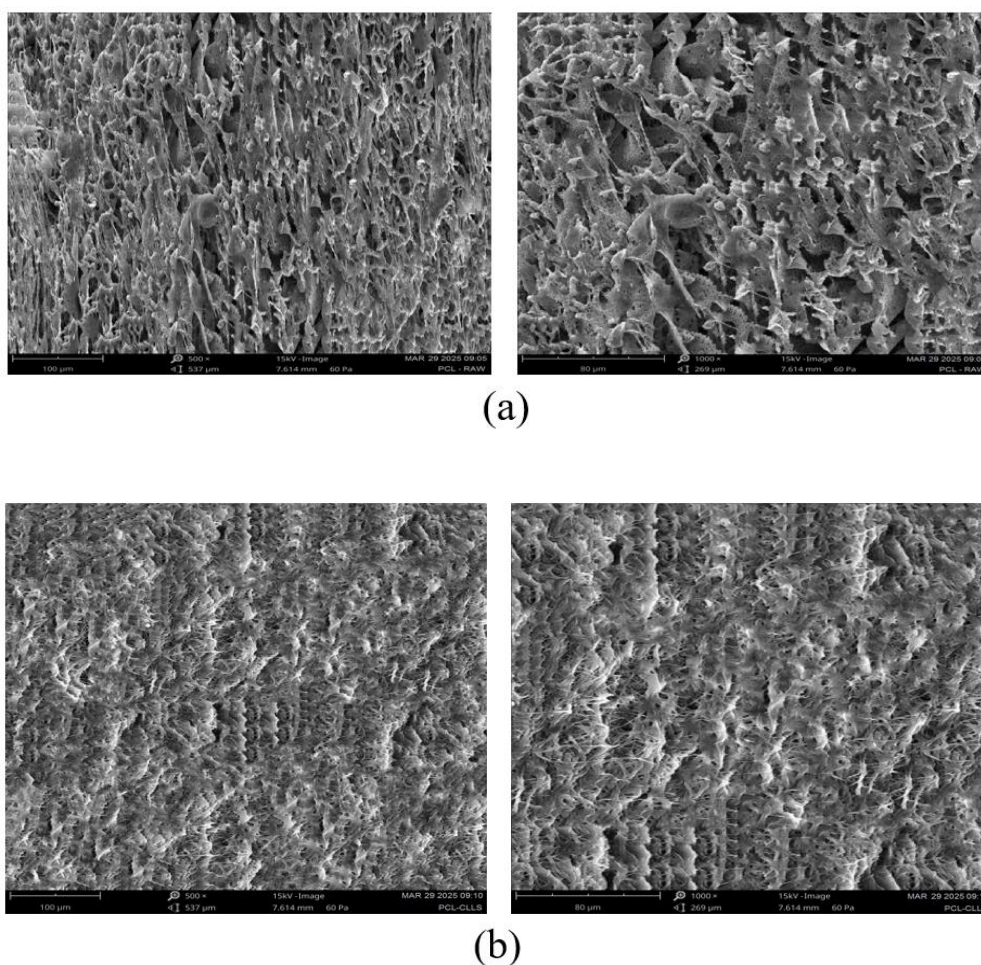
This reduction in crystallinity can be linked to the addition of carboxymethyl groups into the cellulose framework during the carboxymethylation process. The addition of these large, charged carboxymethyl groups interferes with the structured alignment of cellulose chains, resulting in a rise in the amorphous areas of the substance. Consequently, CMC shows a more disordered molecular structure and a lower degree of crystallinity than the original cellulose. This occurrence aligns with earlier research, such as that of Feng and Wen [49],

Tuyet Phan *et al.* [32], and Nguyen *et al.* [39], which emphasized the structural alterations in cellulose due to carboxymethylation, notably the decrease in crystallinity and the creation of increased amorphous structures [32].

The findings showed that the XRD analysis demonstrates a considerable decrease in cellulose crystallinity after carboxymethylation, probably because of the disturbance caused by the inclusion of carboxymethyl groups. These findings align with earlier research and show the effective transformation of cellulose into CMC, a substance with modified structural and crystallinity characteristics appropriate for multiple uses [39].

3.2.3. SEM-EDX Analysis

The scanning electron microscopy (SEM) was utilized to analyze the materials' microstructural and morphological characteristics. Figure 5 shows the SEM micrographs of unprocessed pineapple crown leaf waste (a), cellulose that has been extracted (b), and synthesized CMC (c).



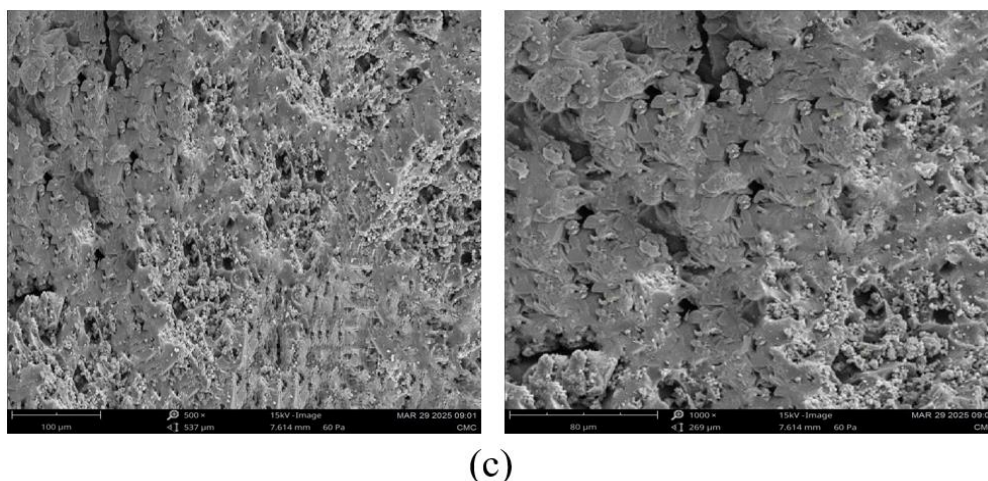
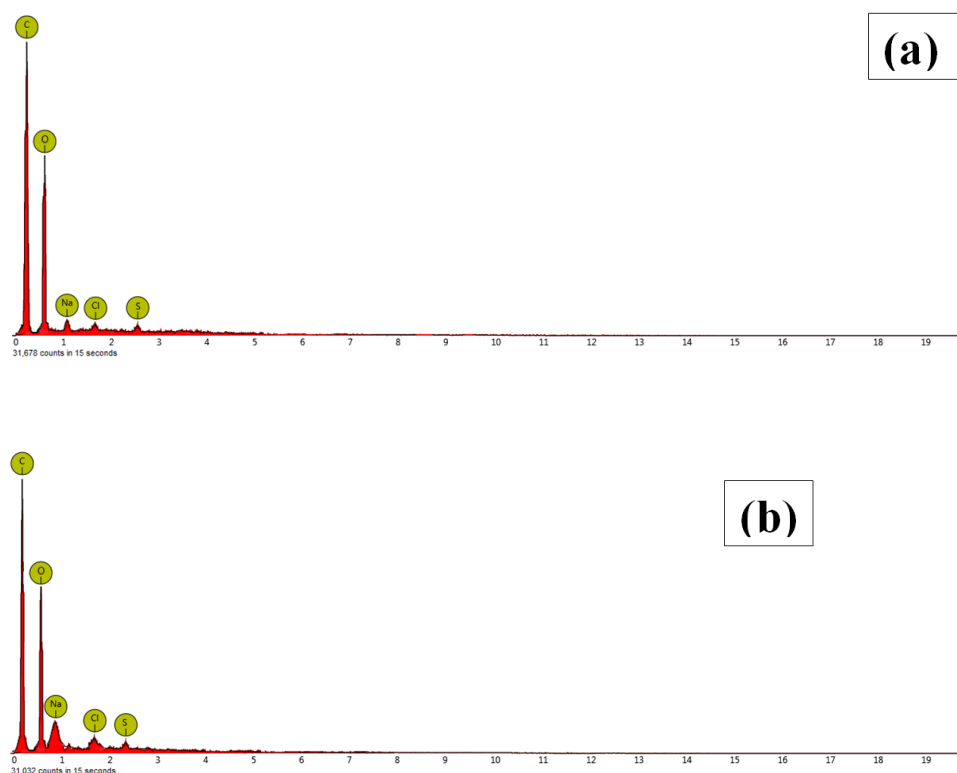


Figure 5. SEM images of raw PCL (a), extracted cellulose (b) and synthesized CMC (c).

The SEM analysis indicates that the treatment method greatly affects the morphological structure of the produced materials. Before treatment, the unprocessed fibers had a coarse surface, while after treatment, noticeable morphological alterations were noticed. Both the extracted cellulose and the synthesized CMC exhibited a ribbon-shaped or rod-like morphology, aligning with observations made in earlier research [44, 46]. Moreover, Figure 5 shows that the obtained cellulose fibers exhibited smooth surfaces with slight structural impairment. In comparison, the surface of the produced CMC seemed larger, uneven, and somewhat caved in. This change in morphology is due to the carboxymethylation pro-

cess, in which cellulose underwent treatment with sodium hydroxide [34].

Furthermore, Figure 5 offers information regarding the particle size distribution of the samples. The waste from raw pineapple crown leaves, the extracted cellulose, and the synthesized CMC showed a particle size distribution focused around 537 µm. This change in surface morphology and particle size distribution further supports the structural alterations caused by the chemical treatment process. Several other studies demonstrate results similar to those mentioned in this article [50-55].



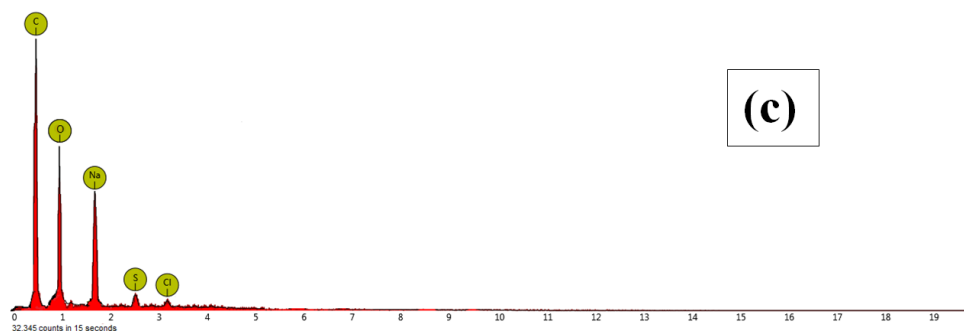


Figure 6. The EDX spectra of raw PCL (a), extracted cellulose (b) and synthesized CMC (c).

Figure 6 illustrates the EDX spectra of untreated PCL waste, extracted cellulose, and produced CMC, displaying distinctive peaks for oxygen (O) and carbon (C), validating the cellulose composition. The spectra demonstrate that cellulose stayed chemically unchanged during all processing stages, such as alkali treatment, bleaching, acid hydrolysis, and carboxymethylation, aligning with earlier research [54-56]. The elemental analysis shows that carbon and oxygen prevail in all samples, with significant variations in their ratios throughout processing. A reduction of carbon in cellulose and a rise in oxygen in CMC indicate chemical changes. The detection of sodium (Na) in CMC signifies the addition of sodium-containing compounds during carboxymethylation, while small quantities of chlorine (Cl) and sulfur (S) imply leftover impurities or byproducts from the treatment process. These findings emphasize the chemical resilience and versatility of lignocellulosic substances during the transformation into cellulose and CMC [54].

4. Conclusion

This research effectively showcases the extraction of cellulose from PCL waste and its later transformation into CMC via an optimized carboxymethylation method. The extraction method using NaOH and HNO₃ at 90 °C achieved a peak cellulose recovery of 51.64 ± 1.33 wt.%. Carboxymethylation performed at 60 °C for 1.5 hours with chloroacetic acid yielded CMC with a substantial degree of substitution (DS = 2.21), signifying effective alteration of the cellulose structure.

Advanced characterization methods offered essential insights into the materials' structural and compositional changes. FTIR analysis validated the efficient elimination of hemicellulose and lignin, along with the successful incorporation of carboxymethyl functional groups. XRD findings demonstrated a notable decrease in cellulose crystallinity after carboxymethylation, underscoring the disturbance of its organized structure from the addition of carboxymethyl groups. SEM imaging additionally highlighted the morphological changes, showing that the extracted cellulose had a smooth surface, whereas the synthesized CMC revealed an extended, rough, and partially collapsed structure as a result of chemical

modification. Moreover, analysis of particle size distribution indicated an average size around 537 µm, while EDX analysis confirmed the high purity of the obtained cellulose.

These results highlight the practicality of transforming pineapple crown leaf waste into valuable biopolymer products. The research not only encourages sustainable waste use but also provides an alternative option for environmentally friendly CMC manufacturing. These advancements carry important consequences for multiple sectors, such as pharmaceuticals, food, and biomaterials, highlighting the importance of renewable resources in material development.

Abbreviations

CMC	Carboxymethyl Cellulose
NaOH	Sodium Hydroxide
HNO ₃	Nitric Acid
DS	Degree of Substitution
FTIR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Diffraction
SEM	Scanning Electron Microscopy
EDX	Energy-dispersive X-ray Spectroscopy
PCL	PINEAPPLE Crown Leaves
H ₂ SO ₄	Concentrated Sulfuric Acid
MCA	Monochloroacetate
-COONa	Sodium Carboxylate Group

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Data Availability

No data was used for the research described in the article.

Conflicts of Interest

The authors declare no conflicts of interest.

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