



Synthesis and Characterization of Carboxymethyl Cellulose from Sugarcane Bagasse Cellulose Using Physical and Chemical Pretreatments

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Abstract: This study investigates the optimization of carboxymethyl cellulose (CMC) synthesis from sugarcane bagasse cellulose through combined physical and chemical pretreatments. Bagasse, an abundant lignocellulosic byproduct of sugarcane juice extraction, underwent a sequential combination of heating, alkali treatment, and chloroacetic acid modification to obtain CMC. The synthesized CMC was characterized for degree of substitution (DS), viscosity, surface morphology, and functional group composition. Viscosity analysis was increased from 1.02 cP (0% w/v) to 6,770.35 cP (2.5% w/v), demonstrating excellent thickening properties suitable for various industrial applications. The DS value of 0.94 ± 0.21 indicated a high degree of hydroxyl substitution by carboxymethyl groups. Scanning electron microscopy (SEM) revealed a highly porous, roughened surface structure, reflecting the removal of lignin and hemicellulose during pretreatment. Fourier Transform Infrared Spectroscopy (FTIR) confirmed the successful incorporation of carboxymethyl groups, with distinct peaks at 1613 cm^{-1} (C=O stretching) and 1152 cm^{-1} (C–O–C stretching). The results demonstrate that optimized pretreatment conditions enhance cellulose purity and reactivity, yielding high-quality CMC with desirable physicochemical properties. This approach offers a sustainable pathway for valorizing agricultural waste into high-value biopolymers with potential applications in the food, pharmaceutical, and cosmetic industries.

Keywords: Carboxymethyl cellulose; sugarcane bagasse; physical pretreatment; chemical modification

1. Introduction

The southernmost provinces of Thailand are predominantly agricultural regions, generating high annual yields that serve as the main source of income and livelihood for local farmers. However, over the past decade, agricultural production has declined, leading to economic losses and daily hardships for farmers. Although government agencies have attempted to revitalize the agricultural economy through various mechanisms and policies, success has been limited. In response, new policies promoting integrated farming have been introduced to increase farmers' income opportunities and economic stability. The promoted crops include banana, durian, cassava, and sugarcane—each offering continuous economic value. Notably, sugarcane can be cultivated year-round and serves as a lignocellulosic raw material composed primarily of cellulose (45–55%), hemicellulose (20–30%), lignin

(15–29%), and extractives (1–5%) [1-3, 5]. During the month of Ramadan each year, in regions where the local population is predominantly Muslim and observes fasting, fresh sugarcane juice is widely consumed and regarded as a highly popular beverage. This seasonal demand leads to a significant increase in sugarcane consumption and results in large quantities of bagasse waste. Bagasse, a fibrous residue from the sugarcane juice extraction process, is highly flammable and primarily composed of carbon, hydrogen, oxygen, and nitrogen [7, 10, 14-16].

Bagasse can be utilized in various ways, such as producing briquette fuel, charcoal briquettes, concrete blocks, renewable energy in the form of bioethanol and biogas, and construction materials. However, research related to extracting carboxymethyl cellulose (CMC) from bagasse remains limited, and further product development is needed. Improving extraction methods begins with pretreating bagasse to obtain high-quality cellulose and hemicellulose, which is therefore an important step. Although previous studies have reported the synthesis of CMC from sugarcane bagasse, limited attention has been given to optimizing pretreatment conditions to improve cellulose quality and their impact on the physicochemical properties of the synthesized CMC. Therefore, this study focuses on improving the pretreatment process to obtain high-purity cellulose and evaluating its suitability for producing CMC with desirable functional properties [3]. Cellulose and Hemicellulose are water-insoluble polysaccharides composed of glucose units, similar to starch. Cellulose molecules are arranged in straight chains without branching, and hydrogen bonding between hydroxyl and oxygen groups results in high crystallinity, superior strength, and greater structural stability compared to starch. Carboxymethyl cellulose is an important material across various industries, including the pharmaceutical industry, where it is used as a binder in tablet production and as a thickening agent in liquid formulations. The food industry uses them as thickeners, texture enhancers, and stabilizers in emulsions. The cosmetic industry has potential as a natural alternative to synthetic thickeners and texture modifiers. However, research in this context remains limited [4-7]. The conversion of lignocellulosic biomass into value-added products, including cellulose and carboxymethyl cellulose (CMC), requires the development of cost-effective processing methods. This process involves several critical stages, including pretreatment of lignocellulosic materials to remove lignin and hemicellulose, isolation and purification of cellulose, and subsequent chemical modification to synthesize CMC. Each stage must be carefully designed to ensure process efficiency, product quality, and scalability. So, it is essential to establish an optimized approach to enhance the effectiveness of biomass conversion and also minimize production costs. The development of cost-effective and efficient methodologies is particularly important for enabling the sustainable utilization of lignocellulosic resources and supporting their potential application in industrial and environmental sectors [11, 19, 21-24]. Therefore, the objective of this study is to explore a novel approach for synthesizing carboxymethyl cellulose using cellulose derived from the pretreatment of sugarcane bagasse. This process aims to develop and create added value to agricultural waste, corresponding for promising strategy for both economic development and sustainable resource use.

2. Materials and Methods

2.1 Synthesis of CMC from Bagasse-Derived Cellulose by Physical and Chemical Pretreatment

Carboxymethyl cellulose (CMC) was synthesized from bagasse cellulose following the method of Kaewprachu et al. (2022), with slight modifications. Briefly, 5 g of purified cellulose was placed into a 250 mL glass beaker. 8 g of Chloroacetic acid was dissolved in 20 mL of isopropanol and added to the cellulose. Subsequently, 40 mL of 40% NaOH solution was slowly added under continuous stirring to initiate the alkalization and etherification reactions. The mixture was then soaked for 30 minutes, covered with aluminum foil, and heated at 55 °C for 3.5 hours. The liquid was filtered out, and the remaining cellulose was washed with 40 mL of 70% methanol, neutralized with 10% citric acid solution, and further washed with 40 mL of 70% ethanol, soaking for 10 minutes each time, for a total of 5 washes. The sample was then dried at 55 °C for 24 hours to obtain CMC powder, which was stored in an airtight desiccator container for further analysis, including CMC yield (1), viscosity measurement, and degree of substitution [4, 8-12, 17]. scanning electron microscopy (SEM), and Fourier-transform infrared spectroscopy (FT-IR). All experiments were conducted in triplicate.

$$\text{Yield (\%)} = \frac{W_{\text{dry CMC}}}{W_{\text{initial cellulose}}} \times 100 \quad (1)$$

Where:

$W_{\text{dry CMC}}$ = weight of dried synthesized CMC

$W_{\text{initial cellulose}}$ = weight of starting cellulose

2.2 Viscosity Testing of Synthesized CMC

CMC solutions were prepared at concentrations ranging from 0.5 to 2.5% w/v by gradually dispersing the CMC powder into distilled water under moderate stirring using a magnetic stirrer (Magnetic Stirrer; IKA, model C-MAG HS 9). Stir until the solution becomes homogeneous and allow it to hydrate for 2 hours. Adjust the test temperature to 25.0 ± 0.1 °C using a temperature-controlled water bath (Water Bath; Memmert, model WNB 9). Viscosity measurements were performed using a Brookfield rotational viscometer (Model DV2T LV, Brookfield Engineering Laboratories, USA) equipped with LV spindle No. 3. The spindle rotation speed was set to 60 rpm, maintaining torque within the recommended range of 10–90%. Record the apparent viscosity (cP) once the reading stabilizes, and repeat the measurement at least three times. [13–15].

2.3 Sample Preparation and Determination of Degree of Substitution (DS) of CMC

The CMC sample was dried at 60 °C for 12 hours, then cooled in a desiccator. Approximately 0.5 g of the CMC sample was accurately weighed and dispersed in 50 mL of distilled water. Then, 25 mL of 0.1 M hydrochloric acid (HCl) was added to convert the sodium carboxymethyl groups into their acid form, and the mixture was treated with 20 mL of 0.1 N hydrochloric acid (HCl) to convert the $-\text{COO}^-\text{Na}^+$ groups into $-\text{COOH}$. The mixture was stirred and heated at 65 ± 5 °C for 15 minutes, followed by the addition of ethanol to precipitate the CMC. The precipitate was filtered and washed with ethanol until free of sodium salts. The precipitate was then redispersed in a known volume of distilled water and titrated with 0.1 N NaOH to the phenolphthalein indicator, turning faint pink and persisting for at least 30 seconds. The volume of NaOH used for the sample (V_{sample}) and for a blank (V_{blank} , prepared under the same conditions without CMC) were recorded. The content of $-\text{CH}_2\text{COONa}$ per gram of sample (A) was calculated using formula (2). [16–20, 22]

$$A = \frac{(V_{\text{Sample}} - V_{\text{Blank}}) \times N_{\text{NaOH}} \times 80}{m} \quad (2)$$

where N_{NaOH} is the normality of NaOH (N), 80 is the molar mass of $-\text{CH}_2\text{COONa}$ (g/mol), and m is the sample mass (g). The Degree of Substitution (DS) was then calculated using formula (3).

$$\text{DS} = \frac{162 \times A}{(58 \times 1000) - (80 \times A)} \quad (3)$$

where A is milliequivalents of carboxymethyl groups per gram of sample; 162 is the molar mass of the anhydroglucose unit (g/mol), 58 is the molar mass of $-\text{CH}_2\text{COO}^-$ (g/mol), and 80 is the molar mass of $-\text{CH}_2\text{COONa}$ (g/mol). The results were reported as the mean $\pm$ standard deviation from at least three replicates.

2.4 Structural Characteristics of CMC from Pretreated Bagasse Observed by Scanning Electron Microscopy

Morphological changes of sugarcane bagasse during processing were examined by scanning electron microscopy (SEM; Model JSM-5800, JEOL, Japan or equivalent). Dried samples, including raw bagasse, extracted cellulose (after pretreatment), and synthesized CMC, were mounted on aluminum stubs using conductive carbon tape. Samples were sputter-coated with a thin gold layer (~5–10 nm) to reduce charging during imaging. SEM observation was performed at an accelerating voltage of 15–20 kV. Micrographs were captured at multiple magnifications (3,000 $\times$ for overall fiber morphology and 50,000 $\times$ for surface microstructure). The magnification and scale bar for each image are shown directly on the micrograph [18, 21, 24–26].

2.5 Functional Group Analysis of Carboxymethyl Cellulose (CMC) Using Fourier Transform Infrared Spectroscopy (FTIR)

The synthesized CMC samples were dried at 60 °C for 24 hours and sieved through an 80-mesh screen to ensure uniform particle size. Approximately 2.00 mg of dried CMC sample was accurately weighed and thoroughly mixed with 98.00 mg of dry spectroscopic-grade potassium bromide (KBr), corresponding to a 2.00 wt% sample-to-KBr ratio. The mixture was finely ground in an agate mortar and pestle and compressed into a transparent pellet under constant pressure using a hydraulic press. FTIR spectra were recorded using a Bruker Tensor II Fourier Transform Infrared Spectrometer (Bruker Optics GmbH, Germany). Measurements were conducted over the spectral range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans per sample. Background spectra were recorded using pure KBr under identical conditions before sample analysis. All measurements were performed at ambient temperature (25 ± 2 °C). The obtained spectra were analyzed to identify characteristic functional groups and confirm the successful substitution of hydroxyl groups by carboxymethyl groups on the cellulose backbone. [17, 25-27]

3. Results and Discussion

When testing viscosity at concentrations ranging from 0 to 2.5% (w/v), it was found that the viscosity values of CMC solutions at various concentrations could be used to evaluate changes in viscosity as the CMC concentration increased. The experimental results showed that the viscosity of the CMC solution increased significantly with increasing concentration, from an initial value of 1.02 ± 0.58 cP at 0% (w/v) (pure water) to $6,770.35 \pm 325.21$ cP at 2.5% (w/v). Low-concentration solutions (0.1–0.5% w/v) exhibited viscosities of 5.53–88.25 cP, indicating a relatively moderate increase. However, as the CMC concentration entered the medium range (0.6–1.5% w/v), viscosity increased sharply from 147.53 ± 21.25 cP to $1,284.77 \pm 135.21$ cP, reflecting the formation of more interconnected polymer structures, which progressively increased flow resistance. At higher concentrations (1.6–2.5% w/v), viscosity rose rapidly and markedly, ranging from $1,682.08 \pm 111.52$ cP to $6,770.35 \pm 286.36$ cP (Figure 1). This rapid increase suggests that the longer the time, the more complex the structures formed in the solution, hence the greater the flow resistance. CMC solutions are well known to exhibit pseudoplastic (shear-thinning) behavior, where apparent viscosity decreases with increasing shear rate due to alignment of polymer chains in the direction of flow. In this study, viscosity was measured at a fixed rotational speed; therefore, the values reported represent apparent viscosity under defined shear conditions. Future work will include rheological characterization across a range of shear rates to fully evaluate non-Newtonian flow behavior. Thus, CMC has great thickening ability and can control liquid viscosity. Thus, it can be used in the cosmetics, agriculture, food, and pharmaceutical industries that require defined viscosity. The higher the CMC concentration, the easier viscosity control is for the intended application. The DS value obtained in this study (0.94 ± 0.21) falls within the typical range reported for commercial CMC products. Food-grade CMC generally has DS values between 0.6 and 0.95, while technical-grade CMC ranges from 0.7 to 1.2. Previous studies on bagasse-derived CMC have reported DS values ranging from approximately 0.65 to 1.10, depending on synthesis conditions. The DS value obtained in this study is therefore comparable to both commercial and biomass-derived CMC, indicating successful etherification and confirming bagasse as a viable raw material for CMC production. [19, 22-24].

To analyze the degree of substitution of the CMC (carboxymethyl cellulose) sample, 1 g of CMC was diluted to 100 mL with distilled water. Then 25 mL of standardized hydrochloric acid (HCl, 0.1 M) was added to the CMC, which converted the carboxymethyl groups ($-\text{CH}_2\text{COONa}$) to carboxylic acid groups ($-\text{CH}_2\text{COOH}$). The suspension was heated to 60–70 °C for 30 minutes to ensure that all carboxymethyl groups were substituted, then cooled. After that, the mixture was titrated with NaOH by adding drops of NaOH solution until the endpoint was reached. The volume of titrated NaOH solution was then used to determine the number of NaOH moles, equivalent to the carboxymethyl groups present in the CMC sample. The degree of substitution for the sample was determined to be 0.94 ± 0.21 , which means that the extent of the substitution of carboxymethyl groups ($-\text{CH}_2\text{COOH}$) with hydroxyl groups ($-\text{OH}$) in the CMC sample was 0.94 ± 0.21 . Shown as Figures 2 and 3.

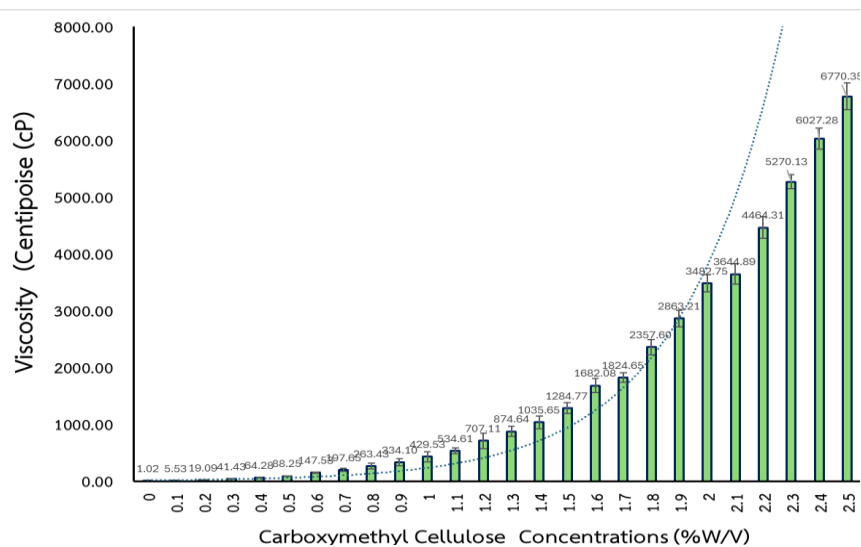


Figure 1. Apparent viscosity of synthesized CMC solutions at different concentrations (0–2.5% w/v). Values represent mean $\pm$ SD (n = 3).

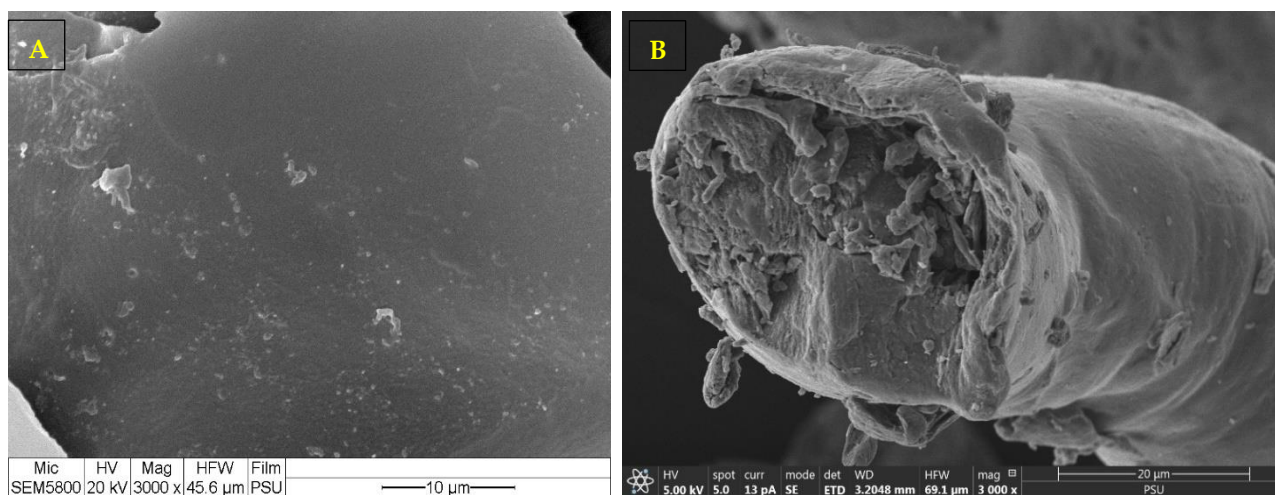


Figure 2. Morphological Characteristics of A) Before Synthesized, B) Synthesized Cellulose from Bagasse Observed by Scanning Electron Microscopy (SEM)

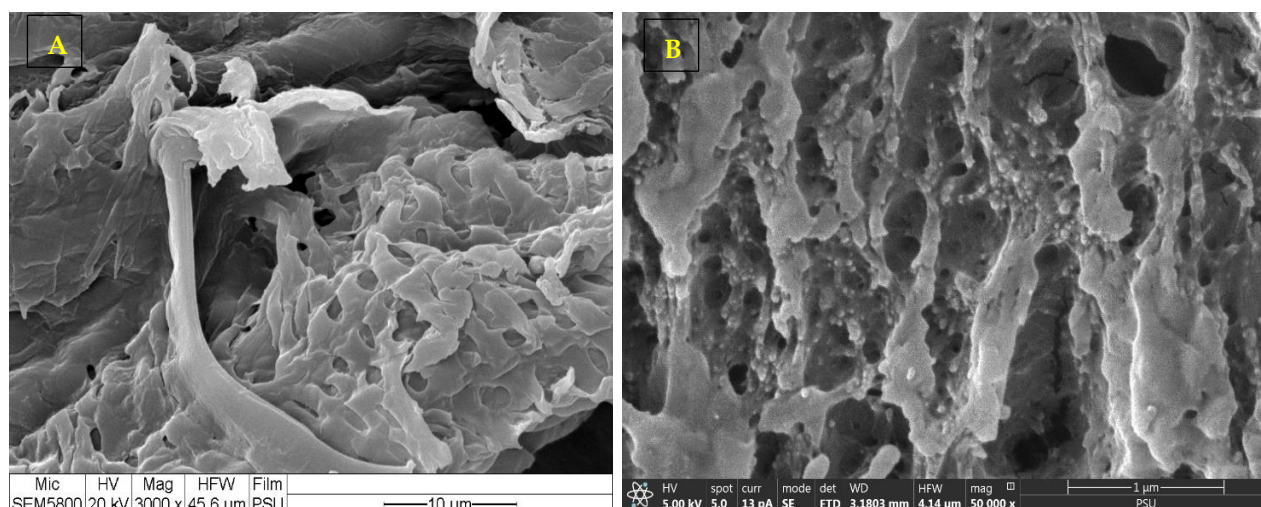
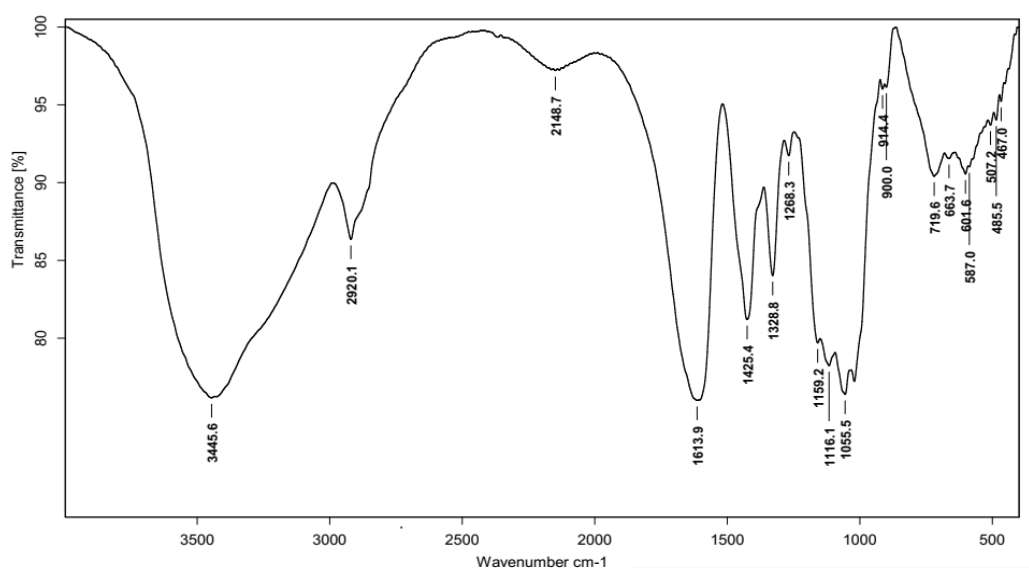


Figure 3. Morphological Characteristics of Synthesized Bagasse-Derived Carboxymethyl Cellulose (CMC) A) 3,000 X and B) 50,000 X observed by Scanning Electron Microscopy (SEM)

SEM images revealed significant structural differences between raw bagasse, extracted cellulose, and synthesized CMC (Figures 2 and 3). Raw bagasse exhibited compact fiber bundles with relatively smooth surfaces, likely due to lignin and hemicellulose acting as binding agents. After alkaline pretreatment and bleaching, the extracted cellulose showed cleaner, more separated fibers with increased surface roughness, indicating partial removal of non-cellulosic components. Following carboxymethylation, the CMC exhibited a substantially altered morphology characterized by disrupted fiber structure, increased surface irregularity, and reduced structural integrity of the original cellulose fibers. Image analysis indicated that the average fiber diameter decreased and the surface became more heterogeneous after modification. These morphological changes are consistent with etherification reactions that introduce carboxymethyl groups into the cellulose structure, disrupting hydrogen bonding and increasing hydrophilicity. The observed structural modification supports the increased solubility and viscosity of the synthesized CMC.

FTIR characterized the synthesized bagasse-derived CMC to confirm the introduction of carboxymethyl functionality. Broad absorption at $\sim 3445\text{ cm}^{-1}$ corresponds to O–H stretching, while the band at $\sim 2891\text{ cm}^{-1}$ is attributed to C–H stretching of the polysaccharide backbone. Importantly, the appearance/intensification of bands at 1613 cm^{-1} and 1424 cm^{-1} is consistent with the asymmetric and symmetric stretching vibrations of carboxylate (COO^-) groups in sodium carboxymethylcellulose (CMC-Na), supporting successful etherification. Additional bands at $\sim 1321\text{ cm}^{-1}$ (C–O stretching associated with substituted polysaccharide structure), $\sim 1152\text{ cm}^{-1}$ (C–O–C stretching of glycosidic linkages), and $\sim 900\text{--}897\text{ cm}^{-1}$ (β -glycosidic linkage-related vibration) were also observed, consistent with cellulose-derived materials. Therefore, it was not used for structural confirmation. The FTIR spectrum of the starting cellulose was included for comparison, showing markedly weaker/absent carboxylate bands, thereby strengthening evidence of chemical transformation to CMC (Figure 4).

Figure 4. FTIR Spectrum Characteristics of Bagasse-Derived CMC



The research began with the production of cellulose from bagasse using a combined physical and chemical pretreatment at high temperatures. This process efficiently extracted hemicellulose and lignin from the primary structure, thereby retaining cellulose largely intact as the main component. Although this pretreatment might have partially fractured cellulose molecules, the overall structure remained sufficiently intact to serve as an adequate substrate for the subsequent synthesis of carboxymethyl cellulose (CMC). These findings are consistent with those of Sharma et al. (2020), who reported that sodium hydroxide pretreatment of biomass cellulose enhances the cellulose fraction while efficiently extracting hemicellulose and lignin. Similarly, Chen et al. (2019) reported that sodium hydroxide facilitates the removal of hemicellulose and lignin, thereby yielding purified cellulose suitable for CMC synthesis. Furthermore, Wang et al. (2019) demonstrated that the application of plant-based CMC can significantly enhance the physicochemical properties of derived products. There are, however, several concerns regarding CMC synthesis, particularly the intensive chemical processes involved and the resulting environmental pollution. It should also be noted that although sodium hydroxide pretreatment yields high cellulose levels [28-30].

The findings of this study demonstrate that sugarcane bagasse is a viable, sustainable raw material for the synthesis of carboxymethyl cellulose (CMC) with favorable physicochemical properties. The applied synthesis route, involving alkaline pretreatment followed by chloroacetic acid etherification, effectively introduced carboxymethyl groups into the cellulose structure. This chemical modification significantly altered the material's physicochemical properties, particularly its solubility and rheological behavior. The formation of CMC occurs through a two-step reaction mechanism. Initially, the hydroxyl groups (-OH) on the cellulose chains react with sodium hydroxide to form alkali cellulose. This activation step increases cellulose reactivity by generating nucleophilic alkoxide groups. Subsequently, during the etherification stage, these activated sites react with chloroacetic acid via a nucleophilic substitution mechanism, in which the cellulose alkoxide group displaces the chlorine atom. This reaction results in the formation of sodium carboxymethyl cellulose (CMC-Na), introducing hydrophilic carboxymethyl groups ($\text{-CH}_2\text{COO}^-$) into the polymer backbone. The presence of these functional groups enhances intermolecular interactions with water molecules, thereby improving solubility and increasing viscosity. Despite the effectiveness of the synthesis process, competing side reactions may occur. In particular, chloroacetic acid may react directly with sodium hydroxide to form sodium glycolate rather than with cellulose. This undesired reaction reduces the availability of the etherifying agent and may contribute to variations in the degree of substitution (DS). Therefore, careful optimization of reaction parameters, including reagent concentration, temperature, and reaction duration, is essential to promote efficient substitution while minimizing by-product formation. The synthesized CMC exhibited a degree of substitution of 0.94 ± 0.21 , indicating a relatively high level of chemical modification. This value is within the typical range reported for commercial CMC, suggesting that the material possesses functional properties comparable to those required for industrial applications. The observed variability in DS may be attributed to structural heterogeneity within the cellulose matrix, which can influence the accessibility of hydroxyl groups during the reaction. Structural characterization using FTIR spectroscopy confirmed the successful incorporation of carboxymethyl groups. The presence of characteristic absorption bands associated with carboxylate functional groups provides clear evidence of chemical modification. In addition, scanning electron microscopy revealed notable changes in surface morphology following modification. The treated material displayed increased surface irregularity and disruption of the original fiber structure, indicating that the chemical reaction altered the physical organization of the cellulose fibers. These structural changes are consistent with increased accessibility of hydrophilic functional groups and improved interaction with aqueous media. The rheological analysis further demonstrated that the viscosity of the CMC solutions increased substantially with increasing concentration, reaching approximately 6,770 cP at 2.5% (w/v). This concentration-dependent increase in viscosity is characteristic of polymer solutions and is associated with increased polymer chain entanglement and intermolecular interactions. The observed viscosity behavior confirms that the synthesized CMC exhibits strong thickening capability, an important functional property for many industrial applications. The purification steps, including washing and neutralization, played an important role in removing residual reagents and by-products. The absence of unexpected functional group signals in the FTIR spectra suggests that these purification procedures were effective in producing a relatively clean final product. Based on its structural characteristics, degree of substitution, and rheological properties, the synthesized CMC shows considerable potential for practical applications. Its viscosity-enhancing properties make it suitable for use as a thickening and stabilizing agent in food systems, pharmaceutical formulations, cosmetic products, and agricultural preparations. In addition, using sugarcane bagasse as a starting material offers significant environmental and economic benefits. Converting agricultural waste into value-added functional materials supports sustainable resource utilization and aligns with the principles of circular bioeconomy and environmentally responsible material development.

4. Conclusions

Producing carboxymethyl cellulose (CMC) from sugarcane bagasse yielded a completely water-soluble product. The viscosity of the CMC solution increased proportionally with the concentration of dissolved CMC. The degree of substitution (DS) was determined to be 0.94 ± 0.21 , indicating successful incorporation of carboxymethyl groups ($\text{-CH}_2\text{COOH}$) in place of hydroxyl groups within the cellulose structure. Scanning electron microscopy (SEM) images revealed a highly porous surface with a complex network of interconnected pores, suggesting that the pretreatment process effectively removed lignin and hemicellulose while modifying the native cellulose structure. This treatment increased surface roughness and facilitated the formation of an open-pore structure. Fourier Transform Infrared Spectroscopy (FTIR) analysis confirmed the presence of distinctive functional groups in the synthesized CMC, with characteristic absorption peaks corresponding to O-H, C-H, C=O, and C-O-C bonds.

Notably, the peaks observed at 1613 cm⁻¹ (C=O stretching) and 1152 cm⁻¹ (C–O–C stretching) strongly supported the successful derivatization of cellulose into CMC.

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