

Synthesis and Characterization of Carboxymethyl Cellulose Derived from Empty Fruit Bunch Cellulose

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Abstract

Empty fruit bunches (EFB), a significant agricultural waste, represent a promising resource for value-added applications. This study focuses on the synthesis of carboxymethyl cellulose (CMC) from cellulose extracted from EFB, emphasizing the effect of sodium hydroxide (NaOH) concentration on the degree of substitution (DS), a critical parameter influencing CMC properties. The synthesis process involved cellulose alkalization followed by carboxymethylation, with NaOH concentration varied to investigate its impact on DS and other physicochemical characteristics. Characterization using Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD), and Scanning Electron Microscopy (SEM) confirmed the successful introduction of carboxymethyl groups, while DS measurements highlighted a positive correlation between NaOH concentration and DS. The highest DS value (1.34) was achieved at 50% NaOH, indicating that sufficient alkali concentration enhances the reactivity of cellulose and promotes substitution efficiency. These results demonstrate the feasibility of converting EFB cellulose into high-quality CMC with tailored properties, suitable for applications in biodegradable polymer materials and other sustainable technologies.

Keywords: carboxymethyl, cellulose, degree of substitution, empty fruit bunches

How to cite

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Highlights

1. Carboxymethyl cellulose (CMC) was prepared from empty fruit bunches (EFB) of palm oil.
2. The synthesis process involved cellulose alkalization followed by carboxymethylation.
3. The synthesized CMC exhibited a degree of substitution (DS) and physical properties comparable to or suitable for substitution with commercial CMC.
4. The DS of CMC increased as the NaOH concentration ranged from 20% to 50%,.
5. The study demonstrates a sustainable and value-added utilization of biomass waste, promoting environmental and economic benefits.



Introduction

Plant waste poses a significant global environmental challenge due to its abundance and underutilization, despite being a renewable resource (Paranjape & Sadgir, 2023). Transforming plant-based waste into valuable products offers a sustainable solution to mitigate environmental issues (Vorobyova et al., 2019). This waste is predominantly composed of carbohydrate polymers, including starch, cellulose, bagasse fiber, and cotton fiber, which can be chemically or physically modified to produce diverse value-added materials (Galiwango et al., 2019). Among such wastes, empty fruit bunches (EFB), a byproduct of the palm oil industry, are generated in substantial quantities (Permana et al., 2024). However, managing EFB effectively remains a challenge, as improper disposal can result in significant environmental pollution (Windiastruti et al., 2022). Simultaneously, the rising demand for biopolymer-based products reflects growing awareness of the importance of developing environmentally friendly and sustainable alternatives, underscoring the potential of plant waste as a valuable resource for green technologies.

Empty fruit bunches (EFB) are composed of cellulose, hemicellulose, lignin, and other components, making them a valuable resource for biomass utilization (Sitorus et al., 2022). Cellulose, the primary component for synthesizing carboxymethyl cellulose (CMC), is a high-molecular-weight linear homopolymer composed of repeating β -D-glucopyranosyl units (Çelikçi et al., 2022). Carboxymethyl cellulose (CMC), derived from cellulose, is a water-soluble anionic polysaccharide with excellent thickening properties (Rasid et al., 2021). The abundant hydroxyl and carboxylic groups in CMC contribute to its strong water-binding and absorption capabilities (Sun et al., 2022). The process consists of an equilibrium reaction where sodium hydroxide (NaOH) interacts with the

hydroxyl groups of cellulose, leading to the subsequent formation of carboxymethyl (CM) groups through the action of sodium monochloroacetate (SMCA) (Konovalenko et al., 2021). Cellulose derivatives such as CMC, which are water-soluble and biocompatible, can function as thickeners, binders, emulsifiers, film-formers, and lubricants (Seddiqi et al., 2021).

The effectiveness of CMC in various applications depends on its degree of substitution (DS), which refers to the number of hydroxyl groups replaced per anhydroglucose unit, with 3.0 being the maximum achievable value. A higher DS increases CMC's resistance to degradation, improving its compatibility with other soluble components (Rasid et al., 2021). Molecular weight and degree of substitution (DS) are important usability parameters of CMC (Candido & Gonçalves, 2016). The hydroxyl groups in cellulose were substituted with carboxymethyl groups primarily at the C-2, C-6, and C-3 positions (Haleem, Arshad, Shahid, & Tahir, 2014). Its hydrophilic properties, gel-forming ability, and adjustable viscosity make CMC versatile.

Limited research has been conducted on the synthesis of CMC from major agricultural crops in Indonesia, particularly EFB. Several studies have reported the synthesis and characterization of CMC from different locations of the EFB source, such as Riau (Hariani & Prayitno, 2020), Aceh (Trisnawita & Putri, 2024) and South Kalimantan (Susi et al., 2024). Many research studies related to CMC synthesis have used cellulose from the pineapple core (Suebsuntorn & Jirukkakul, 2023), wheat straw (Li et al., 2019), corncob (Singh et al., 2022), almond shells and waste paper (He et al., 2021). Therefore, the purpose of this study was to produce CMC from EFB cellulose. The influence of reaction parameters, such as NaOH concentration, was explored. The physical

and chemical changes of EFB cellulose to CMC were elaborated using FTIR and XRD.

Research Methods

Equipment and materials

The EFB cellulose from PT. Damai Sejahtera Kolaka was produced in our previous study (Permana et al., 2024). Methanol and ethanol were provided from the local market. Sodium monochloroacetate (SMCA), isopropanol, and sodium chlorite were purchased from Sigma-Aldrich, USA. Natrium hydroxide, glacial acetic acid, and potassium hydroxide were prepared from Merck Chemical Co., Germany. EFB, cellulose and CMC were characterized using X-ray Diffraction (XRD, XPert MPD) and Fourier Transform Infrared Spectroscopy (FTIR, 8400S Shimadzu). The DS of CMC was determined by using the acid-wash method. A Rapid Visco Analyzer (Model: RVA-4, Germany) was used to measure the viscosity of CMC.

Synthesis of carboxymethyl cellulose (CMC)

The process for preparing CMC from cellulose was taken by (Asl, Mousavi, & Labbafi, 2017). First, 9 g of EFB cellulose was mixed with 30 mL of 20 to 60% (w/v) aqueous NaOH in 270 mL of isopropanol at ambient temperature for 30 min to perform the alkalisation reaction. Subsequently, 10.8 g SMCA were added to the mixture and stirred for 1.5 h before being covered by aluminium foil and baked for 3 h at 55 °C. The filtrate was submerged in 100 mL of methanol (70%)

for 24 h and neutralized with 90% acetic acid to form a yellowish solid product. The solid product was washed with 300 mL of ethanol. Once a consistent weight was achieved, the generated CMC was dried in an oven at 55 °C.

Determination of degree substitution (DS)

The method of measurement described in the literature was used to calculate the DS of the prepared CMC by Sophonputtanaphoca, et al., 2019. CMC (1.0 g) was stirred in 95% ethanol (250 mL) for 5 minutes before 5 mL of 2 M nitric acid was added under continuous stirring for 10 minutes at room temperature. The mixture was heated for 30 minutes until boiling and then left to settle. The acid and salts contained in the mixture were removed by filtration and washing with hot 95% ethanol (100 mL) and absolute methanol, followed by drying at 60 °C for 3 hours. The sample was dried for 3 hours at 90 °C in an oven and then cooled to room temperature.

CMC (0.5 g) was mixed with 100 mL of distilled water and stirred. Subsequently, 25 mL of 0.3 M NaOH was added to the mixture and boiled for 20 min. The solution was titrated with 0.3 M HCl until the phenolphthalein indicator turned from purple to colorless. The determination of the DS was conducted using Equation (1) and (2), where DS is the degree of substitution; % CM is the carboxymethyl content; V_0 is the volume of blank titration (mL); V_n is the volume of sample titration (mL); M is the molarity of HCl; and m is the mass of the sample (grams).

$$\%CM = \frac{[V_0 - V_n] \times M \times 0.059 \times 100}{m} \quad (1)$$

$$DS = \frac{162\% \times \%CM}{[5900 - (58 \times \%CM)]} \quad (2)$$

Determination of viscosity

CMC (3 g) was dissolved in 25 mL of water (4% w/v) and stirred for 10 minutes

at 45 °C to generate the sample solution. Viscosity analysis was done in two stages. The speed was set for 10 seconds at 960

rpm in the first step. The temperature was varied to 20, 30, 40, 50, and 60 °C for five minutes in the second step. Intervals running at 160 rpm. Every measurement was done three times (Sophonputtanaphoca et al., 2019).

Fourier Transform Infrared Spectroscopy (FTIR) measurement

The FT-IR spectra were recorded on an FTIR (8400S Shimadzu) using a KBr disk containing 1% finely ground samples over the wavenumbers of 4000-400 cm^{-1} at a resolution of 4 cm^{-1} .

X-ray Diffraction (XRD) measurement

Diffraction diagrams of samples were recorded between 5° and 60° using an XRD (Philips Xpert MPD, Cu K α , λ = 1,5406 Å). The samples were placed on the pin tube holder and analyzed over a short range of 5°–60° with a step size of 0.017° and using Cu K α radiation.

Scanning Electron Microscopy (SEM) measurement

The surface characteristics of cellulose were determined using scanning electron microscopy (SEM) (SEM, Zeiss-Evo Ma10). SEM was used to examine the membranes in low vacuum mode. The samples were sputtered with gold for approximately 120 minutes before analysis to ensure that there was no charge on the surface.

Results and Discussion

Analysis of degree substitution (DS)

The carboxymethylation reaction of cellulose isolated from EFB was investigated. Carboxymethylation is a chemical modification process used to produce carboxymethyl cellulose (CMC) by introducing carboxymethyl groups into cellulose molecules. This process enhances the solubility, chemical reactivity, and functional versatility of cellulose, making it suitable for a wide range of industrial applications (Mulyatno et al., 2017). The carboxymethylation of cellulose extracted from EFB was studied, focusing on the degree of substitution

(DS), a critical factor for the industrial applications of CMC.

The DS value is technically defined as the average number of reactive groups replaced by other active molecules along the polymer chain. In the synthesis of CMC from cellulose, the DS value is determined by the number of carboxymethyl substituent groups attached to each anhydroglucose unit. The reaction was conducted using varying NaOH concentrations ranging from 20% to 60% (w/w), while maintaining all other parameters constant. As shown in Figure 1, the DS value increased considerably when the concentration of NaOH increased from 20% to 50% and then decreased at 60%. Pushpamalar et al., (2006) reported that a higher concentration of alkali is undesirable because excess sodium hydroxide can react with sodium monochloroacetate, leading to the formation of sodium glycolate. The synthesis of CMC from wheat straw cellulose showed an increase in DS value as the NaOH concentration increased from 5% to 20% and a decrease at 25% (Li et al., 2019).

The concentration of NaOH plays a critical role in determining the degree of substitution (DS) during the synthesis of carboxymethyl cellulose (CMC). NaOH serves as a catalyst and a swelling agent, facilitating the carboxymethylation process by promoting the deprotonation of cellulose hydroxyl groups and enhancing the accessibility of reactive sites (Rahman et al., 2021). At lower NaOH concentrations, the limited availability of hydroxide ions reduces the efficiency of the reaction, leading to a lower DS. Insufficient swelling of the cellulose structure further hinders the diffusion of reagents, resulting in poor substitution. As the NaOH concentration increases, the cellulose fibers swell more effectively, exposing additional hydroxyl groups for reaction and enhancing the DS.

However, excessively high concentrations of NaOH (beyond an

optimal range) can have adverse effects. Over-saturation of NaOH may lead to degradation of cellulose chains, reducing the molecular weight of the resulting CMC and potentially decreasing the DS. Additionally, excessive alkalinity may cause side reactions or waste of reagents, negatively impacting the overall process efficiency. The DS value obtained in this study is higher compared to commercial CMCs, which typically range from 0.7 to 1.2 (Rani et al., 2014). Additionally, the synthetic polymers used in commercially available CMCs are not favorable for long-term environmental sustainability. Using CMC derived from EFB could help reduce reliance on synthetic polymers.

Viscosity measurement

CMC is a polymeric material that forms a viscous solution in an aqueous medium due to its high water solubility. Measuring the viscosity of Carboxymethyl Cellulose (CMC) is a critical step in characterizing its flow behavior and determining its suitability for specific applications, such as thickening, stabilizing, or emulsifying. The viscosity of the medium is directly affected by the concentration of CMC, increasing as the concentration increases and decreasing as it decreases. Furthermore, the viscosity of CMC is influenced by factors related to its source, particle size, molecular weight, and DS, as well as synthesis conditions like NaOH concentration, reaction temperature, and pH (Rahman et al., 2021).

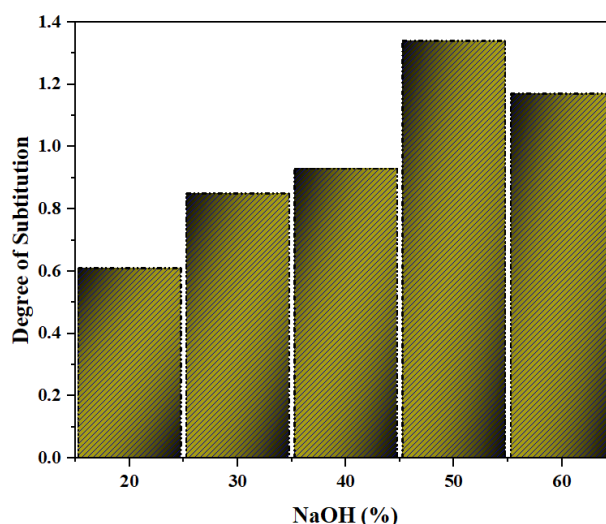


Figure 1. Effect of various NaOH concentrations in the alkalization reaction on the DS of CMC synthesized from cellulose of EFB

Figure 2 shows that the viscosity of CMC increased with the rising concentration of NaOH used during its synthesis. As the NaOH concentration exceeded 20%, the DS of CMC increased and then stabilized (Adinugraha et al., 2005). The increase in NaOH concentration during the synthesis of CMC can influence its viscosity through changes in the DS. An optimal NaOH concentration tends to enhance the

efficiency of the substitution reaction, resulting in a higher DS value. A higher DS indicates a greater substitution of hydroxyl groups on the cellulose, which can enhance CMC's ability to bind water and form a more viscous solution. However, if the NaOH concentration is too high, side reactions may occur, potentially reducing viscosity due to degradation of the polymer structure.

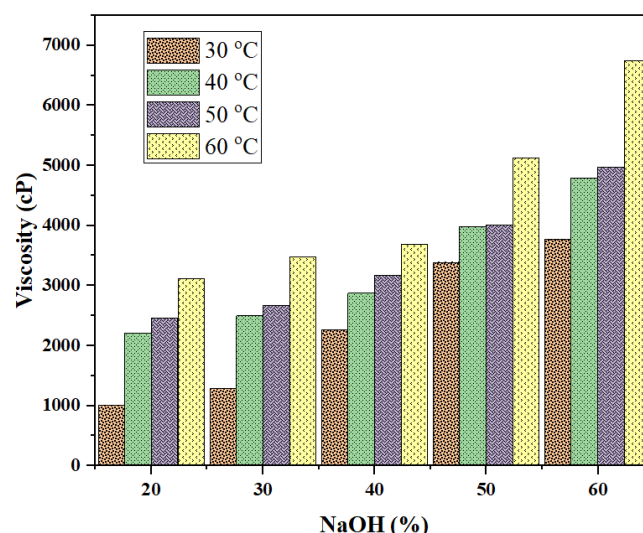


Figure 2. Effect of various NaOH concentrations on the viscosity of CMC synthesized from EFB at different temperatures

Furthermore, the impact of temperature on CMC viscosity was investigated as well. As the temperature increased, CMC viscosity dropped. Increasing the temperature of a CMC solution has a double impact of decreasing cohesive forces and increasing molecular exchange. Shear stress tends to increase with the latter impact whereas it usually decreases with the former (Haleem et al., 2014; Rahman et al., 2021b; Yeasmin & Mondal, 2015). As a result, liquids exhibit a decrease in viscosity as temperature increases.

FTIR characterization

As seen from the FTIR spectra of the EFB in Figure 3, band at 1541 cm^{-1} corresponds to C=C aromatic vibrations, indicating the presence of lignin (Rosa et al., 2012) while the absorption band at 1740 cm^{-1} signifies C=O stretching from hemicelluloses (Nuruddin et al., 2011). The absence of these peaks after the hydrolysis process confirms the successful removal of lignin and hemicellulose from the EFB. The broad absorption band observed between 3400

and 3500 cm^{-1} corresponds to the stretching vibrations of –OH groups, while the band at 2900 cm^{-1} is attributed to CH_2 groups (Jahan et al., 2011). The absorption band at 1319.31 cm^{-1} represents the C-O strain within the cellulose ring. Additionally, the band at 1163 cm^{-1} indicates C-O-C stretching, and the peak at 896 cm^{-1} is associated with the C-H rocking vibration of cellulose (anomeric vibration, characteristic of β -glucosides) (Fahma et al., 2010). The successful of carboxymethylation to produce CMC confirmed by the presence of a new and strong absorption band at 1606 cm^{-1} confirms the presence of the COO- group. The peaks at wave numbers of 1606 cm^{-1} (asymmetric) and 1424 cm^{-1} (symmetric) in the CMC are related to the C-O bond and the intensity of these peaks refers to carboxymethyl group substitution. The band around 1322 cm^{-1} is assigned to -OH bending vibration from CMC. Similar results have been reported for CMC in previous literature (Çelikçi et al., 2022; Hastuti et al., 2024; He et al., 2021)

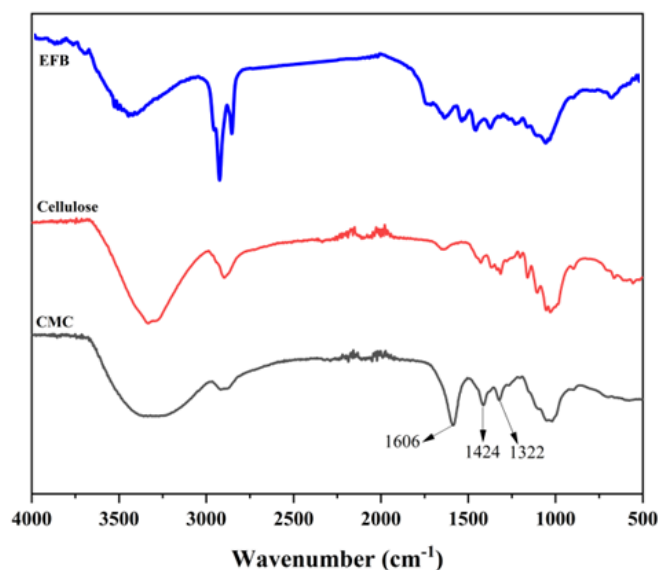


Figure 3. FTIR spectra of cellulose and CMC

XRD characterization

Figure 4 shows the XRD spectra of EFB, cellulose and CMC. The change in crystallinity and structure of EFB into cellulose, followed by the formation of CMC, was confirmed by XRD. The XRD spectra of EFB exhibit a peak around $2\theta = 22.6^\circ$, which is a typical feature of amorphous lignocellulosic structures. In contrast, the primary characteristic peaks of crystalline cellulose appear at $2\theta = 15.0^\circ$, 16.4° , 22.9° , and 34.3° , corresponding to the (110), (110), (200), and (004) lattice planes of cellulose type I, respectively (JCPDS-ICDD: 50-2241)

(Holilah et al., 2022; Kedang et al., 2022).

The highest intensity peak originates from the crystalline fraction of cellulose, while the amorphous portion contributes to the background noise. The synthesized CMC exhibited a disruption of the original cellulose's crystalline structure. The characteristic peaks of type I cellulose vanished and were replaced by those of type II cellulose. During the carboxymethylation process, the conversion of type I cellulose to type II cellulose led to a decrease in the crystallinity of CMC (Li et al., 2019).

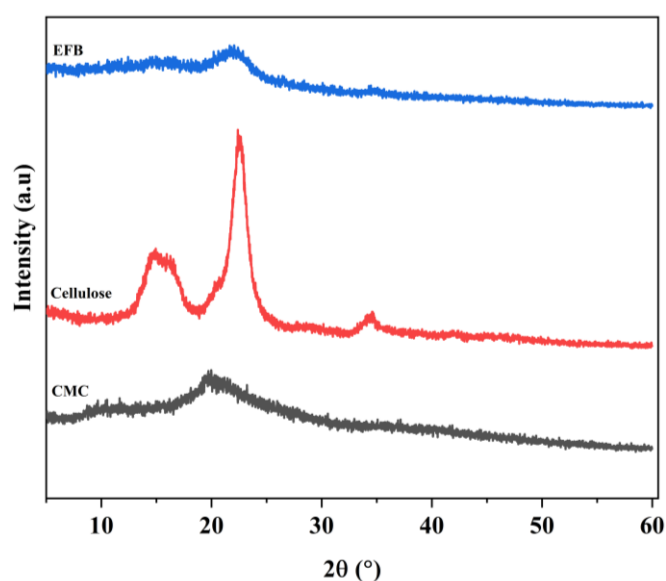


Figure 4. XRD Diffractogram of Cellulose and CMC

SEM characterization

Scanning Electron Microscopy (SEM) was used to evaluate the morphological evolution of the empty fruit bunches (EFB) during cellulose extraction. This analysis provides direct visual evidence of structural transformations that occur throughout the delignification, alkalization, and bleaching stages. SEM allows for the confirmation of lignin/hemicellulose removal and the successful isolation of cellulose microfibrils (Nafisah et al., 2022).

SEM images of untreated EFB (Figure 5a) show a compact and rigid surface. It is

still coated by lignin, hemicellulose, and other impurities indicated by the arrow. The fibers appear tightly bound and coated with a heterogeneous layer of surface impurities. No visible porosity is observed, and the structure appears mechanically robust. Figure 5b reveals major changes in fiber morphology after NaOH treatment. The surface appears fibrillated and more open, with visible pores, cracks, and microfibril exposure. Fibers are beginning to separate, and surface roughness increases significantly (Dimawarnita et al., 2023).

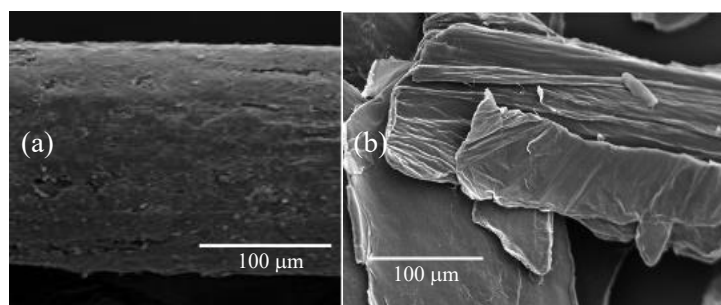


Figure 5. SEM Images of (a) EFB and (b) Cellulose

SEM analysis confirms the progressive removal of non-cellulosic components and the successful exposure of the cellulose structure in EFB fibers. The transformation from dense, lignin-rich surfaces to clean and porous cellulose fibrils support the effectiveness of the chemical treatment sequence employed. These structural changes also establish the suitability of the resulting cellulose for use as a precursor in biopolymer synthesis, including carboxymethyl cellulose (CMC).

Conclusions

The cellulose extracted from EFB was used to produce CMC after a reaction with MCA in basic medium. The performance of this reaction was evaluated by DS and viscosity of CMC. The results of this research showed that EFB could be effectively utilized as a raw material in a variety of NaOH concentrations to produce CMC. This demonstrates that the

primary parameter that correlates with the CMC characteristics is the NaOH content. During CMC synthesis, the DS of CMC increased as the NaOH concentration ranged from 20% to 50%, but it decreased at a NaOH concentration of 60%. However, converting the hemicellulose and lignin obtained into value-added products is expected to be crucial for the economic viability of CMC production using cellulose derived from EFB.

Author Contributions

DP and YIK designed the research study. DP, YIK, and MMK performed the research. LN and JP provide help and advice on analyzing and visualizing the data. DP and YIK wrote the manuscript. All authors contributed and agreed to the final version of the manuscript.

Conflict of Interest

The authors have declared that there is no conflict of interest.

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