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Fabrication of Carboxymethyl Cellulose (CMC) Derived from Themeda Gigantea Cellulose with Various Concentrations of Sodium Monochloroacetate

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Abstract: The purpose of this study was to determine the properties of Carboxymethyl Cellulose (CMC) obtained from pimping stems (Themeda gigantea). There has yet to be a report on utilizing Themeda gigantea as a source of CMC raw materials. CMC was produced by varying the amount of Sodium Monochloroacetate (SMCA) as a carboxymethylation material. This study consisted of five SMCA treatments, adding 5, 6, 7, 8, and 9 g, respectively. The test parameters in this study were the characterization of the cellulose components in pimping stems and CMC produced after the carboxymethylation process. The results of the analysis showed that pimping stems contained relatively high cellulose of 51.83%; thus, it had the potential to be developed as raw materials for CMC. The addition of SMCA significantly impacted the properties of the resulting CMC, except for water content and solubility. Adding 8 grams of SMCA produced the best results in this investigation, as determined by the observation parameters. In summary, the properties of CMC produced in this study are comparable to commercially available CMC and meet the Indonesian National Standard for Class II requirements. Further study should be conducted to improve the purity of CMC and eliminate the presence of NaCl to make CMC that meets class I standards.

Keywords: Carboxymethylation, Cellulose, Degree of Substitution, Lignocellulose, Viscosity

Introduction

Carboxymethyl Cellulose (CMC) is produced through etherification from cellulose as the source material. It has the following properties: Anionic nature, ranging in color from white to yellow, absence of odor and flavor, non-toxic nature, biodegradability, hygroscopic behavior, and non-toxicity (Rahman *et al*., 2021). In general, the uses of CMC are extensive, so this product has excellent economic potential. CMC can be applied in many fields, such as drug carriers, wound healing agents, corrosively prevention, binders in ceramics, composites in hydrogel layers, packaging composites, edible coatings on fresh fruit and vegetables for post-harvest purposes, and hydrocolloids for food products (Liyanapathiranage, 2023). Therefore, developing variations in raw cellulose materials is very interesting in producing CMC. In general, cellulose is obtained from biomass or bacterial cellulose. In this

research, a source of cellulose that has never been reported will be proposed, namely the Themeda gigantea.

Themeda gigantean (Indonesian local name: Pimping) is a prevalent undergrowth untamed plant in the tropical regions of Indonesia that is regarded as an annoyance or weed on agricultural land. This plant is a similar type of grass reed, a member of the grain tribe. Themeda gigantean is widespread in Indochina, the archipelago, and the Pacific. Stems are erect, sturdy, 150-400 cm high, with an average stem diameter of 0.5 inches. Stems are segmented and have pith. The reed segments are hairy or bald in the distal direction. The leaf midrib is keeled; the surface is glabrous and the edges are glabrous or hairy. The ligula (tongue frond) is a hairy membrane, cropped or blunt shape. The leaves gather below like a fan. Ribbon-shaped leaves, 30-100 cm long ×5-25 mm, stiff; the mother of the leaf veins is vast; the surface of cotton strands is rough on the adaxial side; edges of cotton strands are sharp. This plant generally grows wild and has not been utilized. Therefore, there is potential for the development of this plant as a basic material in the production of CMC. According to a study conducted (Susanto *et al*., 2018), pimping is classified as a lignocellulosic plant, comprising 18% lignin, 26% cellulose and 43% hemicellulose. The cellulose content in this particular plant satisfies the minimum criteria for CMC synthesis as a raw material, comprising approximately 27-50% of the total material content (Huang *et al*., 2017).

CMC is produced through the alkalization and carboxymethylation processes. Using NaOH to activate OH groups on cellulose molecules constitutes alkalization. The expansion of the cellulose structure induced by alkalization facilitates the diffusion of carboxymethylation reagents within it (Nisa and Putri, 2014). Following this, the carboxymethylation process ensues, which involves the attachment of a carboxylic group to the cellulose structure via etherification. The carboxymethylation process involves the utilization of sodium monochloroacetate (salt) or monochloroacetic acid as the reagent. This involves the substitution of the -OH group on cellulose with $ClCH_2COONa$ (Wahyuni *et al*., 2019).

The optimization of cellulose utilization from pimping as a cellulose derivative product has yet to occur. The application of this plant was restricted to practical purposes such as constructing avian cages, fishing rods, and tools to support fractured bones; it was also utilized as a material for religious rituals. The carboxymethylation process is one method by which the potential cellulose content of pimping plants can be utilized as source material for the preparation of CMC during the synthesis of CMC.

In order to develop CMC of pimping stem that meets the criteria of the Indonesian National Standard for food additives (food grade), this study investigated the possibility of incorporating Sodium Monochloroacetate (SMCA) as a reagent for the carboxymethylation process. It is anticipated that this research will yield high-quality CMC for pimping stems, thereby making a scholarly contribution to the understanding of how economically valuable untamed plants can be utilized.

Materials and Methods

The research was conducted at the Laboratory of Biochemistry of Agricultural Products and Food Nutrition, Laboratory of Engineering Technology and Process of Agricultural Products, Department of Food & Agricultural Products Technology, Faculty of Agricultural Technology and Research Laboratory, Faculty of Pharmacy, Universitas Andalas, Padang. The study was carried out between July 2023 and September 2024.

Materials

The primary material utilized in this research was pimping stem, which is readily available in the vicinity of the Andalas University campus. Other materials used were distilled water, ethanol 95%, sodium hydroxide, isopropyl alcohol, sodium monochloroacetate (SMCA), glacial acetic acid, sulphuric acid, sodium chlorite, hydrogen peroxide, hydrochloric acid, etc. All chemicals used were pro-analysis grade.

Methodology

The study for the production of CMC from the cellulose of the pimping stem consisted of three stages. First, the cellulose from the pimping stem was determined and extracted. Secondly, the extracted cellulose was purified and processed to form CMC by chemical reaction with the treatment of concentration of SMCA. Lastly, the produced CMC was then characterized and compared to the properties of commercialized ones. The experiments were conducted in triplicates. For data calculation, the statistical analysis with an ANOVA F test was carried out at the 5% probability level.

Preparation of Pimping Stem Powder

The pimping stems were sorted and dried in the sun until they were brownish-yellow in color. Subsequently, the pimping stems were chopped into thin pieces with a thickness of approximately 1 mm. The chopped stems were then placed in an oven set at 60°C and dried for 3 h until their weight remained constant. Then, proceed with 50 mesh sieving and pulverizing with a blender (Rachtanapun *et al*., 2021).

Isolation of Pimping Stem Cellulose (Removal of Lignin)

Pimping stem powder was soaked in 10% NaOH (1:10 b/v). Soaking was carried out for 24 h. Then the

filtering process was carried out using a filter cloth. The obtained residue was then re-soaked for 24 h in 10% H_2O_2 (1:10 b/v). Following filtration, the residue underwent a series of boiling distilled water washes until the H_2O_2 odor was eliminated and the pH of the residue returned to neutral. The residue was then oven-dried at 60°C until its weight remained constant (Nasri *et al*., 2013).

Preparation of CMC from Pimping Stem

During the alkalization procedure, 5 g of pimping stem cellulose by dry weight was introduced into a 250 mL Erlenmeyer. Subsequently, 100 mL of isopropanol was added to the mixture while it was being stirred at 30°C (room temperature) with a hotplate agitator operating at 100 rpm. Then, while continuing to agitate, 20 mL of 15% NaOH solution was added drop by drop for 1 h. Following the conclusion of the alkalization procedure, the carboxymethylation process was further initiated through the addition of SMCA in accordance with the treatment variation as shown in Table (1).

Using a hotplate stirrer, the carboxymethylation process was conducted for 3 h at 60°C. After cooling and neutralizing the carboxymethylated mixture to room temperature (pH 7), 98% glacial acetic acid was used. Following that, it was filtered and subjected to a 24 h purification procedure using up to 100 mL of methanol. After that, the residue was filtered once again and dried in an oven until the weight remained constant at 60°C. After that, the dehydrated CMC was ground up and kept in a confined space (Nasri *et al*., 2013).

Analysis of Extracted Cellulose FROM Pimping Stem

Extractive Substance Content

10 g of powdered pimping stem was placed into a filter paper container with a pre-determined weight. Subsequently, the sample was inserted into an extraction tube and then immersed in a solvent mixture consisting of ethanol and benzene in a ratio of 1:2. The extraction procedure was conducted for a duration of 6-8 h, and upon its conclusion, the lead was eliminated. Subsequently, a 50 cc volume of ethanol was employed to cleanse the substance from benzene, followed by a drying process in an oven set at a temperature of 105°C for a duration of 2 h. Subsequently, the sample was placed into a desiccator and measured for weight (Małachowska *et al*., 2020):

$$\%Extractive = \frac{A-B}{A} X100\%$$

where, *A* (*g*) is the weight of the sample before extraction; *B* (g) is the weight of the sample after extraction.

Lignin Content

2 g of the extracted sample was combined with 25 mL of 72% H_2SO_4 in a 100 mL beaker. The mixture was then left undisturbed at ambient temperature for a duration of 2 h, with intermittent stirring. Following dilution with 500 mL of distilled water, the mixture was brought to a simmer for 4 h and a half. Once the precipitate had cooled, it was filtered through filter paper and rinsed until acid-free with distilled water. At 105°C, the precipitate that formed on the filter paper was desiccated in an oven until its weight remained constant:

$$\%Lignin = \frac{A}{B} X100\%$$

Where *A* (g) is the weight of lignin after the oven; *B* (g) is the weight of the extracted sample.

Holocellulose Content

5 g of the extracted sample was placed in a 200 mL Erlenmeyer. Then, 160 mL of distillate water, 1.5 g $NaClO_2$, and 10 drops of CH_3COOH (acetic acid) were added to the sample. The Erlenmeyer containing the sample was covered with a small Erlenmeyer which was turned upside down and heated in a water bath for 4 h at 70-80°C and shaken occasionally and every 1 h 10 drops of acetic acid followed by 1.5 g $NaClO_2$. After completion, the sample was cooled in ice water and then filtered. The sample was washed with ice water and then with acetone. Next, the samples were dried in an oven at 40°C until constant weight (Małachowska *et al*., 2020):

$$\%Holocellulose = \frac{A}{B} X100\%$$

where, *A* (g) is the weight of holocellulose after oven; *B* (*g*) is the weight of the extracted sample.

Cellulose Content

2 g of holocellulose sample was weighed, then heated for 2 h in a 500 mL beaker containing 200 mL of 1.3% H_2SO_4 added over a water bath containing simmering water. The mixture was filtered and rinsed with 150 mL of distilled water until neutral after 2 h. The neutral sample was subsequently rinsed with ethanol. The sample was desiccated until constant weight in an oven preheated to 105°C (Małachowska *et al*., 2020).

Table 1: Formulation of the synthesis for CMC pimping stem

	Treatments				
Materials	A	B	C	D	E
Cellulose (g)	5	5	5	5	5
SMCA (g)	5	6	7	8	9
Isopropanol (mL)	100	100	100	100	100
NaOH 15% (mL)	20	20	20	20	20
Glacial acetic acid 98 % (mL)	pH 7	pH 7	pH 7	pH 7	pH 7
Methanol (mL)	100	100	100	100	100

$$\%Cellulose = \frac{A}{B} X100\%$$

where, *A* (g) is the weight of cellulose after oven; *B* (g) is the weight of the holocellulose sample.

Hemicellulose Content

The hemicellulose content is determined by subtracting the weight of holocellulose from the weight of cellulose:

$$\%Hemicellulose = A - B$$

where, *A* (g) is the weight of the holocellulose sample; *B* (*g*) is the weight of the cellulose sample.

Produced CMC Analysis

Chemical Properties of CMC

Moisture Content Analysis

3 g of sample was weighed and placed into an empty aluminum cup. The empty aluminum cup was first dried in an oven at 105°C for 30 min, then cooled in a desiccator for 15 min, and then weighed. The sample was then placed in the cup and put into the oven for 4 h. The cup containing the sample was retrieved and placed in a desiccator to cool for a duration of 15 min. Subsequently, proceed to measure its weight. The sample was then re-dried in the oven for 1 h until a consistent weight was achieved (Syukri *et al*., 2022, Anggraini *et al*.,2021):

$$\%Moisturecontent = \frac{A - B}{A} X100\%$$

where, *A* (g) is the initial weight of the sample; *B* (g) is the final weight of the sample (weight of the sample after oven weight of cup)

Degree of Substitution (DS) Analysis

1 g of CMC was put into a beaker and then mixed with 50 mL of 95% ethanol solution while stirring evenly. Then, 5 mL of 2 M HNO_3 was added and the mixture was stirred for 10 min at room temperature. Heat the mixture to boiling for 5 min on a hotplate stirrer, then stir again for 20 min and allow to settle. After that, the mixture was filtered and the residue was washed with 100 mL of 95% ethanol solution until the acid and salt disappeared. The precipitate was washed with methanol in a beaker and heated until the alcohol disappeared. The beaker containing the precipitate was dried in an oven at 90°C for 3 h. 0.5 g of dried residue was put into a 250 mL Erlenmeyer and 100 mL of distilled water was added while stirring. Then 25 mL of 0.5 M NaOH solution was added and heated for 20 min. In the hot state, the mixture was titrated with 0.3 M HCl solution using phenolphthalein as an indicator to observe the color change from dark pink to colorless (Haleem *et al*., 2014).

$$A = \frac{BC - DE}{F} \qquad DS = \frac{0{,}162xA}{1-(0{,}058xA)}$$

where, *A* = milli-equivalents of consumed acid per gram of specimens; *B* = volume of NaOH added; *C* = concentration of NaOH added; *D* = volume of consumed HCl; *E* = concentration of HCl used; *F* = specimen grams used; 162 are the molecular weight of the anhydrous glucose unit and 58 is the net increase in the anhydrous glucose unit for each substituted carboxymethyl group.

Purity of CMC and NaCl Content

In order to determine the purity of CMC, 1.5 g of CMC sample was dissolved in 100 mL of 80% methanol solution, while stirring it for 10 min. Then, the mixture was filtered and the residue was washed again with 100 mL of 80% methanol, then dried for 3 h or until constant weight was achieved. Calculation of CMC content was carried out using subsequent formulae:

$$\%PurityofCMC = W/Wo \times 100\%$$

where, *W*(*g*) is the weight of the dried sample after washing and *Wo*(*g*) is the weight of the sample before washing.

To determine the NaCl content, 2 g of CMC was added to 250 mL of 60% methanol and kept for 5 h. A total of 100 mL of the mixture was neutralized with 0.1N HNO_3. Then the titration process with $AgNO_3$ 0.1 N and K_2CrO_4 5% as an indicator. Calculation of NaCl was conducted using the following formula (Toğrul and Arslan, 2003):

$$\%NaCl\ content = 1{,}461x\frac{V}{m}$$

where, *V*(*mL*) is the amount of $AgNO_3$ and *m*(*g*) is the weight of the dried sample.

Physical Properties of CMC

Viscosity of CMC 1%

A beaker was filled with a 1 g dried weight sample of CMC, followed by the addition of 100 mL of distilled water. Then, the mixture was agitated over 60 degrees Celsius until dissolved. Following 3 min of measuring the sample's viscosity at 60 rpm with a viscometer, a scale reading was obtained. The equation for determining the viscosity value was as follows (Salimi *et al*., 2021):

$$Viscosity(cPs) = scale(dialreading)x\ correction\ factor$$

pH of CMC 1%

0.5 g dried weight of CMC was put into a beaker. 50 mL of distilled water was then added and the mixture was heated to a temperature of 70°C while stirring until dissolved. The pH meter was then put into solution (Zani'ah, 2020).

CMC Solubility Test

0.1 g of sample was put into a test tube. Then 10 mL of solvent (water, 96% ethanol, ethyl acetate, and n-hexane) was added and homogenized. Determine whether the sample is soluble in the solvent employed (Safitri *et al*., 2017).

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

15 milligrams of samples (consisting of cellulose from the stem of the pimping plant, CMC from the stem of the pimping plant, and commercial CMC) were prepared and crushed with 300 milligrams of high-purity KBr on a mortar until a uniform mixture was obtained. Subsequently, the concoction was transferred into a KBr Die. A force of 10 × 1,000 kg, or 10 tons, was exerted to produce the pellets. Subsequently, the sample was subjected to FTIR apparatus (The Nicolet iS10) analysis within the wave number range of 4000-400 cm^{-1} (Safitri *et al*., 2017).

Results and Discussion

Raw Material Characteristics

Table (2) shows the characteristics of the pimping plant. The cellulose content of the pimping plant has been known to be quite large. This data is similar to a study by Susanto *et al.* (2018). The high cellulose content has convinced the potential of pimping as a raw material for producing cellulose, where the cellulose can be further processed into more valuable products such as CMC. However, like other lignocellulose plants, lignin content will be a problem in this pimping plant; the lignin content is also categorized as quite high; therefore, for further processing of this pimping plant, lignin removal is essential. Lignin can inhibit the CMC formation reaction (Yang *et al*., 2023). The lignin removal process is carried out by alkaline hydrolysis, where cellulose that has been separated from lignin was obtained in this study.

Organoleptic and Basic Characteristics of CMC

Observations of CMC products were carried out, starting with physical and chemical observations. Physical observations were carried out by observing the organoleptic CMC crystals produced. Figure (1) shows the physical appearance of the resulting CMC. Visually, the CMC produced was similar to commercial CMC found on the market, although there is a slight difference in the color of the CMC crystals produced. This finding may be due to the difference in SMCA concentration used. Therefore, a chemical assessment must be carried out to determine the characteristics of the CMC produced based on the SMCA treatment.

Table 2: Chemical component analysis of pimping stem

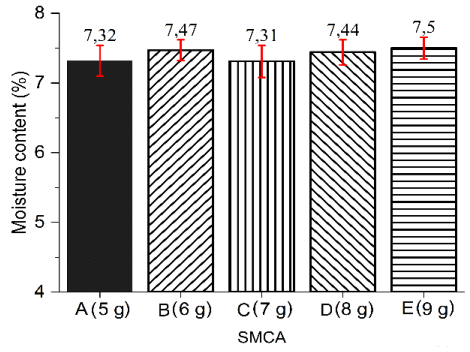
Component analysis	Means ± SD
Extractive substances (%)	7,16±0,18
Lignin content (%)	23,72±0,68
Holocellulose content (%)	76,35±0,28
Cellulose content (%)	51,83±1,01
Hemicellulose content (%)	24,52±0,98

Fig. 1: CMC from pimping stem in various SMCA concentration treatments

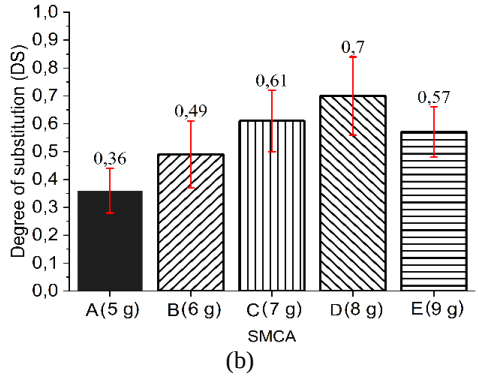
Figure (2) shows the chemical characteristics of the CMC produced in the form of water content, degree of substitution, purity, and NaCl content. These parameters are basic parameters for seeing the characteristics of a CMC product. SMCA treatment does not significantly affect the water content of each CMC produced. Water content is generally related to the ability of the material to bind to water and drying conditions (Syukri *et al*., 2019; Ievinsh, 2023). In the process of making this CMC, neither of these things affected the water content of the CMC produced. In terms of the Degree of Substitution (DS), it is known that the CMC treatment provides different DS values. The degree of substitution is a value that indicates the process of substituting hydroxyl groups in cellulose to produce CMC. Good CMC products have substitution values close to 1 (Wahyuni *et al*., 2019). In the results of this study, it is known that treatment D produces the most considerable DS value compared to other treatments. The advantages of SCMA have made the DS value smaller. Many factors affect this DS value besides the SMCA concentration. The substitution process is a reaction that influences temperature factors, NaOH concentration, and SMCA concentration (Nurfajriani *et al*., 2020).

Furthermore, it was also known that the purity value of CMC was directly proportional to the DS value discussed previously. Treatment D has produced CMC with the highest purity level, namely 95.96%. The best CMC purity value was undoubtedly close to 100%. Therefore, according to the conditions that affect the DS

value, the temperature factor and NaOH concentration need to be studied further in future studies to produce CMC from lead rods with higher purity. Furthermore, observing the NaCl content, it has been known that with the increase in SMCA used to produce CMC, the NaCl content also increases. The NaCl content affects the character of the resulting CMC. NaCl can generally affect the function and purity of the resulting CMC. The formation of NaCl was due to the reaction between sodium monochloroacetate and alkali cellulose (Siqueira *et al*., 2015); this theory shows that excess SMCA or NaOH can cause a lot of NaCl formation. Once again, this provides information that optimizing factors other than SMCA was essential in producing CMC. Because this study has focused on treating SMCA, the data can guide other researchers in optimizing other actions, such as temperature and NaOH concentration.



(a)



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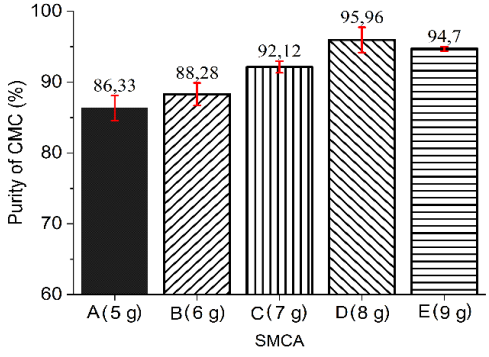

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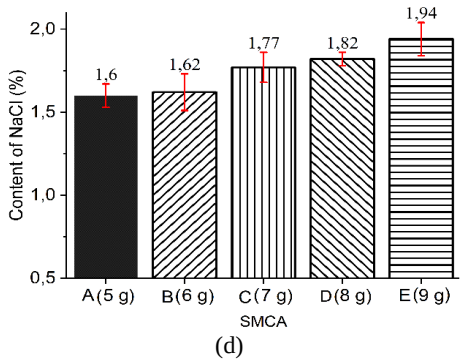

(d)

Fig. 2: The chemical properties of CMC pimping stem; (a) Moisture content; (b) DS; (c) Purity of CMC and; (d) NaCl content

The relationship between the purity value and NaCl content in the CMC pimping stem was illustrated in Fig. (3). The increase in NaCl content generated was directly proportional to the addition of SMCA to the CMC pimping stem. The treatment involving CMC pimping stem (D) with the addition of SMCA (8 g) yielded the highest purity at 95.96%. While the purity value decreased as SMCA was added in excess of 8 g, the resultant NaCl content remained unchanged. This finding suggested that the predominant by-product formation reaction in the synthesis of CMC pimping stem was the production of NaCl and sodium glycolate, which resulted from the reaction between NaOH and SMCA upon the addition of 9 g of SMCA (treatment E).

Physical Observation of CMC Pimping Stem

In addition, the physical characteristics of CMC are observed by testing its solubility. This solubility test will correlate with the purity of the CMC produced (Rahman *et al*., 2021). Pure CMC will readily dissolve in water or polar solvents. If impure CMC is formed, the resulting CMC will not dissolve in water and other polar solvents. Table (3) shows the solubility values of the resulting CMC. From all treatments, it is known that all CMC produced is soluble in water. This data shows that the CMC formation process has been successfully carried out with several variations of SMCA. Although the solubility test was similar for all treatments, other tests need to be carried out because, in terms of application, solubility in water alone was not enough to determine the quality of the resulting CMC.

Figure (4) illustrates the other physical characteristics of the CMC produced in this study. The physical characteristics selected in this study are viscosity and pH. These two things were chosen because the initial purpose of making CMC from cellulose pimping stems was for applications in the food sector, such as thickeners (Suryanti *et al*., 2023). In the manufacture of food products, they use CMC as a thickener, which leads to viscosity and pH values becoming benchmarks for the functional value of the CMC. From Fig. (4), it can be seen

that the viscosity value has the same trend as the purity value of CMC. The higher the purity of CMC, the thicker the solution produced by the CMC. This theory certainly fits the function of the CMC produced.

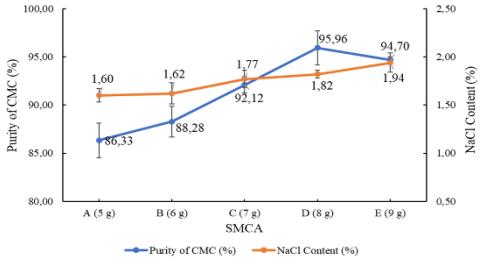

Fig. 3: The effect of SMVA addition on purity and NAVL content

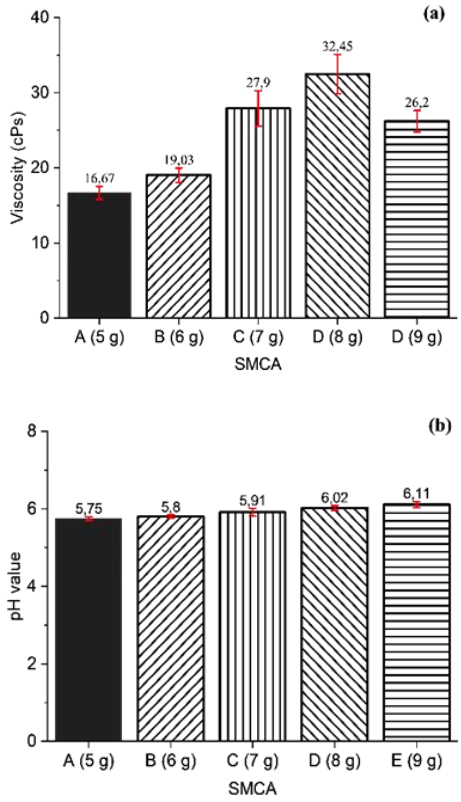

Fig. 4: Physical properties of CMC pimping stem (a) viscosity and (b) pH value

Table 3: Solubility of CMC pimping stem in various solvents

	Solubility			
Treatments	Water	Ethanol 96%	Ethyl acetate	n-hexane
A (SMCA 5 g)	+	-	-	-
B (SMCA 6 g)	+	-	-	-
C (SMCA 7 g)	+	-	-	-
D (SMCA 8 g)	+	-	-	-
E (SMCA 9 g)	+	-	-	-

Where: Sign (+) = CMC can dissolve completely. Sign (-) = CMC cannot dissolve and there was still sediment after stirring.

In contrast to the pH value, all CMC produced from cellulose pimping stems with various SMCA treatments, in their applications, do not give a different pH effect. This finding means that in the resulting CMC, it is very likely that no more excess NaOH can make the pH of the solution alkaline. Although the purity of the CMC produced varies, in its application, the CMC will not affect the pH of the solution, but the viscosity will be affected so that, of course, the purity of the CMC becomes the leading benchmark in the application of CMC in the future.

Fourier Transform Infrared Spectroscopy (FTIR) Analysis

FTIR analysis is an analytical technique used to identify and analyze organic components, chemical bonds, and functional groups in a sample (Suryanto *et al*., 2023; Syukri *et al*., 2024). An FTIR study was performed on the cellulose pimping stem to determine any alterations in the molecular structure following CMC production. This FTIR data will strengthen the explanation related to the characteristics of CMC produced from the need for D because the FTIR results will show a comparison of the spectrum between cellulose, CMC from treatment D, and CMC available in the commercial market. Although this research was conducted on a lab scale, comparing the IR spectrum with commercial CMC will provide higher confidence for product development on a larger scale in the future. The spectra of FTIR cellulose from the stem, commercial CMC, and CMC from the stem D can be observed in Fig. (5).

Three FTIR spectra have been displayed based on the FTIR spectrum shown in Fig. (5). The spectrum of cellulose from the pimping stem and the spectrum of CMC produced by adding 8 g of SMCA (D treatment) and commercial CMC have been compared. The three spectra have the same absorption in the 2800-3400 nm region (Dassanayake *et al*., 2019; Hospodarova *et al*., 2018; Kunusa *et al*., 2018; Rosa *et al*., 2010). This data was because cellulose and CMC both have hydroxyl groups. The fundamental difference between cellulose and CMC is seen in the absorption between 1604-1596 cm^1, which corresponds to the antisymmetric vibration (-COO-), a characteristic of carboxyl compounds (Biswal and Singh, 2004; Suriyatem *et al*., 2020; Zheng *et al*., 2024). Commercial CMC and the production results in this study have different absorption in the 1700-1750 cm^1 region, which is a characteristic of the carbonyl group (C = O) absorption peak in acetic acid (Cukrowicz *et al*., 2020; Dachriyanus, 2004). The pH of the pimping stem CMC analyzed physically is known to be acidic. In addition, it has a slightly pungent aroma reminiscent of an acidic substance. Moreover, the vibration of the -COONa functional group, or carboxyl compound in the form of sodium salt, occurs at a frequency of 1436.56 cm^1 in CMC derived from pimping stem (Hidayat *et al*., 2018).

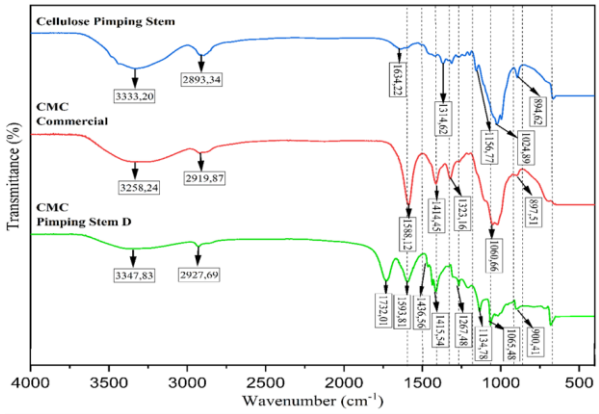

Fig. 5: FTIR spectra of cellulose pimping stem, CMC commercial and CMC pimping stem D

Mondal *et al*. (2015) observed carboxyl functional groups as salts, with an absorption peak at 1423.4 cm^1, during CMC Cavendish banana stem synthesis using sodium monochloroacetate. The C-O-C asymmetric stretching vibration, which has an absorption range of 1165-1150 cm^1, was observed at a specific frequency of 1134.78 cm^1 in CMC pimping stem D (Rosa *et al*., 2010). This functional group was also detected in the cellulose-rich stem, exhibiting an absorption peak at 1156.77 cm^1. This data suggested that just a small portion of the CMC pimping stem D polymer chain remained as cellulose. The solubility test of CMC pimping stem D revealed the presence of a minor fraction of fine cellulose fibers upon its dissolution in a water solvent.

Comparison with CMC from other Sources

The utilization of pimping stems as a source of cellulose to become CMC has been developed in this study. CMC that produced by reacting cellulose with 8 g of SMCA was selected as the optimum product at this time (treatment D). The results of this study provide information that pimping stems which are wild plants and have not been utilized until now have economic potential if processed into cellulose and CMC. Of course, further economic studies need to be carried out. In addition, using a measured amount of SMCA to produce CMC can be reference data for other researchers to optimize the process to produce better quality CMC. These are the things that make this study novel.

Moreover, CMC has been widely produced using cellulose from natural materials. Two types of cellulose are used, natural cellulose, such as cellulose pimping, and cellulose produced by bacteria, also known as bacterial cellulose. In the end, CMC produced from these two types of cellulose will still be influenced by the three factors mentioned earlier, namely SMCA concentration, NaOH concentration, and carboxymethylation reaction temperature. Each factor will differ depending on the raw material (Pushpamalar, 2006; Wijayani *et al*., 2010; Zani'ah, 2020; Ndruru *et al*., 2024; Mulyatno *et al*., 2017). Therefore, for new raw materials such as pimpin, a study to find optimal conditions for converting cellulose into CMC was essential. This study has provided information on the concentration of SMCA that can be used further; only two more factors need to be optimized. This research needs to be considered in the future.

Conclusion

The findings indicated that the inclusion of SMCA during the production of CMC pimping stem had an impact on various factors including the degree of substitution (DS), purity of CMC and NaCl content, viscosity, and pH value. Nevertheless, it had no substantial impact on the moisture content and solubility of the generated CMC. The study determined that adding 8 grams of SMCA was the most effective treatment for synthesizing CMC pimping stem. The success of producing CMC from pimping stems can enrich the cellulose source to produce cellulose. However, several things need to be further developed from this research. CMC from pimping from this research can be developed as an additive for non-food products such as plastic composite mixing materials, corrosive prevention materials, and products for other non-food purposes. To produce food-grade CMC pimping stem products, it is necessary to conduct additional research on optimizing cellulose purity and varying independent variables in the synthesis process. These variables include the alkalization process, reaction temperature, reaction time, and reaction medium. Additionally, it was necessary to employ various types or combinations of solvents and adjust the time of the purification process to eliminate by-products such as sodium chloride and sodium glycolate, ensuring the production of a purified CMC sample that meets food-grade criteria.

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Author's Contributions

Daimon Syukri: Conceptualization and wrote the manuscript.

Heru Suryanto: Funding acquisition.

Anni Faridah: Data curation.

Rini and Ira Desri Rahmi: Project administration.

Risa Meutia Fiana: Laboratory administration.
Cesar Welya Refdi: Formal analysis.
Skunda D: Experimental resources.
Afrizal Saputra and Annisa Rahmi Z.J: Sampling and laboratory analysis.

Ethics

The authors ensured that the material to be published has not been published previously

Data Availability

The datasets generated during and/or analyzed during the current study are available from the corresponding author upon reasonable request.

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