PAPER NAME	AUTHOR
5_Characterization of Carboxymethyl Cellulose.pdf	Suripto Dwi Yuwono

WORD COUNT	CHARACTER COUNT
4987 Words	25362 Characters

PAGE COUNT	FILE SIZE
11 Pages	548.8KB

SUBMISSION DATE	REPORT DATE
Apr 14, 2022 9:16 AM GMT+7	Apr 14, 2022 9:17 AM GMT+7

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Characterization of Carboxymethyl Cellulose (CMC) Synthesized from Microcellulose of Cassava Peel

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Abstract: In this study, carboxymethyl cellulose (CMC) was synthesized from the cellulose of cassava peel using alkalization and etherification reaction, and it was subsequently characterized with various techniques. Microcellulose was obtained by hydrolyzing α-cellulose from cassava peel using H_2SO_4 with concentrations of 45%, 47%, and 49%. The experimental results indicate that the varying concentrations of acid affect the particle size of the cellulose, with 49% H_2SO_4 solution producing a cellulose with particle size of around 0.45-2.42 µm and relative percentage of 11.3% according to PSA analysis. Other analyses conducted included determination of substitution degrees, DTG/DTA/TGA, FTIR, SEM, and XRD. The value of the substitution degree was determined at 0.27. TGA decomposition thermograms at a temperature of 150ºC-320ºC of 19.60 % indicate CMC compounds. DTA thermograms show that these CMC compounds have endothermic properties at 140ºC and exothermic properties at 260ºC. FTIR spectra show the presence of absorption band at wave number 1605 cm^{-1}, which is a characteristic absorption of carbonyl group bound to cellulose. The results of SEM analysis indicate that the CMC has a tenuous surface morphology, and the XRD diffractograms are marked by the presence of weak peak at 2θ = 20º, implying the existence of CMC as mostly amorphous.

Keywords: cassava peel, α-cellulose, carboxymethyl cellulose

1. Introduction

Food is a basic need of every human being. Currently, Indonesia's population is around 265 million [1], making the country as the fourth most populated in the world. Having such a large number, it is no surprise that the food industry is experiencing rapid development. In addition, modern lifestyles have put food products on demand. It has become a challenge that food products not only meet the quantity needed but also the quality standard desired by its consumers. In improving food quality, various additives are used in the production process, and one of the most popular is carboxymethyl cellulose (CMC) [2].

CMC is an ionic polysaccharide derived from cellulose, which has a linear long chain and is soluble in water [3]. Currently CMC has been considered as one of the most popular additives because it is inexpensive and easy to use in many applications [4–9]. One main reason for its popularity is the availability of its raw material, since cellulose is considered to be the most abundant organic compound found naturally. Cellulose is known to contribute about 33 % of plant material, and therefore, many types of agricultural waste are potential sources of cellulose. One of them is cassava peel, which can be found in large amounts in Indonesia, but its use is very limited due to its cyanide content. Cassava peel is an attractive raw material for CMC production because this agricultural residue is known to contain 37.9% cellulose, together with hemicellulose (37%) and lignin (7.5%) [10].

Degree of substitution (DS) and molecular weight are important usability parameters of CMC. The DS is the average number of hydroxyl groups found in the cellulose structure substituted by carboxymethyl and sodium carboxymethyl groups on C-2, C-3, and C-6.

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Increasing the DS will make it more compatible with other components such as salt or other solvents, which can affect its viscosity [11].

In this study, the cellulose obtained from cassava peel was converted into microcellulose in an attempt to increase the DS of CMC produced. This procedure was adopted from a study by Arup [12], who reported the synthesis of microcellulose in four steps: acid hydrolysis, centrifugation, ultra-sonication, and freeze-drying. In this research, acid hydrolysis was performed using sulfuric acid, with concentrations at 45, 47, and 49%. The microcellulose obtained was then used to synthesize CMC, by alkalization and etherification with NaOH concentration at 25%, in accordance with the procedure reported in the previous study [13]. In this regard, the objectives of this study were the isolation of α-cellulose from cassava peel by delignification method, determination of the concentration of H_2SO_4 which produced optimum conversion of α-cellulose into microcellulose, synthesis of CMC using the microcellulose, and characterization of CMC produced. The results of this study are expected to provide information about the potential of cassava peel as an alternative source of cellulose for CMC production, which could also alleviate environmental problems associated with this agricultural waste.

2. Materials and methods
2.1. Materials
The main materials used are cassava peel obtained from local industry in the City of Metro, Lampung, Indonesia which produces cassava chips and fermented cassava (locally known as tape). HNO_3, $NaNO_2$, NaOH, Na_2SO_3, NaOCl, H_2O_2, sodium monocloro acetate (NaMCA), $K_2Cr_2O_7$, ferroin indicator, ferrous ammonium sulfate [$(NH_4)_2)Fe(SO_4)_2.6H_2O$] and H_2SO_4 were obtained from Sigma-Aldrich, isopropyl alcohol, ethanol, universal indicator were obtained from J.T. Baker, all these chemicals are pro analysis reagents and used as received.

2.2. Instrumentations
The IR spectra were recorded on FTIR Prestige Shimadzu with KBr discs in the range of 4000-400 cm^{-1}. Microcellulose was characterized using the Fritsch PSA device, Analysette 22 series, to determine the particle-size distribution. A number of samples were fed into the water-filled chamber in the wet dispersion unit until the indicator displays the numbers 10-12 (green). SEM analysis was conducted using SEM ZEISS EVO MA 10. Before the analysis, the sample was prepared by coated gold metal so it was conductor. Next, the sample was inserted into the specimen. The intensity of the electron fired against the sample was regulated by using the condenser–objective lens. The electron reflection of the sample was captured by the detector, the image of which was displayed. The TGA/DTA was determined using EXSTAR TG/DTA7300. The measurements were performed using nitrogen with flow rate of 20 mL min^{-1}. The samples were heated at temperatures of 30 - 800°C with a heating speed of 20 °C min^{-1}. The measurement of crystallinity in microcellulose was carried out by powder X-Ray Diffractometry (XRD) using Phillips xpert, Netherlands based on the procedure in the literature [14].

2.3. Research procedure
2.3.1. Sample preparation
Cassava peels were obtained from a local food industry operating in the city metro, which products include cassava chips and fermented cassava (locally known as tape). After removing its epidermis layer, the peel was washed with clean water and then blended into smooth slurry. The slurry was washed with clean water until the filtrate was clear, and it is dried into powder by oven drying at 60°C.

2.3.2. Isolation of α-cellulose from cassava peel
Around 75 g of cassava peel powder was placed in a beaker glass, and then 1 L mixture of 3.5% HNO_3 and 10 mg $NaNO_2$ was added, the sample was heated on a hot plate at 90°C for 2 h. The sample was filtered, and the pulp was rinsed with distilled water until the filtrate became neutral. Then the pulp was refluxed with 750 mL solution containing 2% NaOH and 2% Na_2SO_3 at 50°C for 1 h, and then it

was filtered and washed until it became neutral. The pulp was bleached with 250 mL of 1.75% NaOCl solution at boiling temperature for 0.5 h and then filtered and washed until the filtrate reached neutral *p*H. The α-cellulose produced was purified by soaking in 500 mL of 17.5% NaOH solution at 80°C for 0.5 h. The sample was filtered and washed to remove the NaOH and then bleached with 10% H_2O_2 at room temperature for 1 h. Finally, the pulp of α-cellulose was dried at 60°C for 1 h

2.3.3. Determination of α-cellulose content

Determination of α-cellulose was conducted in accordance with the Indonesian National Standard Test Methods (SNI 0444: 2009) [15]. A sample of 1.5 ± 0.1 g t α-cellulose was transferred into a 300 mL tall cup, followed by the addition of 75 mL of 17.5% sodium hydroxide solution. The time when sodium hydroxide solution was added was recorded, and then the temperature of the mixture was adjusted to 25°C ± 0.2°C. The mixture was stirred carefully to avoid the formation of air bubbles, until the pulp was completely dispersed. After completing the stirring process, the stirrer was taken from the cup, and then washed with 25 mL of 17.5% sodium hydroxide solution, which was then added to the cup glass; the needed volume of the pulp suspension in the cup was 100 mL. The pulp suspension was stirred using the stirring rod and stored in a bath at 25°C ± 0.2°C. After 30 min from the first addition of sodium hydroxide solution, 100 mL of distilled water with temperature of 25°C ± 0.2°C was added into the pulp suspension and was stirred immediately using the stirring rod. The cup was placed in the bath for another 30 min so that the total extraction time is around 60 min ± 5 min.

The suspension was stirred again using stirring rod before it was transferred into the drain funnel. The first 10 mL to 20 mL of the filtrate was discharged, and the next 100 mL was collected in a dry clean flask, and 25 mL of this collected filtrate was transferred into a 250 mL flask; 10 mL of 0.5 N potassium dichromate solution was also added. An aliquot of 50 mL of concentrated sulfuric acid was carefully added, and the mixture was gently shaked. The mixture was heated to a temperature of 125°C–135°C for 15 min, and 50 mL of distilled water was added, and it was finally allowed to cool to ambient temperature. Into the solution, two to four drops of ferroin indicator were added and then titrated with 0.1 N ferrous ammonium sulfate solution until it becomes purple in color. At high pulp solubility (low α -cellulose content), with back titration of dichromate less than 10 mL, the volume of filtrate is reduced to 10 mL, and sulfuric acid to 30 mL is added. Blank titration was performed by replacing the pulp filtrate with 12.5 mL of 17.5% sodium hydroxide solution and 12.5 mL of distilled water. The α-cellulose content was calculated using the formula as in Equation 1:

$$X = 100 - \frac{6,85(V_1 - V_2) \times N \times 20}{A \times W} \qquad (1)$$

where

X = α-cellulose (%)
V_1 = the volume of blank titration (mL)
V_2 = the volume of pulp filtration titration (mL)
N = normality of ferrous ammonium sulfate solution
A = the volume of pulp filtrate analyzed (mL)
W = the mass of dry pulp used (gram)

2.3.4. Synthesis of microcellulose

Around 5 grams of sample was placed in a 1000 mL round flask, and 100 mL of H_2SO_4 with specified concentration (45, 47, and 49%) was added. The mixture was refluxed for 40 min at 50°C under stirring, and 100 mL of distilled water was added. The mixture was allowed to cool, and it was centrifuged at 9000 rpm for 15 min, before finally washing with distilling it. The colloidal suspension formed was subjected to ultrasonication for 5 min in ice bath and then freeze-dried.

2.3.5. Synthesis of CMC

The procedure for CMC preparation from α-cellulose was adopted from the method used by other researchers [13]. To commence the preparation, 5g of α-cellulose was put into a cup, followed by the addition of 150 mL isopropyl alcohol. The sample was stirred using a magnetic stirrer while 15 mL NaOH 25% was added. The sample was continuously stirred for another 1 h at room temperature. Into the solution, 6g of solid sodium monochloroacetic acid was added, the mixture was stirred for 1.5 h and then wrapped with aluminum foil and heated in an oven at 60°C for 3.5 h. After that, the sample was immersed into 100 mL of methanol for one night. The next day, the sample was neutralized with 90% acetic acid until the *p*H was neutral and filtered. The final product was washed with ethanol three times by immersing it into 50 mL ethanol for 10 min and then with 100 mL absolute ethanol. The produced CMC was dried in an oven at 60°C until a constant weight was obtained.

2.3.6. Determination of DS

The DS of the CMC prepared was determined in accordance with the method available in the literature [16]. Around 4g of CMC was dissolved in 75 mL of 95% ethanol, followed by the addition of 5 mL of 2 M nitric acid. The solution was put into boil while it is stirred for 10 min, and then the sample was washed with 20 mL 80% ethanol at 60°C five times and washed again with anhydrous methanol. Then the sample was filtered and dried in an oven at 105°C for 3.5 h and cooled in a desiccator for 30 min. Around 0.5 g of dry CMC was dissolved in 100 mL of distilled water and stirred, then 25 mL of 0.3 M NaOH solution was added, and the mixture was boiled for 15 min. The sample was added with PP indicator and titrated with 0.3 M HCl solution. The determination of the DS was conducted using Equation 2 and 3 [15]:

$$\%CM = \frac{[V_0 - V_n] \times M \times 0,059 \times 100}{m} \qquad (2)$$

$$DS = \frac{162 \times \%CM}{[5900 - (58 \times \%CM)]} \qquad (3)$$

where:

DS = degree of substitution
% CM = carboxymethyl content
V_0 = the volume of blank titration (mL)
Vn = volume of sample titration (mL)
M = molarity of HCl
m = mass of sample (gram)

3. Results and discussions

3.1. Determination of α-cellulose content

The principle of determining α-cellulose is to measure the amount of α-cellulose which is insoluble and does not decompose by NaOH solution [17, 18]. The α-cellulose content of the sample prepared in this study is 92.33%. The result obtained in this study is lower than the result reported by others [18], in which the sample prepared from empty bunches of palm oil was reported to contain 97.04% α-cellulose. Since the method applied in this study is practically similar to that applied for the work of Yuwono et al. [18], the differing results in the two studies are most likely due to different raw materials used.

3.2. PSA of microcellulose

Based on the results of the PSA (Figure 1), for every concentration variation of H_2SO_4 used, the particle sizes of microcellulose obtained also varied but not surprisingly the highest population obtained for every H_2SO_4 concentration was the same i.e. belong to particle size of 1.83 µm.
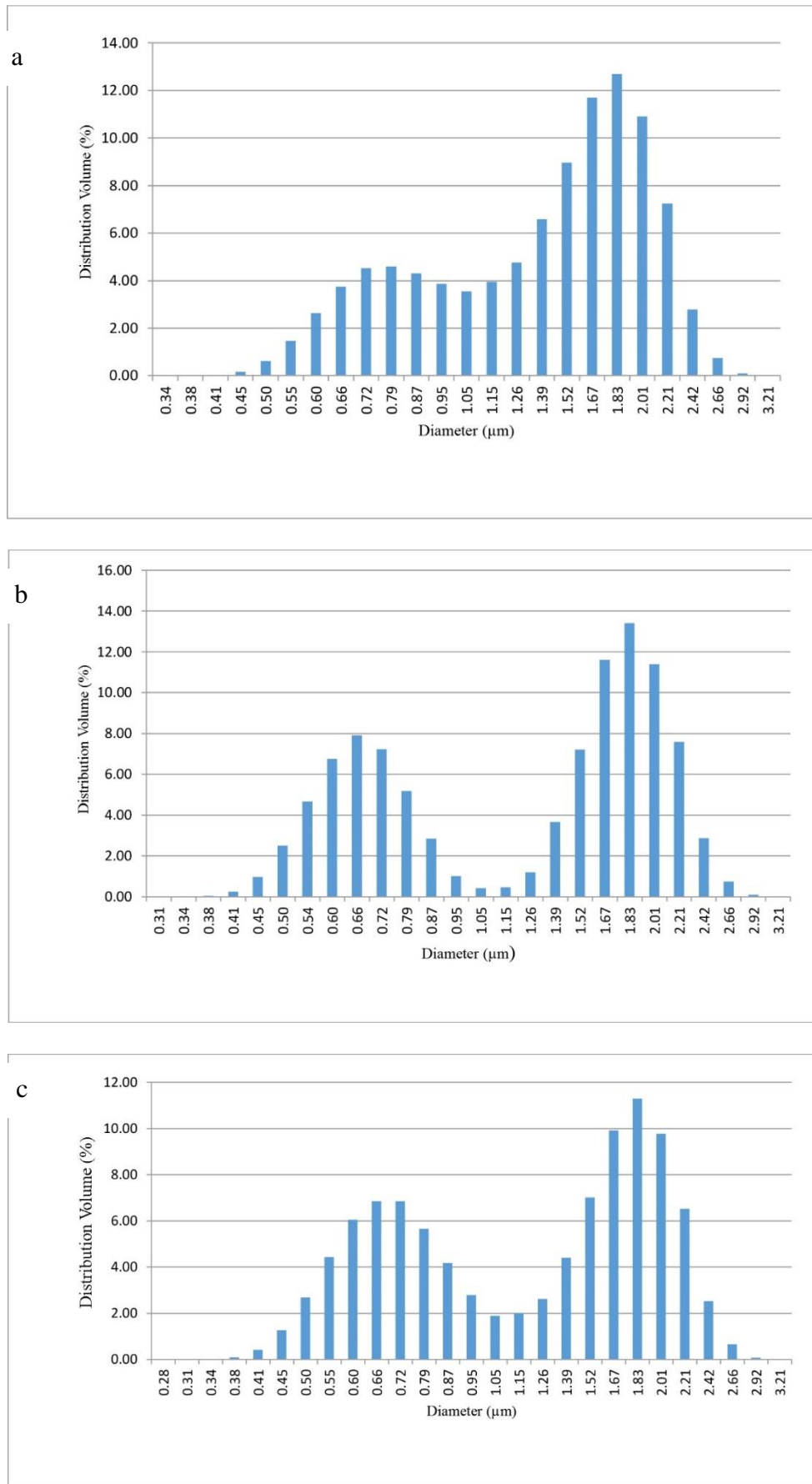
Figure 1. The result of PSA: (a) H_2SO_4 concentration 45%, (b) H_2SO_4 Concentration 47%, (c) H_2SO_4 concentration 49%

At a concentration of H_2SO_4 45%, the particle size of microcellulose was in the range 0.50-2.66 µm, with the highest population of 12.7% belong to particle size of 1.83 µm; in H_2SO_4 47%, the size was in the range of 0.45-2.42 µm and particle size of 1.83 µm was the highest population with total amount of 13.4%; and in H_2SO_4 49%, the particle size was in the range of 0.41-2.66 µm with the highest population of 11.3% belong to particle size of 1.83 µm. These data indicated that the cellulose obtained was like microparticles; thus microcellulose was obtained as the final product. The results obtained are in line with the result reported in other paper [19] which states that the microparticles are formed when the size is less than or equal to 100 µm.

3.3. Determination of CMC DS

The value of the substitution degree of the sample obtained in this study was 0.27 which indicated that the CMC product obtained was slightly soluble in water. The magnitude of the DS in this study is not much different from other works [11, 13] which is equal to 0.25. In this work, there are two factors affecting this DS value, namely, alkalization and carboxymethylation. In alkalization, the activation of OH groups on cellulose takes place, and upon this process causing the cellulose structure to expand; thus it makes the carboxymethylation reagent easy to diffuse in cellulose molecules. The carboxymethylation process is the most important step in the process of CMC synthesis [2, 11].

3.4. DTA/TGA CMC analysis

The derivative of DTA/TGA CMC has two phases of degradation (Figure 2). The first phase is the evaporation of volatile molecules, and the second phase is CMC degradation. The evaporation phase of volatile compounds occurred at 140°C of 0.368 mg/min and the degradation phase of CMC occurred at 260°C of 0.472 mg/min. This result is in accordance with the results obtained by Taghizadeh and Sabouri which explains that CMC degradation occurs at temperatures of 250°C–360°C [20].
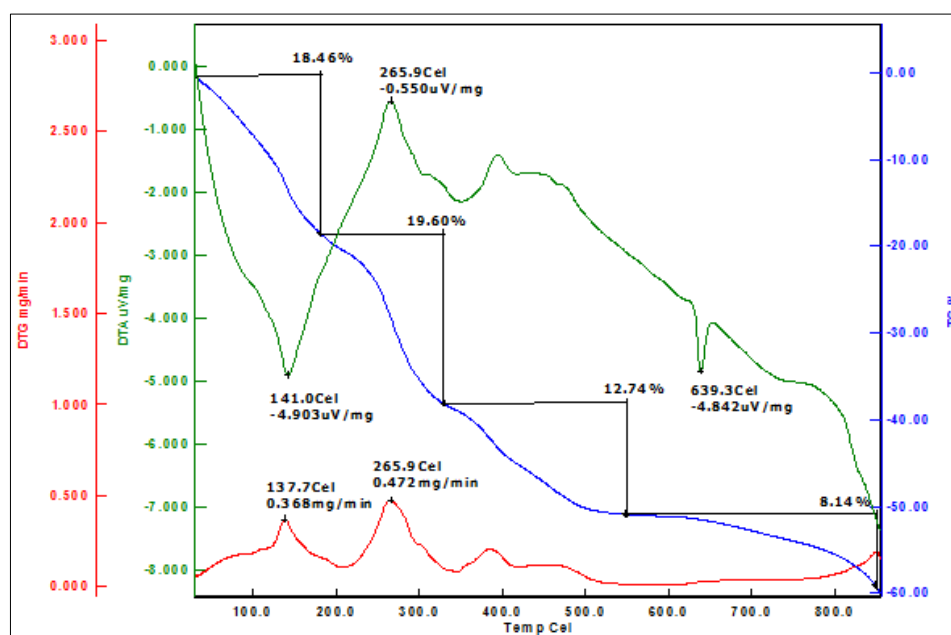

Figure 2. DTA/TGA thermogram of CMC

The DTA/TGA thermogram of CMC is shown in Figure 2. Based on this thermogram, the CMC has endothermic and exothermic properties. The endothermic properties were observed at 140°C to decrease in 4.9 µV due to water molecules. Exothermic properties were observed at 260°C at −0.55 µV due to CMC decomposition. Based on Figure 2, CMC has four decomposition phases. The first phase was water decomposition with mass reduction of 18.46% [21], the second phase was CMC decomposition with

mass reduction of 19.60% [22], and the third phase was decomposition to cross-link bonds in CMC [21]. In the third and fourth phases, there was a mass reduction of 12.74%.

The value of mass reduction in the study was smaller when compared to the reported ressult conducted by others [21, 22]. This is in accordance with the results of Sadeghi [20] which states that CMC compounds with more substitution will have higher thermal stability. As a result, many substituent CMC decomposition processes require higher temperatures than CMC with fewer substituents. Thus, at the same temperature, CMC decomposition which has many substituents results in a smaller mass reduction value than CMC which has little substituents.

3.5. FTIR analysis of carboxymethyl cellulose

The result of FTIR analysis (Figure 3) indicated that α-cellulose has a broad peak at 3418 cm^{-1} showing the present of hydroxy (–OH) band, a peak with moderate intensity at 2901 cm^{-1} was from the methylene (–CH_2–) stretching band, sharp peak with moderate intensity at 1628 cm^{-1} from stretching of the carbonyl group (C = O) from the glucose monomer, the peak with moderate shift at 1427 cm^{-1} from the band of the methylene group (–CH_2–), and strong band at 1057 cm^{-1} from the ether group absorption (COC).
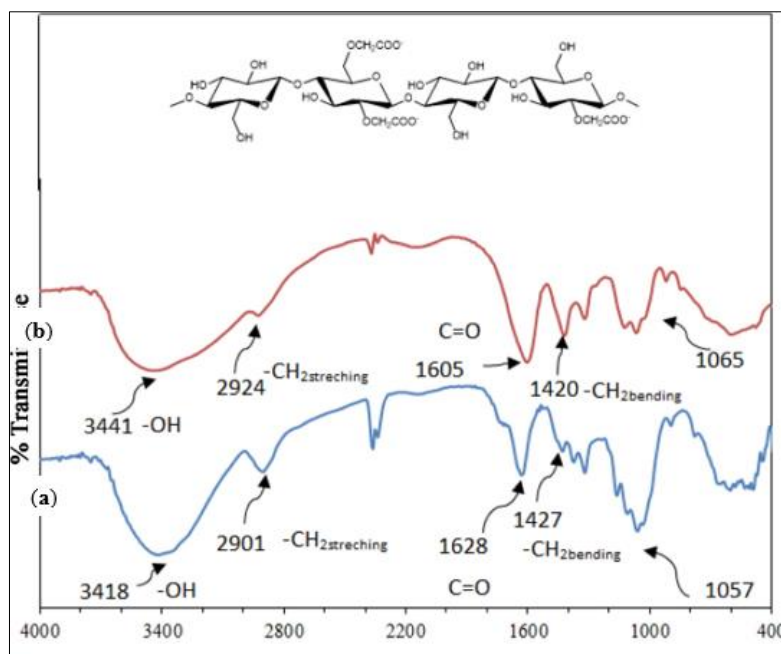

Figure 3. Infrared spectra of (a) α-celullose and (b) CMC

In comparing with IR spectrum of α-cellulose, the IR spectrum of CMC was slightly different. It has a broad peak at 3441 cm^{-1} from the hydroxy (-OH), the peak of moderate intensity was at 2924 cm^{-1} was from the vibration of the methylene group (-CH_2-), sharp peak with moderate intensity was at 1605 cm^{-1} of stretch absorption from carbonyl group (C = O) of glucose monomers, peak with moderate absorption at 1420 cm^{-1} from the band of the methylene (-CH_2-) group and strong band at 1065 cm^{-1} from ether group (COC).

Adel et al. [21] have reported that the results of FTIR analysis of CMC compounds for OH band were seen in wave numbers 3400 cm^{-1}, CH_2 vibrations were seen in wave numbers 2900 and 1424 cm^{-1}, and absorption of carbonyl groups seen in wave number 1720 cm^{-1}. The vibration of the hydroxyl group at CMC is at a larger wave number because of the stronger hydrogen bond due to the addition of the carboxylic acid group at CMC. The vibration of the carbonyl group in CMC appears at the lower wave numbers because the COO–H group in CMC is more easily dissociated. Dissociation of protons in carboxylic acid groups will cause possible electron resonance in carboxylic anions so that the electron density in the carbonyl group is lower.

Table 1 shows that almost all FTIR vibrations of functional groups from CMC compounds between the results of the study and reference are not of much difference, except for the absorption of carbonyl groups. The wave number of the carbonyl group from the results of this study is 1605 cm^{-1}, while the wave number of the reference carbonyl group is 1720 cm^{-1}. This is due to differences in the reagents used. This study uses NaMCA reagents, while research conducted by Adel et al. [21] uses MCA. The carbonyl compound of the CMC compound from this study binds to sodium metal. So the wave number of the carbonyl group results of this study is lower than the carbonyl group wave number that has been reported by Adel et al. [21]. On the other hand, the value of the carbonyl group wave number from the results of this study is closer to the value of the carbonyl group wave number from the research conducted by Eliza et al. [23] which uses the same reagent, which is 1589 cm^{-1}.

Table 1. Comparison of CMC FTIR absorption from research results with references [21]

No	Bond	FTIR vibration of	
		The product in this work (cm^{-1})	References (cm^{-1}) [21]
1	-OH	3441	3400
2	-CH$_2$- (*streching*)	2924	2900
	-CH$_2$- (*bending*)	1420	1425
3	-C=O	1605	1720

3.6. CMC SEM analysis

SEM analysis was used to determine the morphological changes of cellulose compounds to CMC. The results of SEM analysis of cellulose and SEM results of CMC compounds from the results of this study are presented in Figure 4. The results of SEM (Figure 4) indicated that there is a difference in density between cellulose and CMC compounds. CMC compounds have a morphology that is more tenuous than cellulose compounds. This is consistent with the study of Najafpour et al. [24] which explains that CMC compounds have been methylated due to carboxymethyl groups substituted on cellulose molecules so that the morphology of CMC is increasingly insignificant and the distance between molecules is greater.
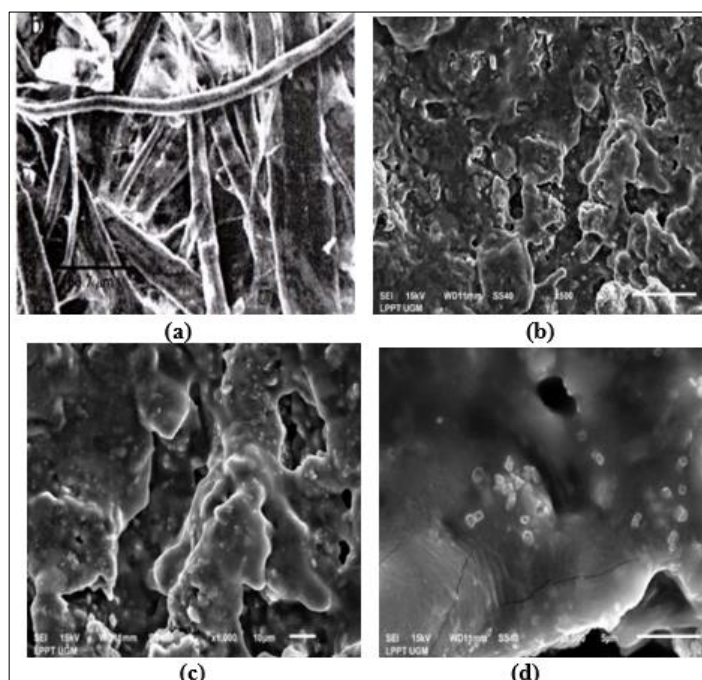

Figure 4. The results of SEM analysis (a) Cellulose,
(b) CMC 500x magnification, (c) CMC 1000x magnification, and
(d) CMC 5000x magnification

3.7. XRD analysis of carboxymethyl cellulose

Based on Figure 5, CMC products have three absorption peaks, namely, at 2θ 20°, 32°, and 45°. CMC compounds can be identified at absorption of 2θ 20°. This is in accordance with the results of the study of Huang et al. [25] which explained that the XRD CMC diffractogram was found at 2θ 20°. The CMC 'compound results of this study are not 100% pure; this is indicated by absorption at 2θ 32° and 45° which indicate there are still impurities in CMC compounds.
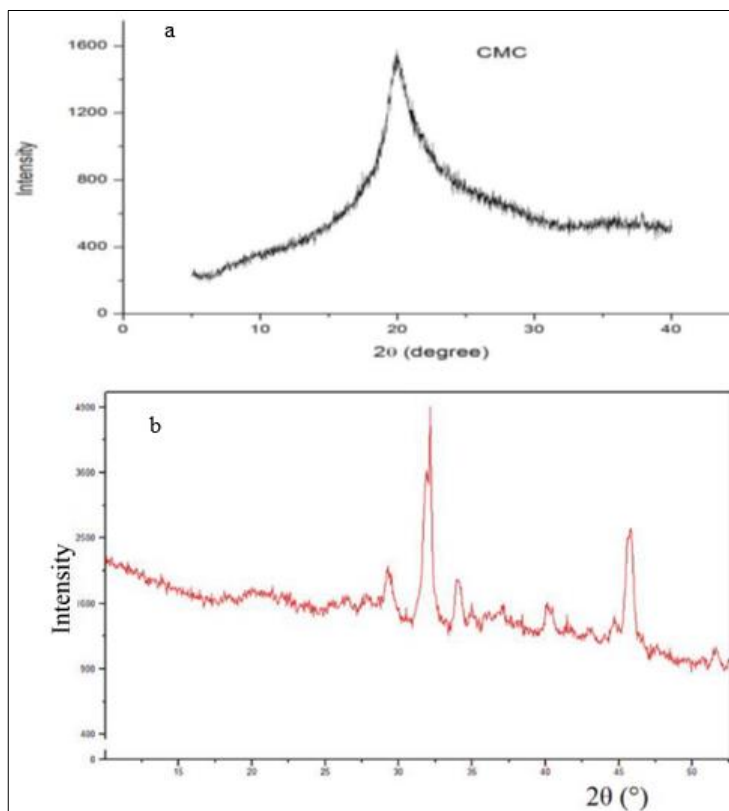

Figure 5. XRD difraktrogram of (a) CMC standard, and (b) CMC research results

4. Conclusions

Based on the results of our observations and experiments carried out, the following conclusions are obtained: (1) results of α-cellulose isolation from cassava peel using the delignification method obtained α-cellulose levels of 92.33%; (2) the optimum results for obtaining microcellulose were found in 49% H_2SO_4 concentrations, indicated by PSA analysis; and (3) in this study, CMC from cassava peel was successfully synthesized and characterized by FTIR (Fourier transform infrared), TGA (thermogravimetric analysis), SEM, and XRD.

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