Study on synthesis of carboxymethyl cellulose from pineapple leaf waste and its potential applications as a thickener

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Abstract:

In this study, cellulose and hemicellulose were successfully extracted from pineapple leaf waste at yields of 58.8 and 16.1% by dried weight, respectively. Carboxymethyl cellulose (CMC) was synthesised from pineapple leaf cellulose by an esterification process using sodium hydroxide (NaOH) and monochloroacetic acid (MCA) with isopropanol as the supporting medium. Preparation of CMC was investigated by varying three free factors, namely, NaOH concentration, MCA dose, and cellulose size. The carboxymethylation process was optimised to produce CMC with differing degrees of substitution (DS). The highest DS of CMC (0.86) was obtained with 15% (w/v) NaOH solution, 0.6 g of MCA/g cellulose, and 50 μm cellulose. The obtained CMC were characterised by FTIR spectroscopy, SEM images and XRD diffractions. Moreover, the thickening performance of obtained CMC was also determined. The influence of the CMC's molecular weight and degree of substitution on the viscosity of 1% (w/v) aqueous solution was tested. The experimental results suggest that the viscosity of the solution increases with increasing molecular weight and degree of substitution of CMC.

Keywords: carboxymethyl cellulose, cellulose viscosity, hemicellulose, pineapple leaf waste.

Classification numbers: 2.2, 2.3

1. Introduction

CMC derivatives are well known and have many applications, for example, they can be used as thickeners, additives, detergents, pharmaceuticals, glues, ceramics, or cosmetics, etc. [1-5]. CMC is obtained when alkali cellulose is reacted with MCA [6]. Some studies show that among the carbon atom positions where sodium carboxymethyl groups are substituted for the OH group, the C2 position is relatively dominant [7]. Hence, the DS is a significant property of CMC particularly because of its water solubility and thickening ability. There are many parameters that affect the DS value of CMC, for example, the concentration of MCA and NaOH, reaction time, reaction temperature, cellulose size, and so on [7, 8]. Several papers have reported that CMC can be synthesised from different cellulose sources like rice straw, corn husk, cotton linter, paper sludge, and pomelo peel [9-15]. Cellulose is usually found in cell walls of plants and is generally associated with lignin and hemicellulose, which make it difficult to extract in pure form. Because of their strong inter- and intra-molecular hydrogen bonds, cellulose molecules associate over extended regions forming polycrystalline, fibrous bundles and several steps must be performed to achieve higher DS, therefore, the extraction process is neither straightforward nor inexpensive [9-12]. Pineapple leaves account for a significant amount of agricultural waste in Vietnam, about 1 million tons annually. After harvesting and processing to obtain a packaged pineapple product, the discarded pineapple leaves make up 70% of the total waste from production. When discharged into the environment, these wastes cause serious pollution. Because pineapple leaves contain about 65-70% dry weight of cellulose, 15-20% hemicellulose, and 4-6% lignin [16-21], they can be used to produce reinforced fibres for polymer composite materials as well as cellulose fibre. However, this resource is yet to be used optimally. In our previous study, Vietnamese pineapple leaves were found to consist of 55% (w/w) cellulose content [22-26]. Thereby, in a scientific aspect, this shows using cellulose from pineapple leaves for applications has high feasibility.

Although hemicellulose is a common polysaccharide, representing about 20-35% of lingo-cellulosic biomass [27-30], it has not yet found wide industrial application. In addition, hemicellulose is a non-food substitute for starch polyols and is also a substitute for petroleum-based polyols [31-35]. Thus, it can be said that hemicellulose is a potential feedstock source for biomaterial synthesis. To the best of our knowledge, no report has been found in the literature where extracts of both hemicellulose and cellulose were prepared from pineapple leaf waste. Thus, it is essential to carry out research on the separation of cellulose and hemicellulose from pineapple leaves. The successful separation and effective use of cellulose and hemicellulose from Vietnamese pineapple leaf waste will be of great significance to environmental protection while adding value to processed fruit products. Therefore, the aim of this research is to confirm the potential separation of both hemicellulose and CMC from Vietnamese pineapple leaf waste and examining if this CMC can be further used as a thickener.

2. Materials and methods

2.1. Materials

Pineapple leaves were supplied by the Pineapple Suoi Hai farm, Ba Vi, Hanoi, Vietnam. They were cut into slices 5 mm in size.

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Acetic acid 99.9% and MCA 99.7% were purchased from the UK and nitric acid 65% and sodium hydroxyl 99.9% (Merck) from Japan. Methanol 99.8%, ethanol 99.9% (Xilong Chemical), isopropanol 99.7% (Merck), and acetone 99.8% (Merck) served as solvents.

2.2. Methods

2.2.1. Hemicellulose extraction: The recovery of hemicellulose was conducted by alkaline extraction. Specifically, 10 g of dried pineapple leaf powder was mixed with 250 ml of x mol-NaOH-solution (x=0.25, 0.50, 0.75, 1.00, and 1.25 M) at 90°C while stirring. The mixing time was varied (t=60, 90, 120, and 150 min). The resulting dark slurry was filtered and washed with 250 ml of distilled water. The hemicellulose was separated in an Erlenmeyer flask.

Then, 25% acetic acid solution was used to adjust the filtrate pH 5-6. Afterward, 96% ethanol was added while maintaining the temperature at 4°C. Then the mixture was allowed to rest for 24 hours for the hemicellulose to precipitate. The liquid part was removed by vacuum. The obtained hemicellulose was washed out 3 times with a 70% ethanol solution. Then, the hemicellulose was dried in an oven at 40°C for 30 hours. Powdered hemicellulose was obtained by grinding. The final yield of hemicellulose was calculated as the average yield of three extractions and expressed as the ratio of its dried weight to 100 g of dried pineapple leaf powder before extraction using the following formula:

$$H(\%) = \frac{m}{m_0}x100 \qquad (1)$$

where H is the yield of hemicellulose; m is the weight of the obtained hemicellulose; m_0 is the weight of initial dried pineapple leaf powder.

2.2.2. Cellulose recovery: The obtained solid residual was treated with 150 ml of HNO_3 solution with y M concentration (y=0.25, 0.50, 0.75, 1.00, 1.25 M). Then, it was thermally treated at 90°C for 90 min. The mixture was filtered and washed out many times with cold distilled water until the indicator colour of the filtrate did not change. The residue was thoroughly dried at 60°C, then ground into a fine powder, and finally placed in a plastic bag. Then, the cellulose yield (H) was calculated according to Eq. (1).

2.2.3. Synthesis of CMC: To synthesise CMC, 5 g of cellulose powder was added to 150 ml of isopropanol under continuous stirring for 60 min. Next, 15 ml of x NaOH solution (x=10, 15, 20, 25% w/v) was added dropwise while stirring. The stirring was continued for 1 hour at room temperature. Finally, y g of MCA (y=1.0, 2.0, 3.0, 4.0, and 5.0 g) was added to the mixture. It was stirred for another 90 min at 60°C. The solid part was neutralised by acetic acid to pH=7.0 and immersed in 20 ml of ethanol three times for 10 min each. The filtered CMC was thoroughly dried in an oven at 60°C and stored in a plastic bag. CMC productivity was calculated according to the following formula:

$$H_{CMC}(\%) = \frac{m_{CMC}}{m_C}x100 \qquad (2)$$

where H_{CMC} is CMC yield; m_{CMC} is the weight of obtained CMC; m_C is the weight of initial cellulose.

2.2.4. Infrared spectroscopy: FTIR spectra were obtained on an FT/IR-6300 spectrometer with a resolution of 4 cm⁻¹. The absorption band was 600-4000 cm⁻¹.

2.2.5. X-ray diffraction studies: The X-ray diffraction (XRD) curves were recorded on an X-ray diffractometer XRD-6100 (SHIMADZU) with Cu K_α radiation at 30 kV and 15 mA. The diffraction angle was in the range of 5 to 80° (0.05°/min).

2.2.6. Scanning electron microscopy: To study the surface structure, SEM images were taken on a JSM-5000 model scanning electron microscope at 5.0 kV (Hitachi).

2.2.7. Determination of DS: The DS of CMC was determined according to ASTM (1994) [23]. To prepare the sample, 5 g of sample was added to a 500 ml conical flask containing 350 ml of ethanol. The contents were shaken for 30 min. The solution was filtered through a porous funnel. The solvent was evaporated at 100°C for 60 min under light vacuum. Finally, the sample was thoroughly dried in a crucible at 110°C.

The measurement procedure began with 2 g of the dry substance, prepared as above, placed into a tared porcelain crucible. The crucible was carefully charred with a low flame, then with a larger one for 10 min. Then, the residue was treated with 3-5 ml of concentrated sulfuric acid. Finally, it was heated carefully until no smoke was seen. Next, the crucible contents were mixed with 1 g of ammonium carbonate. The crucible was further heated with a low flame until no smoke was seen. Finally, the crucible was cooled and weighed. The sodium content was calculated using the following formula:

$$A(\%) = \frac{a \times 32.28}{b} \qquad (3)$$

where the weight of residual sodium sulphate is represented by a and the weight of the dry sample by b.

The following formula was used to calculate the DS.

$$DS = \frac{162 \times A}{2300 - 80 \times A} \qquad (4)$$

where 162 denotes the molecular weight of an anhydrous glucose unit; 80 represents the net increment in the anhydrous glucose unit for every substituted carboxymethyl group.

2.2.8. Viscosity measurement method: In this study, viscosity was determined by using an Ubbelohde capillary viscometer. Through the Mark-Houwink-Sakurada equation, the intrinsic viscosity was calculated as follows:

$$[\eta] = K.M^\alpha \qquad (5)$$

where [η] (cm³.g⁻¹) represents the intrinsic viscosity and K and α are the specific solvents and polymers, respectively. For example, in the case of CMC, K=7.3×10⁻³ and α=0.93 measured at 25°C in a 6% NaOH solution [24-26].

To carry out the rheological measurements, a Gilmont viscosimeter was used with 1% (w/w) CMC solution prepared in water. The temperature was maintained at 25°C during the reaction.

3. Results and discussion

3.1. Hemicellulose and cellulose extraction from Vietnamese pineapple leaf waste

3.1.1. Extraction of hemicellulose: Effect of the NaOH concentration on the hemicellulose extraction yield: The hemicellulose extraction yield depends on the concentration of the NaOH solution. Fig. 1A shows the highest yield at a NaOH concentration of 0.75 M. A very low yield (about 4.8%) was obtained at an alkaline concentration of 0.25 M NaOH. Indeed, at such a low NaOH concentration, the hemicellulose could not be solubilised since the hemicellulose was still bound to the lignin. Therefore, the hemicellulose extraction yields increased (16.1 and 16.8%) with higher NaOH concentrations (0.5 and 0.75 M, respectively). This is because the higher solubility of hemicellulose relates to the breakdown of the ester bonds between ferulic acid and hemicellulose under these conditions. However, as shown in Fig. 1A, further increasing the concentration of NaOH solution (1.00 and 1.25 M) leads to decreasing hemicellulose yield (15.1 and 13.7%, respectively). This is due to the degradation of hemicellulose [26-28]. Notably, no significant change in the hemicellulose yield could be seen in the NaOH concentration range of 0.5-0.75 M. Thus, 0.5 M NaOH is considered the most suitable.

3.1.2. Dependency of the hemicellulose extraction yield on the extraction time: Fig. 1B shows the dependence of the hemicellulose extraction yield on extraction time (60, 90, 120, and 150 min). In this research, the extraction temperature was 90°C and the NaOH concentration was maintained at 0.5 M. This shows that the hemicellulose yield increased upon increasing extraction time to 90 min. However, the hemicellulose yield declined with further increase of the extraction time to 120 min and 150 min. The partial degradation of hemicellulose may be the reason for this [25-27]. Thus, the extraction time of 90 min was considered the most appropriate.
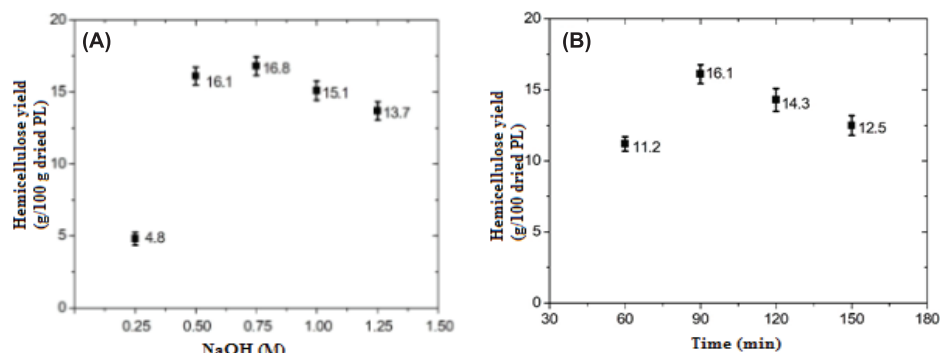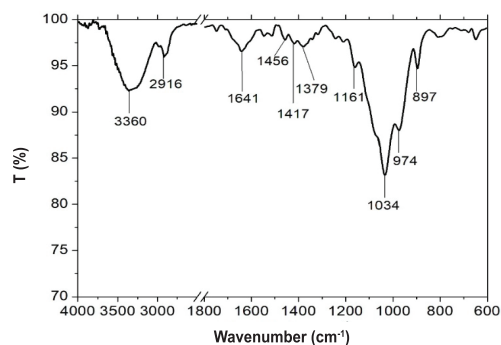

Fig. 2. FTIR spectroscopy of hemicellulose.

The FTIR spectroscopy analysis is shown in Fig. 2. In this figure, the absorption peaks at 1456, 1417, 1379, 1161, 1034, 974, and 897 cm^{-1} are specific for hemicellulose [29, 30]. The C-O-C stretching of the glycosidic linkages of the xylans were demonstrated by a band at 1034 cm^{-1} [29, 31]. The signal at 974 cm^{-1} was due to the presence of arabinose units [32]. The β-(1,4)-glycosidic linkages between the sugar units were characterised by a strong absorption band at 897 cm^{-1} [27, 33]. The broadband at 3360 cm^{-1} belonged to the OH stretching vibration, while the signal at 2916 cm^{-1} was evidence of the stretching vibrations of the CH_2 group. The peak at 1379 cm^{-1} was due to the stretching and deformation vibrations of the C-H group in the glucose unit. It is likely that the 1641 cm^{-1} peak was due to trace amounts of water [26]. In addition, the bands at 1242 and 1161 cm^{-1} were believed to belong to C-H stretching and O-H or C-O bending vibrations. The small peak at 1546 cm^{-1} likely embodies the aromatic skeletal vibrations due to the presence of trace amounts of lignin. Furthermore, the band at 1740-1710 cm^{-1} for the carbonyl groups is not seen, meaning no xylan acetyl groups were present in the alkaline solution. Overall, these FTIR results are similar to those of other studies [29, 34].

3.1.3. Cellulose extraction: Optimal HNO_3 concentration: The yield of cellulose recovery depends on the concentration of HNO_3. The results are shown in Table 1.

Table 1. The yield of carboxymethylation with different concentrations of HNO_3.

C_{HNO_3} (M)	0.25	0.50	0.75	1.00	1.25	1.50
H_c (%)	56.75±0.81	58.80±.65	55.40±1.13	49.20±0.71	43.20±0.42	37.20±0.40

It can be seen from Table 1 that the cellulose recovery yield does not vary much when the concentration of HNO_3 increases from 0.25 to 0.75 M. However, the yield reduces from 55.40±1.13 to 37.20±0.40% when the HNO_3 concentration continues to increase to 1.5 M. This could be due to the 0.75 M HNO_3 solution not only removing the residual hemicellulose but also cutting the cellulose fibres. During washing, some short cellulose

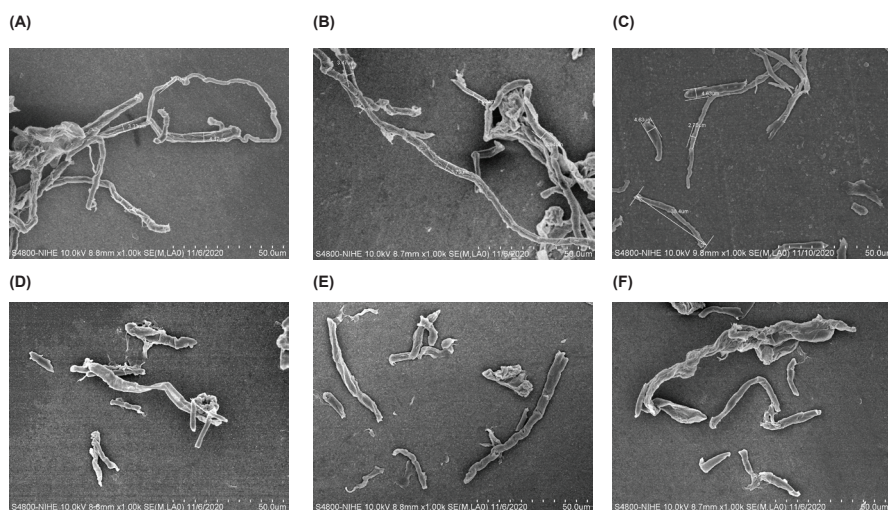Fig. 1. Dependence of the hemicellulose extraction yield on NaOH concentration (A) and extraction time (B).

Fig. 3. SEM images of pineapple leaf cellulose treated with different HNO₃ concentrations: (A) 0.25 M, (B) 0.50 M, (C) 0.75 M, (D) 1.00 M, (E) 1.25 M, (F) 1.50 M with 1000x magnification.

fibres may be lost, resulting in smaller cellulose recovery yields. The SEM image in Fig. 3 also supports this point of view.

As Fig. 3 shows, pineapple leaf cellulose are cylindrical fibres with an average diameter of 3-4 µm. At an HNO₃ concentration of 0.25-0.50 M, the length of the cellulose fibres are relatively long, however, at a concentration of 0.50 M, the fibre surface is smoother than that at 0.25 M. It is assumed that the impurities could have been more thoroughly removed at that concentration. It is also clear that from the HNO₃ concentration of 0.75 M that the cellulose fibres were truncated, and the number of short fibres increased with further increase in HNO₃ concentration. However, there was no difference in fibre size in the concentration range of 1.0-1.5 M. Thus, the cellulose fibre length can be adjusted by the concentration of the HNO₃ solution. At a concentration of 0.50-0.75 M, the obtained cellulose fibres have an average size of about 50-200 µm with an average diameter of about 3-4 µm. Thus, a suitable concentration of HNO₃ to treat the pineapple leaves to achieve the highest cellulose recovery efficiency is in the range of 0.50-0.75 M, with the choice depending on the desired fibre length.
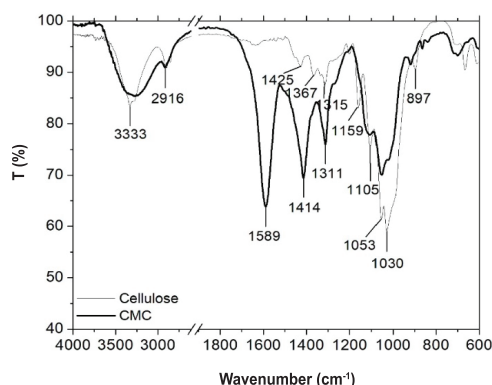

Fig. 4. FTIR spectroscopy of extracted cellulose and CMC from pineapple leaf waste.

FTIR spectroscopy of the cellulose is displayed in Fig. 4. The band at 3333 cm^{-1} characterised the OH stretching mode, while the signal observed at 2916 cm^{-1} was attributed to the stretching and deformation vibrations of the C-H groups in the glucose units. The bands at 1367 and 1425 cm^{-1} characterised the C-H and C-O linkages in the polysaccharide rings, respectively. The absorption peak at 1159 cm^{-1} belongs to the -C-O-C stretch of the β-(1,4)-glycosidic linkage, which is typical for cellulose samples. The peak at 1425 cm^{-1} is typical for the asymmetric bending of the -CH₂ group. The absorption band at 1105 cm^{-1} was assigned to the -C-O group of secondary alcohols and ether functions existing on the cellulose chain backbone. Lastly, the band range of 897-1053 cm^{-1} characterised the β-(4,1)-glycosidic linkages between the glucose units in cellulose [26]. Interestingly, there are no peaks at 1600-1800 cm^{-1}, which characterises the C=O groups and the aromatic rings of hemicellulose and lignin molecules [34-36]. This result proves that the obtained cellulose is pure, that is, free from hemicellulose and lignin. It is worth mentioning that the separation of cellulose described in this work is easier and the yield of cellulose is higher compared to that mentioned in our previous report [36]. This interesting result may be due to the different states of starting material. Therefore, the use of fresh pineapple leaves brings higher economic efficiency than its dried state.

3.2. Synthesis of CMC from Vietnam's pineapple leaf cellulose

3.2.1. Effect of NaOH concentration on DS and yield of CMC: The treatment of cellulose with alkali intentionally causes cellulose fibres to swell, making the substitution reaction easier. The relationship between the DS of the CMC and NaOH concentration is shown in Table 2.

Table 2. The yield and DS of CMC with various NaOH concentrations.

NaOH (%wt)	10	15	20	25
H$_{CMC}$ (%)	160.1	172.3	168.8	166.6
DS	0.64	0.86	0.75	0.71

As seen in Table 2, upon increasing NaOH concentration, the DS of the CMC increased. The DS reached a maximum of 0.86 at a NaOH concentration of 15% (w/v). However, the DS value then decreased by further increasing the NaOH concentration. This observation can be explained as follows. At the beginning of the carboxylation process, as NaOH reacts with the cellulose-OH groups, the cellulose morphology is altered. Its crystalline domains transform into amorphous domains [9]. However, this morphology change only occurs up to a certain level, since carboxylation is also influenced by other factors like solvents and reagent ratios. Furthermore, the reaction between MCA and

NaOH can occur during carboxylation. In particular, this reaction is more likely to occur at high concentrations of NaOH, leading to a reduced etherification probability. In addition, at a high NaOH concentration, degradation of CMC polymer chains may occur. The decreasing DS value at high NaOH concentration was previously found in research by K.M. Hong (2013) [8] and J. Chumee, D. Seeburin (2014) [10]. The CMC yields at different NaOH concentrations are also provided in Table 2 and the trend was similar to that of DS.

3.2.2. Effect of MCA dose on DS and yield of CMC: The yield of CMC and the DS value greatly depend on the MCA dose. The weight ratio of MCA to cellulose changed from 0.2 to 1.0. Table 3 shows that the DS of the CMC increases with increasing the weight ratio of MCA to cellulose, from 0.2 to 0.6, then it decreases lightly. The DS reached a maximum at the weight ratio of MCA to cellulose of 0.6. This may relate to undesired side reactions at a high MCA dose, leading to the reducing CMC yield. This similar range of DS values (from 0.58-0.86) can also be seen in another research for passage waste [9, 12]. Like DS, a similar varying trend of CMC yield is also seen in Table 3.

Table 3. The yield and DS of CMC synthesised with various amount of MCA.

m_{MCA} (g)	0.2	0.4	0.6	0.8	1.0
H_{CMC} (%)	143.7	160.7	172.3	154.0	150.1
DS	0.34	0.60	0.86	0.46	0.41

In summary, suitable carboxylation conditions were the weight ratio of MCA to cellulose of 0.6, and 15 ml of 20% w/v NaOH solution. With these conditions, the CMC had a DS of 0.86.

3.2.3. Effect of cellulose size on DS and yield of CMC: The effect of cellulose fibre size on the carboxymethylation reaction was studied. Two fibres with an average diameter of 50 and 200 μm were selected and the results are shown in Fig. 5.
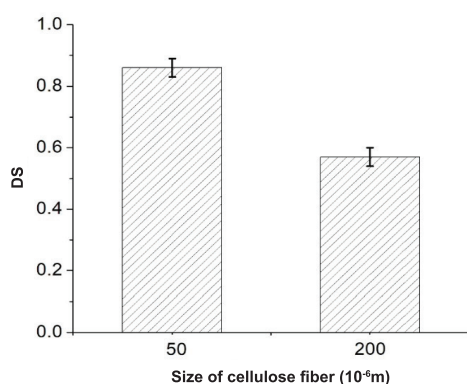

Fig. 5. DS of the synthesised CMC depending on cellulose fibre size.

As Fig. 5 shows, the size of the raw cellulose fibre material greatly affects the DS of CMC. It is well known that increasing cellulose fibre length makes DS decrease. Indeed, for the 200 μm fibre, DS was only 0.57±0.05, while DS reached 0.86±0.03 with the 50 μm fibre.

3.2.4. Structural characterisation of CMC: In our research, cellulose fibres of 200 μm were used to synthesise CMC. The FTIR spectroscopy technique was used to confirm the structure of the extracted cellulose and synthesised CMC (see Fig. 4). It can be seen that the OH-stretching vibrations appear at a broad peak of 3332 cm⁻¹. The peak at 2916 cm⁻¹ shows the stretching C-H-linkage. The bands at 1315 and 1159 cm⁻¹ are due to C-O-C stretching vibrations in the β-(1,4)-glycosidic linkage. The -C-O- group of secondary alcohols and ethers in the cellulose backbones is characterised by the band at 1105 cm⁻¹. The β-(1,4)-glycosidic linkages between the glucose units in cellulose appear at 1053 and 1022 cm⁻¹ [9, 12, 26]. For the CMC, the presence of the -COO and -COONa groups are confirmed by C=O stretching absorption at 1589 and 1414 cm⁻¹, respectively. These peaks do not exist in the cellulose spectrum (Fig. 4), meaning that etherification was successful. These FTIR results are similar to those of J. Chumee and D. Seeburin (2014) [10], S. Sophonputtanaphoca, et al. (2019) [13] research on Thai pineapple leaves. The microstructures of the materials were studied by SEM. Photos of the cellulose and CMC are shown in Fig. 6 and their SEM images are shown in Fig. 7.
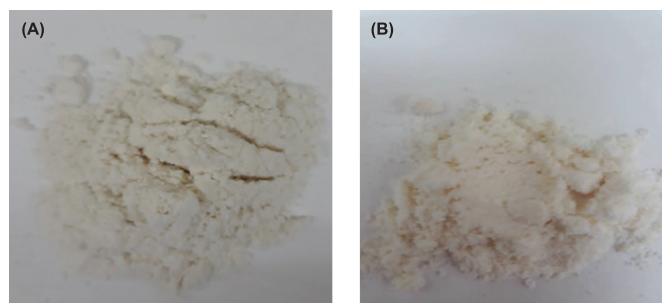

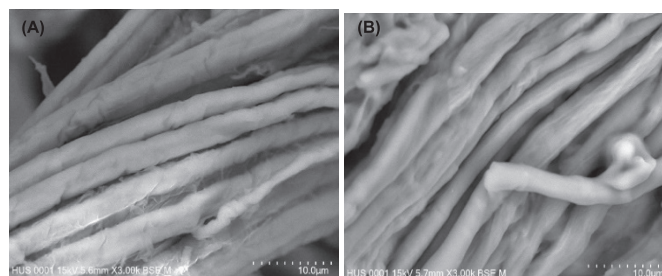**Fig. 6. Images of pineapple leaf cellulose (A) and CMC (B).**

Fig. 7. SEM images of pineapple leaf cellulose (A) and CMC (B) with 3000 x magnification.

As seen in Fig. 7, cellulose and CMC possess a ribbon-shaped or rod-like morphology. The same morphology has been shown by other researchers [13, 14, 19]. This figure also clearly shows the fairly smooth surface of the extracted cellulose fibres. As for CMC, the surface is more extended, rough, and collapsed. The reason for this lies in the fact that the extracted cellulose was treated with sodium hydroxide during carboxymethylation. The size of the cellulose is 3.0-4.0 μm and that of the CMC is 3.5-4.5 μm.

We used XRD to investigate the degree of cellulose crystallinity [12, 13, 20]. Fig. 8 shows the X-ray diffractogram of isolated cellulose and synthesised CMC.
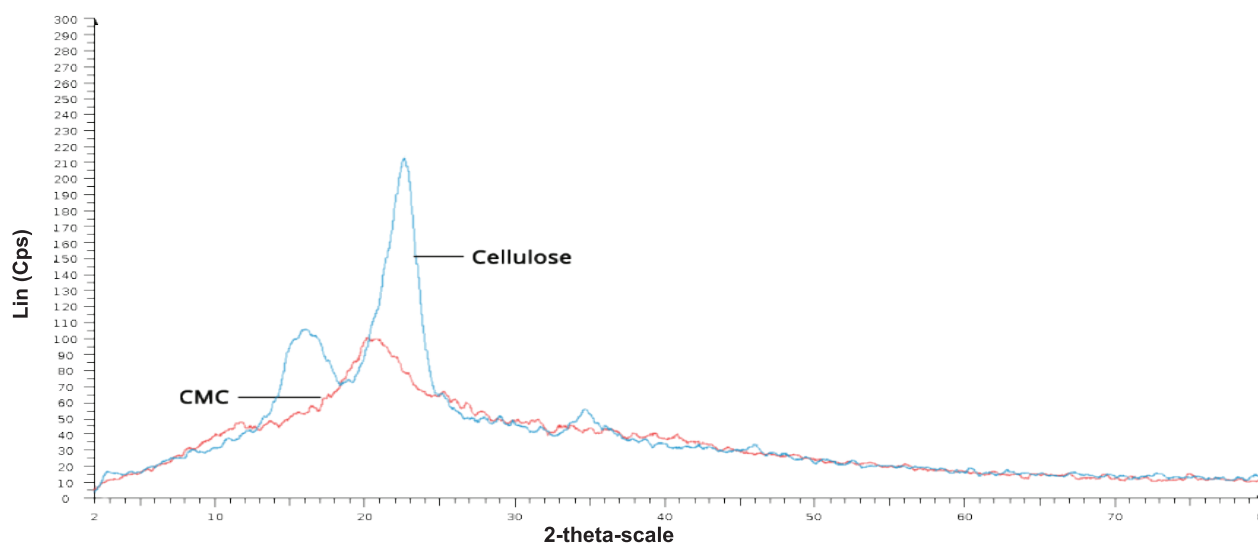
Fig. 8. XRD diffraction pattern of cellulose (blue curve) and synthesised CMC (red curve) from Vietnamese pineapple leaf waste.

Looking at Fig. 8, it can be assumed that holocellulose has a higher crystallinity compared to CMC. Specifically, the holocellulose curve has three peaks at 2θ=16.3, 23.4, and 34.7°. Among them, the two peaks at 2θ=16.3 and 23.4° are sharp, showing larger crystalline phases of the holocellulose core structure. Note that CMC has fewer peaks with lower intensity compared to holocellulose. This is due to a more amorphous structure of CMC compared to that of cellulose. One can assume from the above observation that unlike cellulose, CMC could have a disordered molecular arrangement. This is because of the presence of carboxymethyl moieties in the CMC product. The molecular weight (M) of CMC is one of the most important parameters. By extrapolation of the reduced viscosity [η_{red}] to zero concentration, one can obtain the intrinsic viscosity [η] (Fig. 9). In order to determine the molecular weight by the viscosimetric method, the Mark-Houwink-Sakurada empirical equation (Eq. (5) is used. The [η] values can be estimated from the intercept of the plot, specifically, [η]=188.17 (ml/g). The CMC average molecular weight is 55400±250 g/mol.
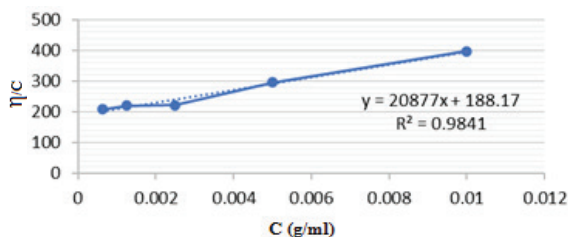

Fig. 9. The Mark-Houwink-Sakurada plot for CMC in 6%wt NaOH at 25°C.

3.3. Evaluation of the thickening ability of CMC products

The thickening ability of the CMC product was evaluated with varying DS values (DS=0.34, 0.60, and 0.86) and different molecular weights (M_w=22000±221 and 55400±250 g/mol). The

samples were prepared with a CMC mass concentration of 1% in water. The values of viscosity measurements with a gilmont viscometer are given in Table 4.

Table 4. The dynamic viscosity of the CMC solution at 25°C.

DS	0.34	0.60	0.86	0.86	
M (g/mol)	55400±250			22000±221	55400±250
Viscosity (mPa.s)	385	520	650	250	650

Table 4 shows that DS and M have a great impact on the viscosity of the solution. The viscosity of the 1% CMC solution increases with an increase of DS. This can be explained by the fact that a high value of DS creates a greater number of hydrophilic groups, leading to increasing viscosity (at the constant temperature). This observation is supported by other reports [11, 13, 21].

The molecular mass of CMC has a decisive influence on the thickening ability of CMC. At the same 1% CMC concentration, the viscosity of the solution increases sharply, from 250 to 650 mPa.s, when the molecular weight changes from 22,000 to 55,400 g/mol. This is understandable because the larger the molecular mass, meaning the bulkier the molecules, the harder it is for them to move in solution. This similar observation can be seen in other reports [10, 20]. It is expected that the synthesised CMC may be useful as a thickening agent in many applications.

4. Conclusions

The extraction of cellulose and hemicellulose from Vietnamese pineapple leaf waste was successfully performed. Hemicellulose extraction yield was 16.1% (w/w) at a NaOH concentration of 0.5 M at 90°C for 90 min. The cellulose recovery yield was 58.8% while treating for 90 min with 0.5 M HNO_3. The highest DS of CMC was obtained (0.86) with 15% (w/v) NaOH solution, 0.6 g of MCA/g cellulose, and a cellulose size 50 μm. The CMC yield was 172.3%.

The cellulose fibre size depends on HNO_3 solution concentration. The length of the cellulose chains strongly influences the effectiveness of the carboxymethylation reaction. The DS and molecular weight of CMC have a significant impact on thickening ability. The viscosity of the CMC solution increases with increasing DS and molecular weight. This study shows that using Vietnamese pineapple leaf waste to separate cellulose and hemicellulose has great potential and high feasibility. This research opens a new direction to effectively use pineapple leaf waste while contributing to reducing environmental pollution.

CRediT author statement

Phan Thi Tuyet Mai: Review, Methodology, Formal analysis, Writing, Editing; Nguyen Hai Linh: Data analyst; To Phuong Linh: Data analyst; Vu Tien Manh: Data analyst. Ngo Hong Anh Thu: Editing; Pham Ngoc Lan: Review, Editing; Luu Thi Hue: Review, Editing.

ACKNOWLEDGEMENTS

COMPETING INTERESTS

The authors declare that there is no conflict of interest regarding the publication of this article.

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