

Preparation of hydrogel based on cellulose from sugarcane bagasse

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Abstract. In this study, the preparation and characterization of hydrogels from sugarcane bagasse were investigated. The preparation of hydrogels was made with cellulose extracted from sugarcane bagasse in the presence of an initiator, a monomer and a crosslinking agent. The structural characterization of the hydrogels was performed with Fourier-transform Infrared spectroscopy and Focused Ion Beam Scanning Electron Microscopy. The properties of the hydrogel were investigated with regards to mechanical properties, swelling degree and Thermogravimetric Analysis. The suitable condition for the preparation was found at 1.0 g initiator/kg cellulose, 1.6 g monomer/kg cellulose and 0.2 g crosslinking agent/kg cellulose. It was found that both anion and cation can be absorbed in the crosslinked structure of the hydrogel. The Young's modulus, stress at break, elongation at break and Charpy impact of the hydrogel prepared in the suitable condition is 1.3 MPa, 5.2 MPa, 89.0 % and 7.2 kJ/m², respectively. The hydrogel is better at thermal resistance than the cellulose. The results show that the obtained hydrogel may be suitable for electrolyte membrane used in battery.

Keywords: hydrogel, cellulose, sugarcane bagasse.

Classification numbers: 2.3.1, 2.3.2, 2.5.2.

1. INTRODUCTION

Hydrogels are defined as hydrophilic polymers of which structure is 3D-network. Hydrogels may act as a medium for the transportation of ions or molecules such as water, carbon dioxide, lithium ion, and proton. They can also keep larger ions or molecules in their 3D-network. Therefore, hydrogels have a great opportunity for applications in drug delivery vehicles, electrolyte membrane, tissue engineering, reverse osmosis membrane and so forth [1, 2].

It has been shown that cellulose has interesting properties and is promising as a cheap and green material to form a hydrogel. Ammonium persulfate, acrylic acid, and acrylamide are usually used in the preparation of hydrogels from cellulose. In the first stage, ammonium

persulfate (APS) initiator reacts with cellulose to generate the cellulose radical. In second stage acrylic acid (AA) monomer graft-copolymerizes on the cellulose radicals. In the third stage, N,N'-methylenebisacrylamide (MBA) forms crosslinks in hydrogel [3,4]. The schematic preparation of hydrogel based on cellulose, acrylic acid (AA), APS, and MBA is shown in Figure 1 [4].

In the previous works, hydrogels were successfully prepared from crude cellulose, carboxymethyl cellulose, or cellulose purified from tea residue, pineapple leaf, etc. [5 - 8]. The properties of the hydrogels depend on the structure and crystalline degree of cellulose. In other words, the different resources of cellulose will generate cellulose hydrogels with respective properties. The study in preparation and characterization of hydrogels from various cellulose resources are still ambiguous.

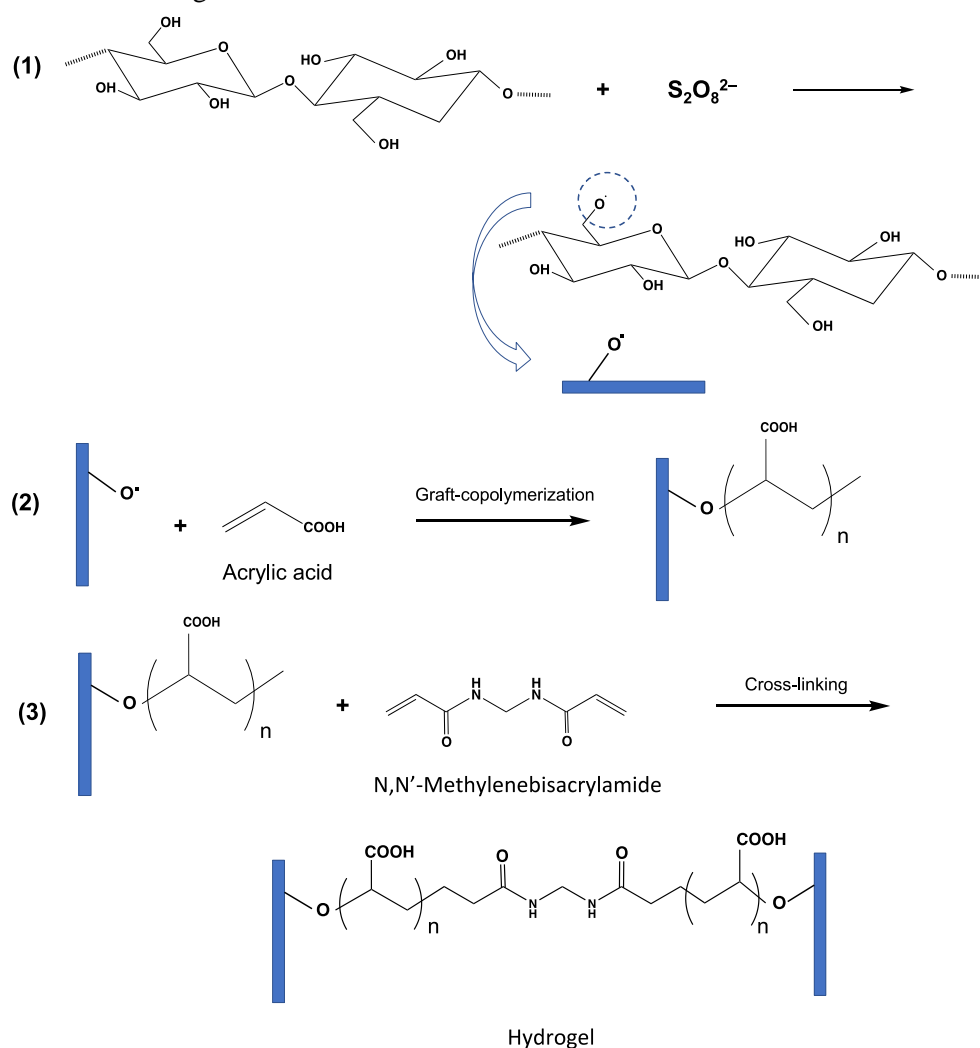


Figure 1. Schematic preparation of hydrogel based on cellulose [4].

The percentage of cellulose in sugarcane bagasse is about 40 %, and the rest is hemicellulose and lignin [9]. In Viet Nam, there is a large amount of sugarcane bagasse, but the treatment of sugarcane bagasse has not been paid the appropriate attention. When sugarcane

bagasse is used to prepare hydrogel, the obtained hydrogel is a green material, and the preparation procedure may provide the strategy to increase the value of sugarcane bagasse. The preparation of hydrogel based on sugarcane bagasse has been reported in literature [3, 10, 11]. For instance, Mondal and co-workers [3] used sugarcane bagasse containing cellulose, hemicellulose, lignin, fatty and waxy materials, ash to produce hydrogel. In the research work of Pei Xin Chia *et. al.* [11], carboxymethyl cellulose prepared from sugarcane bagasse was used to prepare hydrogel. In those works, the condition for preparation and structural characterization were carried out. However, the properties which are useful for practical applications were not thoroughly characterized. Therefore, it is necessary to investigate the preparation procedure and the properties of cellulose hydrogel from sugarcane bagasse to find the suitable applications.

In this work, cellulose extracted from sugarcane bagasse was used to prepare hydrogel in the presence of AA, MBA, and APS. The preparation procedure followed the method reported in previous works [12, 13]. The suitable condition to prepare the hydrogel was investigated. The structure and morphology characterization of the hydrogel was made through Fourier Transform Infrared (FT-IR) spectroscopy and Focused Ion Beam Scanning Electron Microscopy (FIB-SEM). The mechanical properties in terms of Young's modulus, stress at break and elongation at break and Charpy impact were studied. The thermal resistance and swelling degree of the hydrogel were also investigated. The results expressed that the obtained hydrogel may be suitable for electrolyte membrane used in battery.

2. MATERIALS AND METHODS

2.1. Materials

Sugarcane bagasse was from sugarcane tree *Saccharum officinarum L.* cultivated in the North of Viet Nam in summer. Hydrochloric acid solution (HCl, 36 % in H₂O), sodium hydroxide (NaOH, powder, 97 %), acrylic acid (AA, anhydrous, 99 %), N,N'-Methylenebisacrylamide (MBA, powder, 99.5 %) were purchased from Sigma Aldrich. Sodium dodecyl sulfate (SDS, 98 %), sodium hypochlorite solution (NaClO, 10 % in H₂O), ammonium persulfate (APS, 98 %) were purchased from Nacalai Tesque Inc.

2.2. Preparation of materials

2.2.1. Purification of cellulose from sugarcane bagasse

The purification of cellulose from sugarcane bagasse was carried out according to our previous works [14, 15]. The sugarcane bagasse was cut into small pieces, then washed many times with tap water, followed by drying in sunlight. To remove hemicellulose and lignin, sugarcane bagasse was soaked in the solution of HCl 4 % and NaOH 10 % in sequence. After that, the sample was soaked in NaClO 4 % for 1 hour at room temperature. The obtained powder was washed with distilled water, then filtered. The resulting material was cellulose. The cellulose was dried at 50 °C in reduced pressure for a week.

2.2.2. Preparation of hydrogel from cellulose

The procedure for preparation of hydrogel cellulose was schematically illustrated in Figure 2. Cellulose was dispersed in NaOH 4 M solution at 60 °C for 30 minutes. The cellulose colloid was obtained. Thereafter, APS, AA and MBA were added into the colloid. The graft-

copolymerization reaction occurred at 60 - 70 °C for 5 hours. The resulting product, i.e. the cellulose hydrogel, was casted film. The film was preserved in desiccator before characterization. The raw material, i.e. sugarcane bagasse, and the product, i.e. hydrogel, are shown in Figure 3.

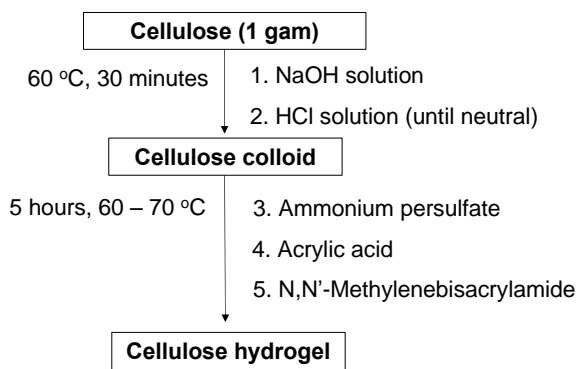


Figure 2. Procedure for preparation of hydrogel cellulose.



Figure 3. Image of sugarcane bagasse and the hydrogel based on cellulose.

To find the suitable condition for the preparation of hydrogel, we used different amounts of APS, AA, and MBA. The names of the samples prepared in various conditions are shown in Table 1.

Table 1. The conditions for the preparation of hydrogel.

Sample	APS (g/kg cellulose)	AA (g/kg cellulose)	MBA (g/kg cellulose)
Hydrogel -1	1.2	1.6	0.2
Hydrogel -2	1.0	1.6	0.2
Hydrogel -3	0.8	1.6	0.2
Hydrogel -4	1.0	1.4	0.2
Hydrogel -5	1.0	1.8	0.2
Hydrogel -6	1.0	1.6	0.1
Hydrogel -7	1.0	1.6	0.3

2.3. Characterization

FT-IR spectra of the samples were performed in the attenuated total reflection mode (ATR) at resolution of 4 cm⁻¹ in the region of 400–4000 cm⁻¹ and 64 scans using a JASCO FT-IR 4600 spectrometer.

The surface of samples which were sputter coated with a gold layer was observed through a scanning electron microscope NovananoSEM at the accelerating voltage of 5 kV. FIB-SEM was used to observe the morphology of hydrogel, using NovananoSEM at an acceleration voltage of 5 kV. Depth profiles and elemental mapping studies were carried out using positively charged ions generated at a current of 1.4 nA.

The hydrogel film was cut into a dumbbell shape according to ASTM D882. The Young's modulus, stress at break and elongation at break were measured using a Zwick Tensiler Z 2.5 testing machine with a load cell of 5 kN. The speed of crosshead was 2 mm/minute. The measurement was conducted at 25 °C. The measurement was repeated 3 times for each sample.

Charpy impact for hydrogel samples was measured in a Charpy impact tester (Toyoseiki). The measurements were performed at 25 °C, and repeated 3 times for each sample.

The swelling degree (SD) of hydrogel films was investigated at 25 °C. The hydrogel films were cut into 2 cm × 2 cm pieces, weighed and immersed in 168 hours into respective liquids, which were HCl solution (pH 4), deionized water (pH 7), and NaOH solution (pH 10). After that, the samples were taken out, wiped with tissue paper to remove liquid on the surface and weighed. The SD was calculated as the following equation:

$$SD (\%) = \frac{W_f - W_i}{W_i} \times 100$$

where W_f is the weight of sample after the measurement, and W_i is the weight of initial sample.

The measurement of SD was repeated 3 times for each sample.

Thermogravimetric analysis (TGA) was carried out in a Netzsch TG 209F1 Libra. The sample was heated from room temperature to 450 °C in nitrogen atmosphere. The heating rate was 10 °C/minute.

3. RESULTS AND DISCUSSION

3.1. FTIR spectra

Figure 4 shows the FT-IR spectrum of the cellulose and a hydrogel. Herein, the FT-IR spectrum of Hydrogel-2 is displayed. The FT-IR spectra of other hydrogels are identical and are not shown here. In Figure 4a, the characteristic signals of cellulose appeared. The broad peak at 3340 cm⁻¹ was the characteristic signal of O–H bonds stretching vibration in polysaccharide [16]. The signal at 2904 cm⁻¹ was the characteristic signal of C–H stretching vibration in the chain of polysaccharides [17]. The signal at 1628 cm⁻¹ may be assigned to bending vibration of O–H bonds of water absorbed in the sample [16]. The signal at 1050 cm⁻¹ was attributed to stretching vibrations of C–O bonds in cellulose [18]. The characteristic signals of cellulose appeared in FT-IR spectrum of the sample confirmed that cellulose was successfully purified from sugarcane bagasse. FT-IR spectrum of the hydrogel based on cellulose is shown in Figure 4b. The signal from 2924 cm⁻¹ to 3300 cm⁻¹ which was assigned to characteristic signal of O–H was broadened [16]. The broad signal may be due to the large amount of absorbed water present in the hydrogel. This signal may cover other characteristic signals in this region.

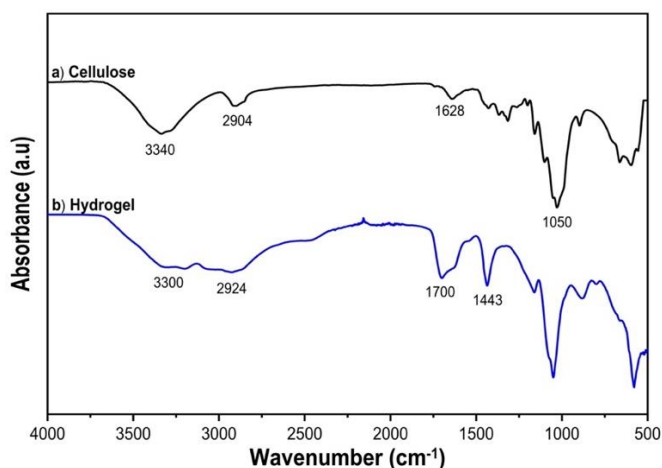


Figure 4. FTIR spectrum of sample: (a) cellulose, (b) hydrogel.

The noteworthy is that the presence of signal at 1700 cm^{-1} and 1443 cm^{-1} . The signal at 1700 cm^{-1} may be attributed to the stretching vibration of $\text{C}=\text{O}$ bonds of acrylic acid unit which was graft-copolymerized in the hydrogel [19]. The signal at 1443 cm^{-1} may be associated to the bending vibration $\text{C}-\text{H}$ in CH_3 group of acrylamide which was present in crosslinking junctions [19]. The appearance of these signals may confirm that AA and MBA reacted with cellulose during the preparation process of hydrogel.

3.2. Morphology

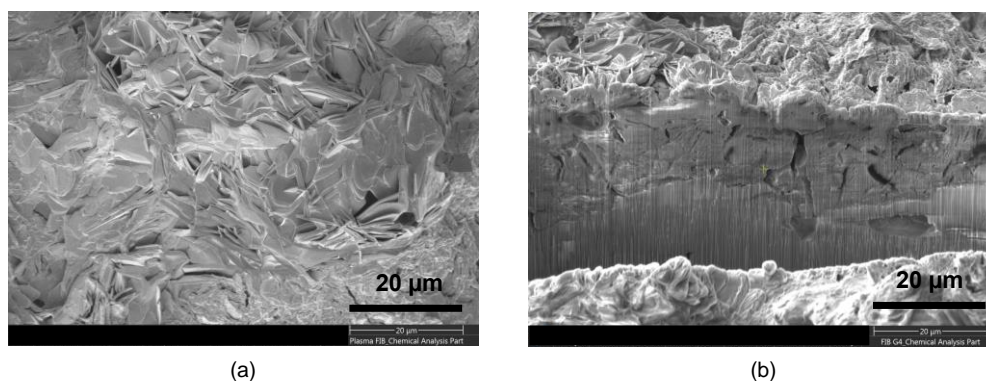


Figure 5. SEM image of sample (magnification x2500): (a) SEM image of surface of the hydrogel, (b) FIB-SEM image of the hydrogel.

The morphology of the hydrogel was observed through SEM and FIB-SEM image as shown in Figure 5. Herein, the SEM and FIB-SEM image of Hydrogel-2 are displayed. The observations of other hydrogels showed that their morphology was similar. Thus, the SEM and FIB-SEM images of other hydrogels are not shown here. The SEM image indicated that the surface of the hydrogel was not homogeneous with many drains. These can afford the adhesion of liquid on the surface of the hydrogel. In FIB-SEM image, it was clearly seen the voids inside the hydrogel. These voids may provide space for water or electrolyte solution. When water or electrolyte solution resides in the voids, the hydrogen bonding between cellulose chains and

water may firm water inside the structure of the hydrogel. Therefore, the hydrogel prepared in this work may be an ideal material for electrolyte membrane used in battery.

3.3. Swelling degree

The swelling degree of the hydrogel in water depends on the pore size and crosslink density of the sample. This is determined by the preparation process of the hydrogel, in which the content of APS, AA, and MBA plays an important role. In this part, we investigated the relationship between the swelling degree of the hydrogel in various media and the content of APS, AA, and MBA.

Table 2. Swelling degree (%) of hydrogel in various media.

Sample	In acidic medium (pH = 4)	In neutral medium (pH = 7)	In basic medium (pH = 10)
Hydrogel -1	233 ± 5	219 ± 4	358 ± 7
Hydrogel -2	231 ± 1	217 ± 1	356 ± 6
Hydrogel -3	199 ± 1	157 ± 5	289 ± 8
Hydrogel -4	224 ± 3	209 ± 7	314 ± 3
Hydrogel -5	229 ± 2	218 ± 2	354 ± 5
Hydrogel -6	200 ± 2	185 ± 6	305 ± 6
Hydrogel -7	235 ± 6	220 ± 2	361 ± 4
Cellulose	44 ± 0	30 ± 2	32 ± 3

Table 2 tabulated the swelling degree of the hydrogel in HCl solution, deionized water, and NaOH solution. It could be seen the swelling degree of the hydrogels was much higher than that of the cellulose. The swelling degree in basic medium, i.e. NaOH solution, was the highest, followed by that in acidic medium. The swelling degree in neutral medium, i.e. deionized water, was the lowest. This phenomenon suggested that the hydrogel could absorb OH⁻ and H⁺ in its structure, and the OH⁻ absorption was better than the H⁺ absorption. It also implied that the hydrogel prepared in this work could absorb both anion and cation, which enabled the hydrogel to be the media for the drug delivery, ion transportation and so forth.

As comparing the swelling degree of the hydrogel prepared with various content of APS, AA, and MBA; it was found that when the amount of APS, AA, and MBA increased, the swelling degree also increased. This was probably explained that the increase of APS amount would cause an increase of radical concentration, which improved the grafting efficiency of AA. The increase of the grafting efficiency and the amount of AA and MBA would increase the crosslink density of the hydrogel [4]. When the amount of APS, AA and MBA was lower than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the swelling degree of samples decreased sharply as seen in Hydrogel-3, Hydrogel-4, Hydrogel-6. In the other hand, when the amount of APS, AA and MBA was higher than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the swelling degree of samples slightly increased as seen in Hydrogel-1, Hydrogel-5, Hydrogel-7. This phenomenon was probably due to the redundancy of the reagents. When the APS amount was so high, the

radical concentration was also high. In this case, two free radicals would be ready to react with each other to form a stable, non-radical adduct as reported in literature [15]. The remaining AA and MBA might not support the formation of crosslinked structure in the hydrogel. Therefore, it could be concluded that 1.0 g APS/kg cellulose, 1.6 g AA/kg cellulose and 0.2 g MBA/kg cellulose were the suitable amounts to prepare the hydrogel with good swelling degree.

3.4. Mechanical properties

Table 3. Mechanical properties of the samples.

Sample	Young's modulus (MPa)	Stress at break (MPa)	Elongation at break (%)	Charpy impact (kJ/m ²)
Hydrogel -1	1.2 ± 0.0	5.3 ± 0.9	90.3 ± 7.7	7.3 ± 1.8
Hydrogel -2	1.3 ± 0.2	5.2 ± 0.7	89.0 ± 5.0	7.2 ± 1.1
Hydrogel -3	0.5 ± 0.1	2.9 ± 0.7	55.6 ± 1.2	5.1 ± 0.6
Hydrogel -4	0.8 ± 0.1	3.5 ± 0.2	75.8 ± 6.9	4.8 ± 0.9
Hydrogel -5	1.9 ± 0.3	4.0 ± 0.5	44.2 ± 4.1	6.2 ± 1.3
Hydrogel -6	0.2 ± 0.0	3.3 ± 0.1	30.1 ± 2.5	3.3 ± 0.7
Hydrogel -7	1.4 ± 0.3	4.9 ± 0.2	91.5 ± 8.7	7.8 ± 0.9

Young's modulus, stress at break, elongation at break and Charpy impact are shown in Table 3. The mechanical properties of Hydrogel-1, Hydrogel-2 and Hydrogel-7 were quite similar, and the quantities are higher than those of Hydrogel-3, Hydrogel-4, Hydrogel-5, Hydrogel-6. It was found that when the amount of APS, AA and MBA was higher than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the mechanical properties of the samples did not change much. However, when the amount of APS, AA and MBA was lower than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the mechanical properties reduced significantly. It may be explained that this content of APS, AA and MBA is suitable for the reaction to form radicals from cellulose and form crosslinks between cellulose chain. When the content of APS, AA and MBA was lower, the crosslink density of the hydrogel was probably lower. These crosslinks enhanced the mechanical properties of the hydrogel. In other hand, when the content of APS, AA and MBA was higher than 1.0, 1.6 and 0.2 g/kg cellulose, the crosslink density might be saturated, which led the constancy in the mechanical properties. This phenomenon suggested that the suitable amount of APS, AA, and MBA to prepare the hydrogel was 1.0, 1.6, and 0.2 g/kg cellulose. The mechanical properties of Hydrogel-1, Hydrogel-2 and Hydrogel-7 were quite similar, whereas the amount of APS, AA and MBA for preparation of Hydrogel-2 was the smallest. The result in the mechanical properties was in good agreement with that in swelling degree. We used Hydrogel-2 for further investigation.

Young's modulus, stress at break, elongation at break and Charpy impact of Hydrogel-2 is 1.3 MPa, 5.2 MPa, 89.0 % and 7.2 kJ/m², respectively. These values are acceptable for application of the material in electrolyte membrane.

3.5. Thermal resistance

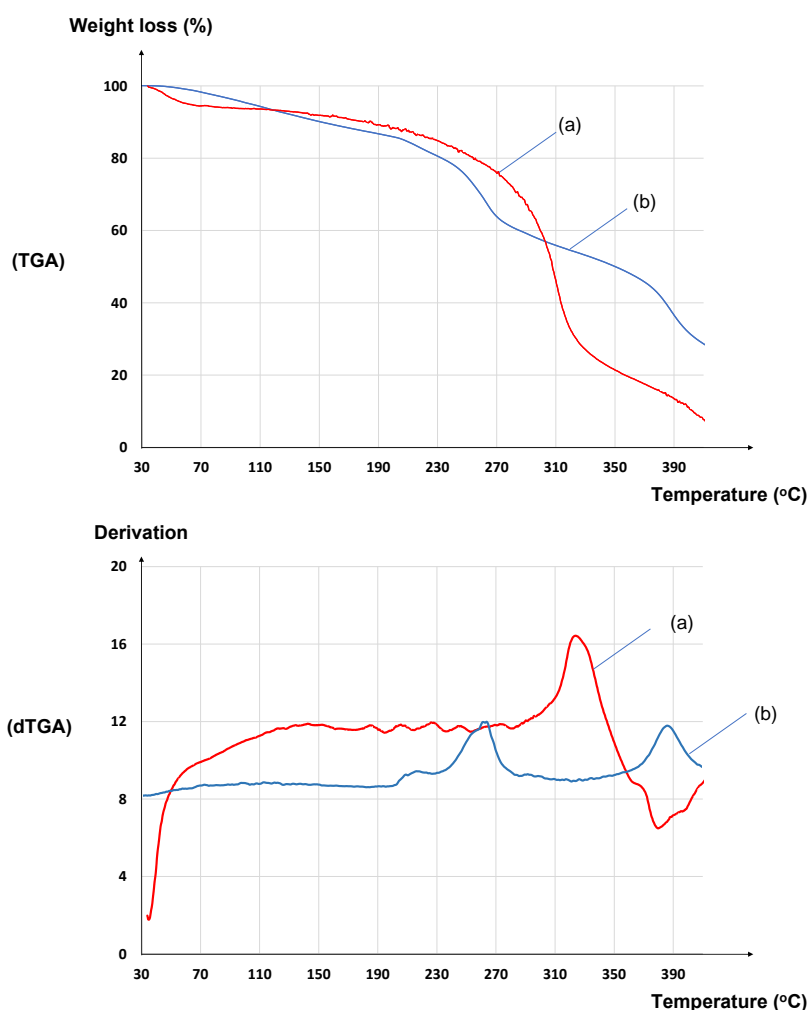


Figure 6. TGA and dTGA thermogram of the sample: (a) cellulose, (b) hydrogel.

To investigate the thermal resistance of the hydrogel, we used Hydrogel-2 for the characterization. Figure 6 shows the TGA and dTGA thermograms of the cellulose and the hydrogel. The single-step loss of weight was found for the cellulose. The maximum rate of weight loss was found at 320 °C as observed in dTGA thermogram of the cellulose. This observation might be attributed to the decomposition of the cellulose to form carbon and water. The weight loss of the cellulose was due to the evaporation of water and sublimation of carbon. At the final temperature recorded in the experiment, the remaining weight of the cellulose was about 7 %.

In the case of the hydrogel, the two-step loss of weight was observed. There were two values of T_{max} observed at 265 and 392 °C as seen in dTGA thermogram of the hydrogel. These may be associated to weight loss caused by the evaporation of absorbed water in the hydrogel and the decomposition of the hydrogel, respectively. The decomposition of the hydrogel occurred at higher temperature compared to that of the cellulose. Furthermore, at the final temperature recorded in the experiment, the remaining weight of the hydrogel was about 28 %,.

which is four times as high as that of the cellulose. This evidence may indicate that the hydrogel was better at thermal resistance than the cellulose.

4. CONCLUSIONS

The hydrogel was successfully prepared using the cellulose extracted from sugarcane bagasse. The suitable for the preparation was determined, which was the initiator amount of 1.0 g APS/kg cellulose, the monomer amount of 1.6 g AA/kg cellulose and the crosslinking agent amount of 0.2 g MBA/kg cellulose. The obtained hydrogel exhibited a high swelling degree in acid, neutral and basic media. This may suggest that the hydrogel could absorb both anions and cations in aqueous media. The Young's modulus, stress at break, elongation at break and Charpy impact of the hydrogel prepared in the suitable condition was 1.3 MPa, 5.2 MPa, 89.0 % and 7.2 kJ/m², respectively. The thermal resistance of the hydrogel was better than that of the cellulose. These properties of the hydrogel from sugarcane bagasse indicated that the material is suitable for the preparation of electrolyte membrane in battery.

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CRedit authorship contribution statement. Nguyen Thu Ha: Investigation, Funding acquisition, Supervision. Tran Ngoc Anh: Methodology. Nguyen Thi Quyen: Formal analysis. Vu Anh Duc: Formal analysis. Hoang Thu Hang: Formal analysis. Do Thu Ha: Formal analysis. Nguyen Ngoc Mai: Methodology.

Declaration of competing interest. The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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