

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, ammonium persulfate as an initiator, acrylic acid as a monomer, and N,N'-methylenebisacrylamide as a crosslinking agent. The characterization of structure and properties of the hydrogels were carried out through Fourier-transform Infrared spectroscopy, Focused Ion Beam Scanning Electron Microscopy, and the determination of Young's modulus, tensile strength, elongation at break and Charpy impact, swelling degree and Thermogravimetric Analysis. The suitable conditions for the preparation were found to be 1.0 g ammonium persulfate/kg cellulose, 1.6 g acrylic acid/kg cellulose and 0.2 g N,N'-methylenebisacrylamide/kg cellulose. It was found that both anions and cations can be absorbed into the crosslinked structure of the hydrogel. The Young's modulus, tensile strength, elongation at break, and Charpy impact of the hydrogel prepared under the suitable conditions were 1.3 MPa, 5.2 MPa, 89.0 % and 7.2 kJ/m², respectively. The hydrogel has better thermal resistance than the cellulose. The results show that the hydrogel obtained may be suitable for use as an electrolyte membrane in batteries.

Keywords: hydrogel, cellulose, sugarcane bagasse.

Classification numbers: 2.3.1, 2.3.2, 2.5.2.

1. INTRODUCTION

Hydrogels are defined as hydrophilic polymers with a 3D-network structure. 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, hydrogel has a great opportunity for applications in drug delivery vehicles, electrolyte membrane, tissue engineering, reverse osmosis membrane and so forth [1, 2].

Cellulose is the most abundant natural polymer on Earth. It has been shown that cellulose has interesting properties and is promising as a cheap and green material to form hydrogel. Ammonium persulfate, acrylic acid, and acrylamide are usually used in the preparation of hydrogels from cellulose. In the first step, the radicals are generated by the reaction of ammonium persulfate (APS) initiator and cellulose. In the second step, graft-copolymerization of acrylic acid

on the generated radicals occurs. In the third step, acrylamide is added to form crosslinks in hydrogel [3, 4]. The schematic diagram of hydrogel preparation based on cellulose, AA, APS, and N,N'-methylenebisacrylamide (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 result in the different properties of cellulose hydrogels. Research into the preparation and characterization of hydrogels from various cellulose resources still leaves many unanswered questions.

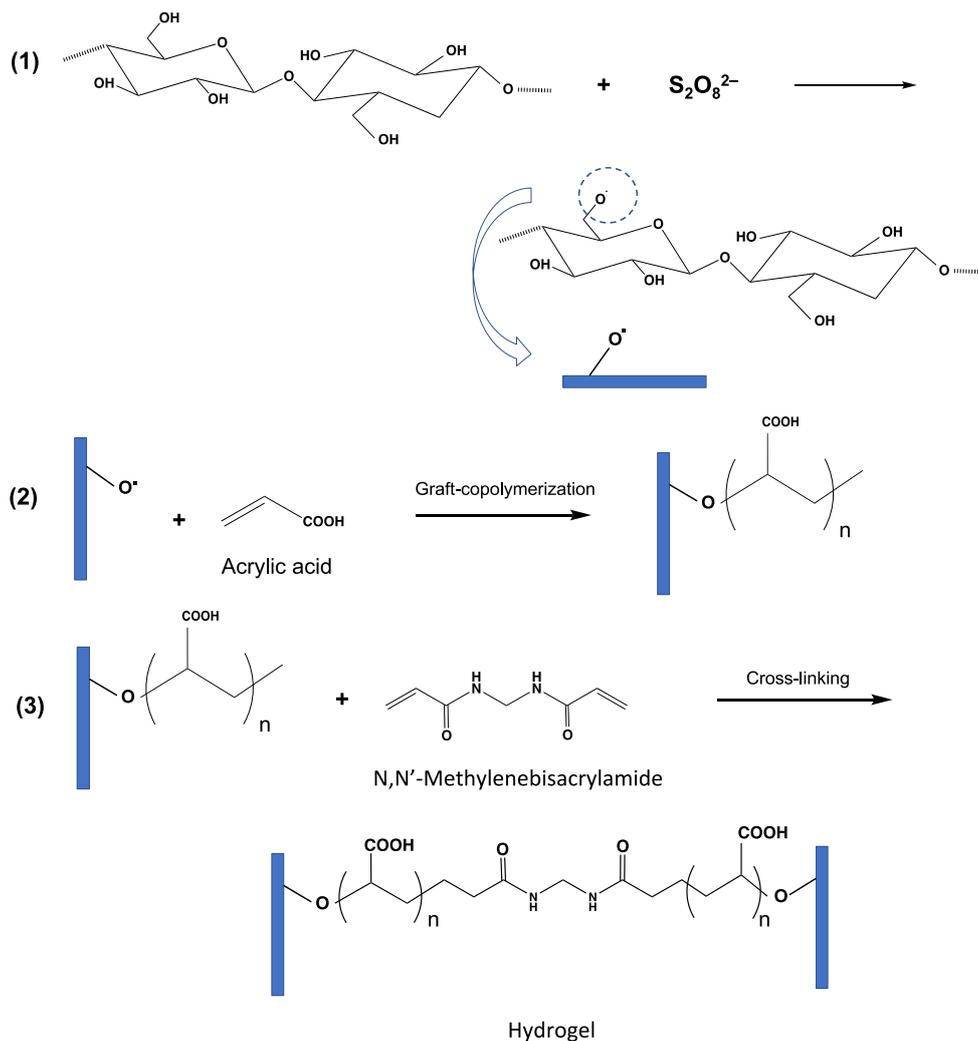


Figure 1. Schematic diagram of cellulose-based hydrogel preparation [4].

Sugarcane bagasse is a by-product of the sucrose production. The cellulose content in sugarcane bagasse is about 40 %, with the remainder being hemicellulose and lignin [9]. In Viet Nam, there is a large amount of sugarcane bagasse, but its treatment has not received appropriate attention. When sugarcane bagasse is used to prepare hydrogel, the obtained hydrogel is a green material, and the preparation procedure may provide a strategy to increase the value of sugarcane

bagasse. The preparation of hydrogel based on sugarcane bagasse has been reported in the literature [3, 10, 11]. For instance, Mondal and co-workers used sugarcane bagasse containing cellulose, hemicellulose, lignin, fatty and waxy materials, and ash to produce hydrogel. In the research of Chia, Tan, Ying Huang, Chiang ChanWei Wong [11], carboxymethyl cellulose prepared from sugarcane bagasse was used to prepare hydrogel. In those works, the conditions for the preparation and structural characterization were established. However, the properties which are useful for practical applications were not thoroughly characterized. Therefore, it is necessary to investigate the preparation procedure and properties of cellulose hydrogel from sugarcane bagasse to find 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 conditions for preparing 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, tensile strength and elongation at break and Charpy impact were studied. The thermal resistance and swelling degree of hydrogel were also investigated. The results showed that the hydrogel obtained may be suitable for use as an electrolyte membrane in batteries.

2. MATERIALS AND METHODS

2.1. Materials

Sugarcane bagasse was gathered from sugarcane tree *Saccharum officinarum* L. cultivated in the North of Viet Nam during the 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 work [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 successively in 4 % HCl and 10 % NaOH solutions. After that, the sample was soaked in 4 % NaClO for 1 hour at room temperature, followed by washing with distilled water and filtering to obtain cellulose. The cellulose was dried at 50 °C under reduced pressure for one week.

2.2.2. Preparation of hydrogel from cellulose

Cellulose was dispersed in a 4 M NaOH 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 cast into a film. The film was preserved in a desiccator before characterization.

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

Table 1. Conditions for preparing 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

Fourier transform infrared spectroscopy (FT-IR) spectra of the samples were performed in the attenuated total reflection mode (ATR) with a resolution of 4 cm^{-1} in the region of $400\text{--}4000\text{ cm}^{-1}$ and 64 scans using a JASCO FT-IR 4600 spectrometer.

The surfaces of the samples, coated with a gold layer by sputtering, were observed through a scanning electron microscope NovananoSEM at an accelerated voltage of 5 kV. FIB-SEM was used to observe the morphology of hydrogel, using NovananoSEM at an accelerated 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, tensile strength 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/min. Measurements were conducted at $25\text{ }^{\circ}\text{C}$ and repeated 3 times for each sample.

Charpy impact tests on hydrogel samples were carried out using a Charpy impact tester (Toyoseiki). Measurements were performed at $25\text{ }^{\circ}\text{C}$ and repeated 3 times for each sample.

The swelling degree (SD) of hydrogel films in acidic, neutral, and basic media was investigated at $25\text{ }^{\circ}\text{C}$. The hydrogel films were cut into $2\text{ cm} \times 2\text{ cm}$ pieces, weighed and soaked in HCl solution ($\text{pH} = 4$), deionized water ($\text{pH} = 7$), NaOH solution ($\text{pH} = 10$) for 168 hours. After that, the samples were taken out, wiped with tissue paper to remove any liquid from the surface, and weighed. The SD was calculated using the following equation:

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

where W_f is the final weight of the swollen sample and W_i is the initial weight of the sample.

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

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

3. RESULTS AND DISCUSSION

3.1. FT-IR spectrum

Figure 2 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 4(a), the characteristic signals of cellulose appeared. The broad peak at 3340 cm^{-1} was assigned to the stretching vibration of the hydroxyl group (OH) in polysaccharide [16]. The signal at 2904 cm^{-1} was the characteristic signal of CH stretching vibration in the chain of polysaccharides [17]. The signal appearing at 1628 cm^{-1} was assigned to the bending vibration 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 presence of characteristic signals in the FT-IR spectrum of the sample confirmed the structure of the cellulose separated from sugarcane bagasse. The FT-IR spectrum of the cellulose-based hydrogel is shown in Figure 4(b). The signal from 2924 cm^{-1} to 3300 cm^{-1} assigned to the characteristic signal of OH was broadened [16], which may be due to the presence of absorbed water in the hydrogel. This signal may cover other characteristic signals in this region.

The presence of signals at 1700 cm^{-1} and 1443 cm^{-1} is noteworthy. The signal at 1700 cm^{-1} may be attributed to the stretching vibration of the C=O group of the acrylic acid unit in the crosslink of the hydrogel [19]. The signal at 1443 cm^{-1} may be associated with the C–H bending vibration in the CH₃ group of acrylamide at the crosslinking junction [19]. The appearance of these signals may confirm that crosslinks formed by AA and MBA are present in the hydrogel.

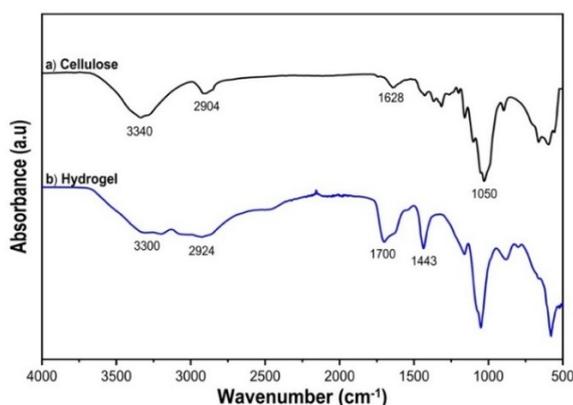


Figure 2. FT-IR spectrum of sample: (a) cellulose, (b) hydrogel.

3.2. Morphology

The morphology of Hydrogel-2 was observed through SEM and FIB-SEM images as shown in Figure 3. Other hydrogels showed similar morphology, hence their SEM and FIB-SEM images are not shown here. The SEM image indicated that the surface of the hydrogel was not homogeneous with many drains, which can afford the adhesion of liquids to the hydrogel's

surface. In the FIB-SEM image, the voids inside the hydrogel were clearly visible. These voids may provide space for water or electrolyte solution. When water or electrolyte solution resides in the voids, the hydrogen bonding between the cellulose chains and water may firm the water inside the hydrogel structure. Therefore, the hydrogel prepared in this work may be a suitable material for electrolyte membranes used in batteries.

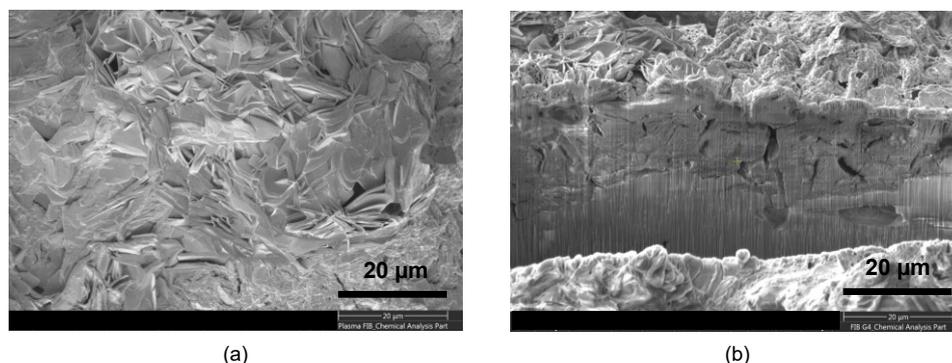


Figure 3. SEM image of sample (magnification x2500): (a) SEM image of surface of the hydrogel, (b) FIB-SEM image of the hydrogel.

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 presents swelling degrees of hydrogels in acidic, neutral, and basic media. It is clear that the swelling degree of the hydrogels was much higher than that of the cellulose. The swelling degree in basic medium was the highest, followed by that in acidic medium. The swelling degree in neutral medium was the lowest. This phenomenon suggested that the hydrogel could absorb OH^- and H^+ into its structure, and the OH^- absorption was better than the H^+ absorption. This also implies that the hydrogel prepared in this work can absorb both anions and cations, allowing the hydrogel to become a medium for drug delivery, ion transportation and so forth.

As comparing the swelling degrees of hydrogels prepared with various contents 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 by the fact that an increase in APS amount would cause an increase in 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 amounts of APS, AA and MBA were lower than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the swelling degree of samples decreased sharply as seen for Hydrogel-3, Hydrogel-4 and Hydrogel-6. On the other hand, when the amounts of APS, AA and MBA were higher than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the swelling degree of samples slightly increased as seen for Hydrogel-1, Hydrogel-5 and 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 the literature [15]. The remaining AA and MBA might not support the formation of crosslinked structure in the hydrogel. Therefore, it can be concluded that 1.0 g APS/kg cellulose, 1.6 g AA/kg cellulose and 0.2 g MBA/kg cellulose are suitable amounts for preparing hydrogels with good swelling properties.

3.4. Mechanical properties

Table 3. Mechanical properties of the samples.

Sample	Young's modulus (MPa)	Tensile strength (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

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

The mechanical properties of the hydrogel samples in terms of Young's modulus, tensile strength, 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 they are higher than those of Hydrogel-3, Hydrogel-4, Hydrogel-5, and Hydrogel-6. It was found that when the amounts of APS, AA and MBA were 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 amounts of APS, AA and MBA were lower than 1.0, 1.6 and 0.2 g/kg cellulose, respectively, the mechanical properties were significantly reduced. It can be explained that these contents of APS, AA and MBA are suitable for the reaction that generates radicals from cellulose and forms crosslinks between cellulose chains. When the contents of APS, AA and MBA were lower, the crosslink density of the hydrogel was probably lower. These crosslinks enhanced the mechanical properties of the hydrogel. On the other hand, when the contents of APS, AA and MBA were higher than 1.0, 1.6 and 0.2 g/kg cellulose, the crosslink density might be saturated, leading to stability in mechanical properties. This phenomenon suggested that the suitable amounts of APS, AA, and MBA for

preparing hydrogels were 1.0, 1.6, and 0.2 g/kg cellulose. The mechanical properties of Hydrogel-1, Hydrogel-2 and Hydrogel-7 were quite similar, while the amounts of APS, AA and MBA for preparing Hydrogel-2 was the smallest. The results regarding mechanical properties were in good agreement with those regarding swelling. We used Hydrogel-2 for further investigation.

3.5. Thermal resistance

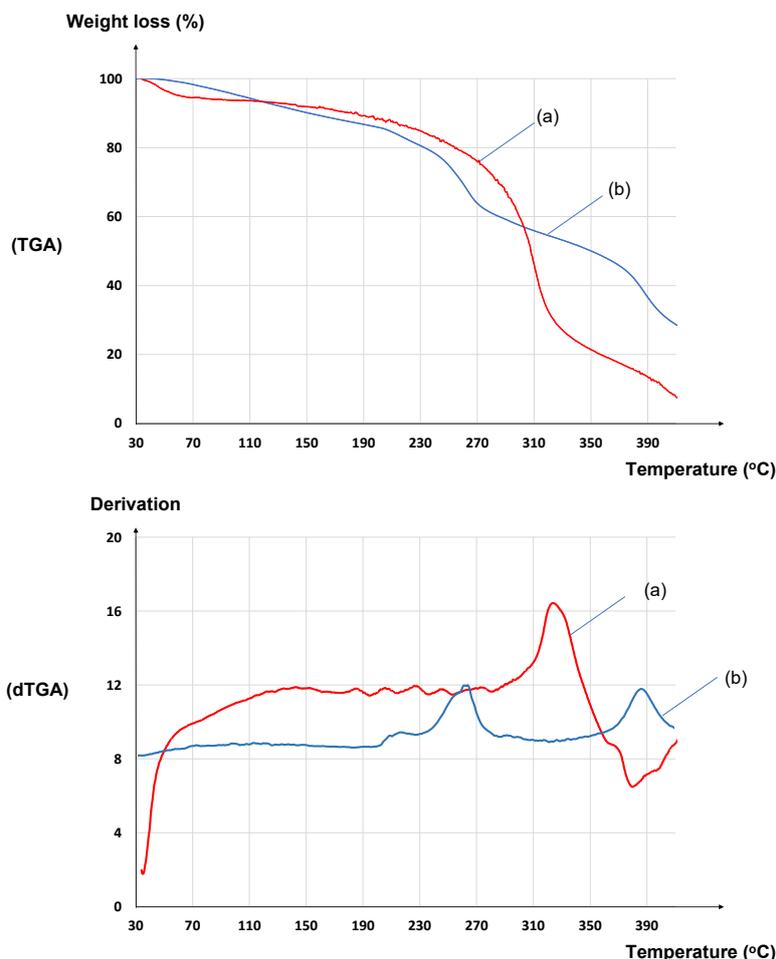


Figure 4. TGA and dTGA thermograms of the samples: (a) cellulose, (b) hydrogel.

To investigate the thermal resistance of the hydrogel, we used Hydrogel-2 for the characterization. Figure 4 shows the thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (dTGA) thermogram 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 temperature at maximum weight loss rate (T_{max}) observed at 265 and 392 °C as seen in dTGA

thermogram of the hydrogel. These may be associated with the 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 a 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 %, four times that of the cellulose. This evidence may indicate that the hydrogel has better thermal resistance than the cellulose.

4. CONCLUSIONS

The hydrogel was successfully prepared using the cellulose extracted from sugarcane bagasse. The suitable conditions for the preparation were determined, namely an initiator amount of 1.0 g APS/kg cellulose, a monomer amount of 1.6 g AA/kg cellulose, and a crosslinking agent amount of 0.2 g MBA/kg cellulose. The hydrogel obtained 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, tensile strength, elongation at break and Charpy impact of the hydrogel prepared under the suitable conditions were 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 membranes in batteries.

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