Egypt. J. Chem. **Vol. 66**, No. 9 pp. 11 - 19 (2023)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Synthesis of Hydrogels from Banana Stem Cellulose (*Musa paradisiaca* L.) with Chitosan and Ethylenediaminetetraacetic Acid

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Abstract

Research on the preparation of cellulose hydrogel with chitosan and ethylenediaminetetraacetic acid (EDTA) has been successfully carried out. This study aims to determine the cellulose content in banana stem and the characteristics as well asthe effect of the composition chitosan and ethylenediamintetrasetic acid (EDTA) in synthesis cellulose hydrogels on the swelling value. The method used in cellulose hydrogel synthesis is the freeze-thaw method. The characterization of hydrogel was carried out using Fourier Transform Infrared spectroscopy (FTIR), Scanning Electron Microscopes (SEM) and X-Ray Diffraction (XRD). FTIR analysis on cellulose hydrogel showed specific functional groups O-H (3331), C-H (2899), C=O (1616), CH2 (1420), C-O (1323) and C-N (1037). SEM analysis shows a wavy surface, forming a slightly hollow aggregate that affects the degree of development. XRD analysis showed that the cellulose hydrogel was amorphous. The highest deswelling value was obtained in the cellulose hydrogel (H1) of 221.52%.

Keywords: *Banana Stem; Cellulose; Freeze-Thaw and Cellulose Hydrogel*

1. Introduction

Indonesia is known as a producer of bananas in the world. One of the largest banana producers in Indonesia is in the South Sulawesi area at 142,492 tons/ha/year[1]. The number of banana plants owned can produce a relatively high amount of waste[2]. Banana stem waste is one of the primary alternative raw materials for manufacturing cellulose. Banana stem waste contains 46% cellulose, 9% lignin and 38.54% hemicellulose[3]. The high cellulose content in banana stem can be used as raw material for hydrogel production.

Hydrogel is a network of hydrophilic polymer chains that can absorb up to thousands of times their dry weight[4,5]. This makes hydrogels widely used in the pharmaceutical, forestry, agricultural and cosmetic fields. Hydrogels can be synthesized from synthetic polymers such as polycaprolate, polyvinyl pyrrolidone (PVP), polyethylene glycol (PEG) and polyvinyl alcohol (PVA) and natural polymers such as chitin, cellulose, chitosan, starch, gums and alginates[6]. In addition to using hydrogel polymers, plasticizers are also used to reduce brittleness,

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Received date 06 July 2022; revised date 08 September 2022; accepted date 14 January 2023.
DOI: 10.21608/EJCHEM.2023.149022.6451

increase flexibility and increase water resistance properties [7].

Research conducted by Abdulhameed et al. (2020) using cellulose from rice husks using the crosslinking method in the manufacture of hydrogels obtained a high absorption value of 1162%[8]. In addition, research conducted by Ritonga*et al.* (2019) synthesized hydrogel from ethylenediaminetetraacetic acid (EDTA) and chitosan, which functioned to improve the performance of soybean plants[9].

Currently, crosslinking methods have been developed in the manufacture of hydrogels, one of which is the freeze-thaw method or freeze-melt technique, which can be used to induce crosslinking between polymers[10,11]. This method is widely used because it is safer, cheaper and environmentally friendly[12]. This study aims to determine the cellulose content in banana stem and the ability of the resulting hydrogel to swelling and retain water in it, which is then analyzed using instruments Fourier Transform Infrared Spectroscopy (FTIR), X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM).

2. Experimental

Materials and Equipment

The materials used aquadest (H_2O), acetic acid (CH_3COOH) (Merck), banana stem (*Musa paradisiaca* L.),ethylenediaminetetraacetic acid ($C_{10}H_{16}N_2O_8$), hydrogen peroxide (H_2O_2) (Merck), PH paper, Whatman 42 filter paper, chitosan $(C_6H_{11}NO_4)n$, sodium hydroxide (NaOH) (Merck), sodium sulfite (Na_2SO_3) (Merck) and apple extract. The equipment used Fourier Transform InfraredSpectroscopy (FTIR), X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM). freezer, analytical balance, hotplate stirrer, sieve shaker, buchner funnel and hammer mill.

Methods

Preparation of Banana Stem

Banana stem are taken in the BontoBangun area, South Sulawesi, Indonesia which is then washed thoroughly and cut to a size of 5 cm and then dried in the sun for four days. The dried banana stem were mashed with a hammer mill and then sieved with a 100 mesh sieve.

Isolation of Cellulose from Banana Stem

Prepared three beaker glasses, then weighed 30 grams of banana stem powder each, then added 500 mL of NaOH 2%. Next, it was heated for 1 hour at 100°C. The mixture was then filtered and washed with distilled water until the pH was neutral and dried in an oven at 105°C. The next step is delignification using Na_2SO_3 18%, 20%, and 22% in as much as 100 mL. Cellulose is heated for 2 hours at a temperature of 105°C. The cellulose obtained was then filtered and washed with distilled water until the pH was neutral. The resulting cellulose is then dried at 100°C. The next step is bleaching using 300 mL of H_2O_2 3% for 2 hours at 60°C. The cellulose obtained was filtered and washed with distilled water until clean. Furthermore, the cellulose was dried at a temperature of 50°C for 24 hours, and then the final weight was weighed. The yield of cellulose can be calculated with the formula:

$$\%\text{yield} = \frac{\text{weight of yield cellulose}}{\text{weight of banana stem powder}} \times 100\% \qquad (1)$$

Synthesis Cellulose Hydrogel

Weighed 2 grams of cellulose then added chitosan and ethylenediaminetetraacetic acid (EDTA) in a ratio of 2.25:0.75 (H1), 2:0.5 (H2) and 1.75:0.75 (H3). The composition of the hydrogel is presented in Table 1 and dissolved with 50 mL of 0.6 M acetic acid. The mixture was then stirred using a magnetic stirrer for 15 minutes. Added apple extract as much as 4 mL. After that, NaOH 2 M was dropped into the solution to form a gel. The gel formed was put into a mold and frozen at -20°C for 18 hours, then placed at room temperature for 6 hours. The treatment was carried out for 4 cycles, then the hydrogel was baked in an oven at 50°C for 6 hours. For an illustration of making cellulose hydrogel was presented in Figure 1.

Table 1. Variation Cellulose Hydrogel

Cellulose (gram)	Chitosan (gram)	EDTA (gram)	Code
2	2.25	0.25	H1
	2	0.5	H2
	1.75	0.75	H3

Swelling Test

The initial weight (w_d) of the cellulose hydrogel was then soaked in distilled water for 24 hours. After

that, the excess water on the hydrogel surface was removed with filter paper and the final weight (w_s) was weighed. The measurement of the percent water swelling in the hydrogel can be determined by the equation:

$$\%S = \frac{w_s - w_d}{w_d} \times 100\% \qquad (2)$$

Deswelling Test

The dried and weighed cellulose hydrogel was put in water for 6 hours, then put into a nylon bag and hung in a room at a temperature for 15 minutes and then weighed (m_e). Next, the nylon bag containing the hydrogel was put into a water bath at a constant temperature of 60 °C for 10 minutes, then removed and hung in the room for 15 minutes, and then weighed (m_t). The water retention in the gel is calculated as Wr (%) with the equation:

$$\%Wr = \frac{m_t}{m_e} \times 100\% \qquad (3)$$

Characterization of Cellulose Hydrogel

The functional group of banana stem, cellulose banana stem and cellulose hydrogel was investigated using the Fourier Transform Infrared spectroscopy (FTIR). The morphology of cellulose hydrogel was examined by Scanning Electron Microscope (SEM), the determine shape of the crystallinity and size of the crystal lattice in a cellulose hydrogel was examined using the X-Ray Diffraction (XRD).

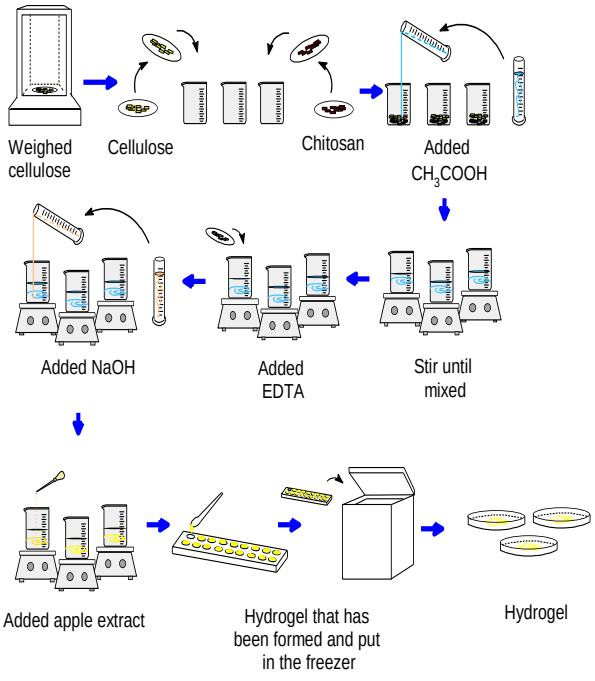


Figure 1. Ilustration of SynthesisCellulose Hydrogel

3. Result and Discussion

Isolation of Banana Stem Cellulose

The results of the isolation of cellulose from the banana stem in the form of a fine powder, yellowish-white, odorless and insoluble in water. The results of cellulose content obtained from variations in Na_2SO_3 concentration are presented in Table 2.

Table 2. Variation Cellulose

Concentration of Na_2SO_3 (%)	First Weight (gram)	Final Weight (gram)	The Yield of Cellulose (%)
18	30	6.6875	22.292
20	30	9.1422	30.474
22	30	7.7854	25.951

Table 2 presented that the highest amount of cellulose content was obtained at 20% Na2SO3 concentration, which was 30.474%. The data above, it is following several studies that have been conducted by Lismeri, 2019 which stated that the highest cellulose content was at a concentration of 20%, which was 55.59 %[13] and research conducted by Sumada*et al.* (2011) obtained α-cellulose content of 88,90%. This indicates that the optimum concentration in the use of Na_2SO_3 in the delignification process is 20% because at that concentration, the soluble lignin content increases, increasing the cellulose content of the sample[14]. Based on the value of cellulose content obtained, what is used in the manufacture of hydrogel is banana stem cellulose with a concentration of 20%.

Synthesis Cellulose Hydrogel

The process of synthesizing cellulose hydrogels begins with the addition of chitosan, which aims to increase hydrogel absorption and surface area and improve the appearance of the hydrogel. The addition of EDTA serves as a chemical crosslinking agent and a link between one polymer and another. The addition of NaOH is expected to accelerate gel formation. Then the addition of apple extract as a plasticizer is helpful in increasing the flexibility and elasticity of the polymer made so that the resulting hydrogel is not quickly broken when the solution is absorbed. After that, a freeze-thaw process was carried out, which functioned to induce crosslinking

between polymers due to the resulting hydrogel having a stable structure (Fig. 2).

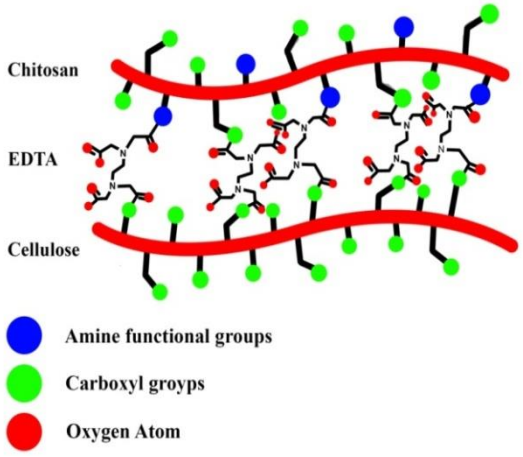


Figure 2. Prediction of Cellulose Hydrogel Formation

Table 4.Swelling Test and Deswelling Test of Cellulose Hydrogel

Hydrogel	Swelling Test			Deswelling Test		
	First Weight (Gram)	Final Weight (Gram)	Swelling (%)	Weight before Heating (gram)	Weight after Heating (gram)	Deswelling (%)
H1	0.5106	1.6417	221.52	0.4702	0.4689	99.72
H2	0.3581	1.1079	209.38	0.4184	0.3924	93.78
H3	0.3081	0.7614	147.12	0.2916	0.2487	85.28

Swelling and Deswelling Test

The results indicate that the cellulose hydrogel with the highest water absorption is the hydrogel variation H1 of 221.52% and the lowest hydrogel with the variation of H3 is 147.12%. From these data, it is known that the more crosslinkers, namely EDTA, used, the lower the hydrogel's ability to absorb[9]. This is because crosslinking at higher concentrations can create crosslink density. The hydrogel can be absorbed with a smooth surface, slightly hard but flexible because it is influenced by adding apple extract as a plasticizer which increases the elasticity of the hydrogel[15]. This is evidenced by the SEM results obtained. Hydrogel has an irregular surface, and there are lumps. This is due to the presence of anionic groups, so the bonds between molecules become less tight. As a result, there is more space to accommodate the liquid so that the absorption capacity becomes greater.

Hydrogels that have absorbed water can suffer structural damage if there are extreme environmental changes, such as high-temperature changes. This can cause the hydrogel structure to break down. From Table 4 above, the most significant water resistance at high temperatures is found in hydrogels with H1 variations of 99.72% and the lowest hydrogels with H3 variations of 85.28%. This shows that the more crosslinks added, the lower water resistance of the hydrogel. When the temperature is increased, the longer polymer chains will close tightly, making it difficult for water to enter the hydrogel polymer [16]. The swelling and deswelling test values for cellulose hydrogel are presented in Table 4 and the Comparison Graph of cellulose hydrogel composition to the degree of swelling and deswelling can be seen in Fig 3. Based on the swelling and deswelling values obtained, the next step in the characterization process is hydrogel H1.

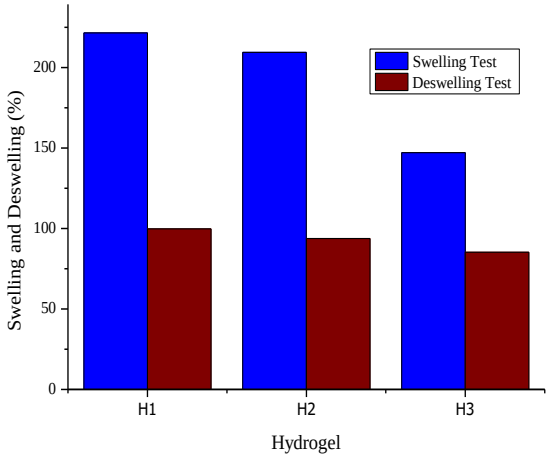


Figure 3. Comparison Graph of The Composition of The Hydrogel to The Degree of Swelling and Deswelling

Characterization of Banana Stem, Cellulose Banana Stem and Cellulose Hydrogel

The FTIR readings on a banana stem (Fig. 3a). Absorption peak at 1238 cm^{-1} is the vibration of the phenolic hydroxyl group of lignin[17]. In addition, the absorption region of 1739 cm-1 was associated with the C=O stretching of the acetyl groups and hemicellulose esters or the carbonyl group bonds of the lignin ester units [18]. The absorption peak was not found in the FTIR spectrum of banana stem cellulose (Fig. 3b). This proves that the process of removing non-cellulose components during the delignification and bleaching process is successful[19].

The FTIR spectrum of banana stem cellulose (Fig. 3b). The absorption peak at wave number 3331 cm^{-1} is associated with the O-H stretching vibration andthe absorption peak in the 2906 cm^{-1} region is the C-H symmetric vibration of the CH2 group[20–22]. This is reinforced by the presence of a peak in the area of 1431 cm^{-1}, which is a -CH2-(C6)- bending[23]. The appearance of an absorption peak in the 1641 cm^{-1} region indicates there is H_2O content in the cellulose[24]. The absorption peaks of the 1373 cm^{-1} and 1319 cm^{-1} regions show C-O or C-H symmetric stretching in cellulose polysaccharides and C-H bending[17]. The absorption peaks of 1162 cm^{-1}, 1057 cm^{-1}, and 894 cm^{-1} are characteristic of C-O-C glycosidic bond vibration, C-O bond stretch, and glycosidic linkage of glucose units in cellulose[25,26]. The main constituent components of cellulose are the OH, CH, and CO groups.

The FTIR spectrum of cellulose hydrogel H1 (Fig. 3c) when compared with the FTIR spectrum of cellulose, it showed some significant changes. These changes were caused by the presence of chitosan and EDTA in the cellulose hydrogel H1. This is reinforced by the shift of the vibrational peak of the O-H group 3331 cm^{-1} to a higher wavelength region of 3462 cm^{-1}[9,10]. The absorption peak in the 2899 cm^{-1} region was associated with the C-H symmetric stretch of the saccharide ring in chitosan and cellulose[27]. The absorption peak in the 1420 cm^{-1} region is associated with the bending vibration of CH2[28]. The absorption peak is at 1323 cm^{-1} which is associated with the C-O stretching vibration[29]. The absorption peak in the 1037 cm^{-1} region is associated with the C-N stretching vibration[9]. The occurrence of crosslinking between cellulose-chitosan-EDTA was identified at the absorption peak in the 1616 cm^{-1} (H1) region, which was associated with the C=O stretching vibration[9,27]. The results of characterization using FTIR showed the success of cellulose-chitosan synthesis using EDTA to form a hydrogel.

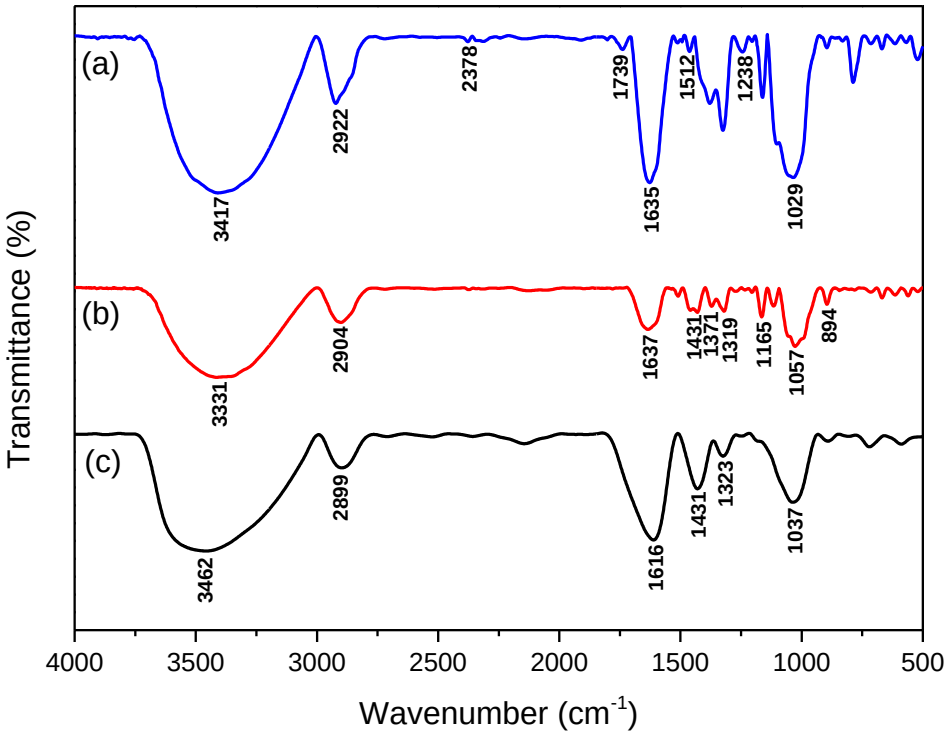


Figure 4. FTIR Spectrum of a. Banana Stem; b. Cellulose Banana Stem and c. Cellulose Hydrogel

Table 3. FTIR Absorption Peak of Banana Stem, Cellulose Banana Stem andCellulose Hydrogel

Functional Group	Wavenumber ($cm^{-1)}$			Ref
	Banana Stem	Cellulose Banana Stem	Hydrogel	
OH	3417, 1238	3331	3462	[9,10,17,20–22,30]
CH	2922	2904	2899	[20–22,27,31]
CH bending	-	1319	-	[17]
CH_2 bending	-	1431	1420	[23,28]
CO(C-O-C)	-	1165, 1057	-	[25,26]
C=O	1739	-	1616	[9,18,27]
C=C	1512	-	-	[30,31]
C-N	-	-	1037	[9]
C-Obonding	-	894	-	[25,26]
C-O streching	-	-	1323	[29]
C-O	-	1371	-	[17]
H_2O	1635	1637	-	[24]

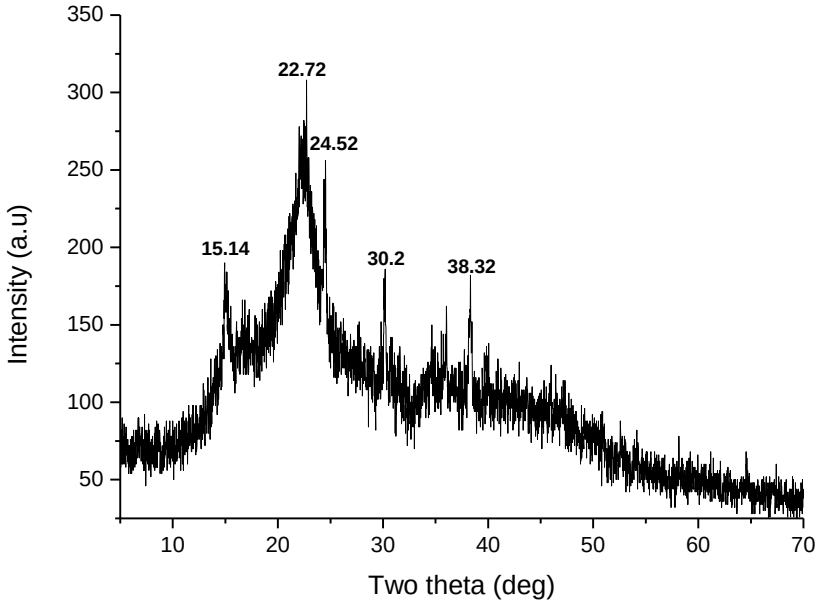
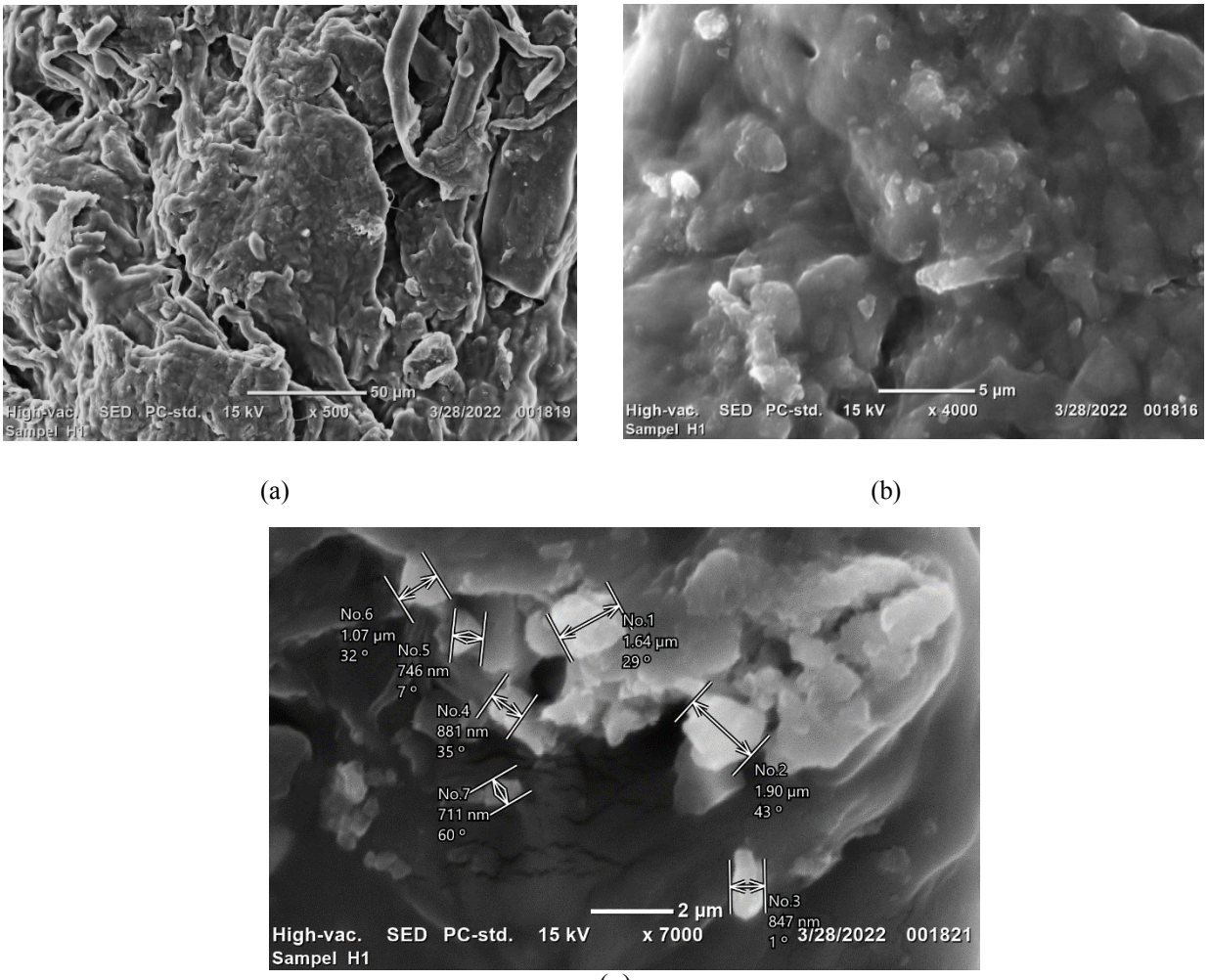


Figure 5.Diffractogram of Cellulose Hydrogel

Figure 5 shows a diffractogram of cellulose hydrogel. The crystalline polymer material will show the diffraction peak as a sharp peak at a certain angle, while the amorphous part will appear as a broad peak. The hydrogel diffractogram showed vast peaks with diffraction angles (2θ), namely 17.31º, 22.46º, 30.18º, 38.77º, and 36.04º with an average crystal size of 11.35 nm. Therefore, the obtained hydrogel has amorphous properties.

The results of the analysis using SEM with magnifications of 500 and 7000 on the cellulose hydrogel are shown in Figures 3a and 3b. Figures 3a and 3b show that the cellulose hydrogel has a wavy or uneven surface, forming slightly hollow aggregates so that it can absorb water and there are granules on the hydrogel surface which have been identified as cross-links. Gao *et al*[32] , stated that the adsorption capacity is influenced by the voids contained in a material, namely the larger the cavity, the greater the absorption capacity. The results of the SEM characterization in Figure 3c show that there are several grain sizes obtained in the cellulose hydrogel, namely 1.64 um, 1.90 um, 847 nm, 881 nm, 746 nm, 1.07 um and 711 nm.



(a) (b)

(c)

Figure 6. SEM results of (a) Cellulose Hydrogel 500X, (b) Cellulose Hydrogel 4000X and (c) Cellulose Hydrogel 7000X

4. Conclusion

The results of cellulose isolation from banana stem obtained the highest cellulose content value at 20% Na_2SO_3 concentration of 30.474%. The results of FTIR characterization of cellulose hydrogels showed the presence of a C=O and C-N functional group which indicated the occurrence of cross-linking between cellulose, chitosan and EDTA. while the SEM results showed the presence of hollow lumps on the hydrogel surface which affected the swellling and deswelling values of the hydrogel. The highest swelling and deswelling values were obtained in the H1 hydrogel variation is 221.52% and 99.72%.

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