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Preparation and Characterization of Superabsorbents Based on Durian Seed Starch (*Durio zibethinus*) and Carboxymethyl Cellulose Using Citric Acid Crosslinking

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ABSTRACT

Superabsorbents are materials that can absorb a liquid beyond its initial volume. Natural polymer-based superabsorbents can be obtained by combining CMC and durian seed starch. The combination of these two materials was cross-linked using varying amounts of citric acid: 0.0 grams, 0.2 grams, 0.4 grams, and 0.6 grams. The resulting superabsorbent has a cross-linking degree of about 41%, shows the presence of OH and CH groups, has a rough and dense surface, and a maximum water absorption capacity of 4130% with a water absorption rate of 41.3 g/g. Durian seed starch has potential as a superabsorbent material.

Keywords: CMC, Cross-linking, Modified Durian Strach, Superabsorbent

ABSTRAK

Superabsorben adalah material yang dapat mengadsorbsi suatu cairan melebih volume awalnya. Superabsorben berbasis polimer alam dapat diperoleh dengan kombinasi CMC dan Pati Biji Durian. Kombinasi kedua bahan tersebut diikat silang menggunakan variasi asam sitrat sebesar 0,0 gram, 0,2 gram, 0,4 gram dan 0,6 gram. Superabsorben yang dihasilkan, memiliki derajat ikat silang sekitar 41%, memperlihatkan adanya gugus –OH dan –CH, memiliki permukaan kasar dan rapat, dan daya serap air maksimum terhitung sebesar 4130% dengan serapan air 41,3 g/g. Pati biji durian memiliki potensi sebagai bahan pembuatan superabsorben.

Kata Kunci : CMC, Ikat Silang, Pati Biji Durian Modifikasi, Superabsorben

1. Introduction

Polymer-based superabsorbents (PS) can be produced from natural polysaccharide derivatives such as starch, collagen, flour, gelatin, cellulose, and chitosan, because of hydrophilic groups and a strong affinity for water [1]. One of the natural polymers that is often used is starch because it is cheap, readily available, and environmentally friendly [2]. Durian (*Durio zibethinus Murr*) is a fruit belonging to the Bombacaceae family, commonly found in tropical regions, and its seed have a starch content of 43.6%. Therefore, it has the potential to be used as a material for making natural superabsorbent polymers [3].

The main groups of starch are hydrophilic groups, such as hydroxyl (-OH) and carboxyl (-COOH) groups, that easily absorb water. When starch is introduced into water, there will be an interaction between the hydrophilic groups of starch and the hydroxyl (-OH) groups of water molecules. However, starch has deficiencies in mechanical and thermal properties [4]. Therefore, starch modification is necessary to address these deficiencies, thereby enhancing the performance of starch-based durian seed superabsorbents.

PS is a three-dimensional polymer chain network with cross-links as its basic structure [5]. Cross-linking is a method that causes superabsorbents to take on a three-dimensional shape and create space to trap water molecules. The method also prevents unlimited swelling of PS due to water absorption [6]. PS has ionic functional groups like carboxylate, carbamide, hydroxyl, amine, imide, and more. In addition, PS is capable of absorbing large amounts of water, salt solutions, and other polar liquids with an absorption rate of 10–1000 times its initial weight and can retain them [7].

2. Materials and Methods

2.1. Equipment

The equipments used in this research include a blender, measuring cup, analytical balance, hotplate stirrer, magnetic stirrer, stand and clamp, three-neck flask, condenser, oven, thermometer, FT-IR spectrophotometer, scanning electron microscope, and sample cup.

2.2. Materials

The materials used in this study include durian seed starch (s), aquadest (aq), $NaIO_4$ (s), ethanol (aq), CMC (s), H_2SO_4 (aq), citric acid(aq).

2.3 Research Procedures

2.3.1 Preparation of Durian Seed Starch

The starch from durian seeds is prepared by washing, peeling, cutting, and re-washing until it is free from slime. Next, the chopped pieces are added with water, then crushed using a blender and filtered using a cheesecloth. The filtrate is then left overnight to produce a precipitate. The precipitate is separated from the filtrate, washed again, and re-precipitated before being dried using an oven. The dry precipitate (the starch) is then ground using a blender. Durian seed starch (DSS) then was analyzed using FT-IR spectrophotometry.

2.3.2 Preparation of Superabsorbent

2 grams of DSS and 1 gram of CMC were dispersed into 100 mL of distilled water and heated it at 100 °C for 1 hour. Next, we added 1 mL of H_2SO_4 and citric acid to the DSS/CMC solution, homogenizing it with a magnetic stirrer. The mixture is then cast using a mold and dried in an oven at 55°C until it becomes a film. The formed DSS/CMC film was subsequently characterized using FTIR and SEM, and its potential as a superabsorbent was tested using water absorption capacity and crosslinking degree.

3. Results and Discussion

3.1 Isolation of Durian Seed Starch

The durian seeds used to produce starch (DSS) come from Kualasimpang. A total of 5 kg of durian seeds yields 738 grams of starch. Figure 1 presents the FT-IR characterization results of DSS, revealing peaks at wave numbers 3266.1 cm^{-1}, 2922.2 cm^{-1}, 2102.2 cm^{-1}, 1640 cm^{-1}, 1341.8 cm^{-1}, and 1148 cm^{-1} [8].

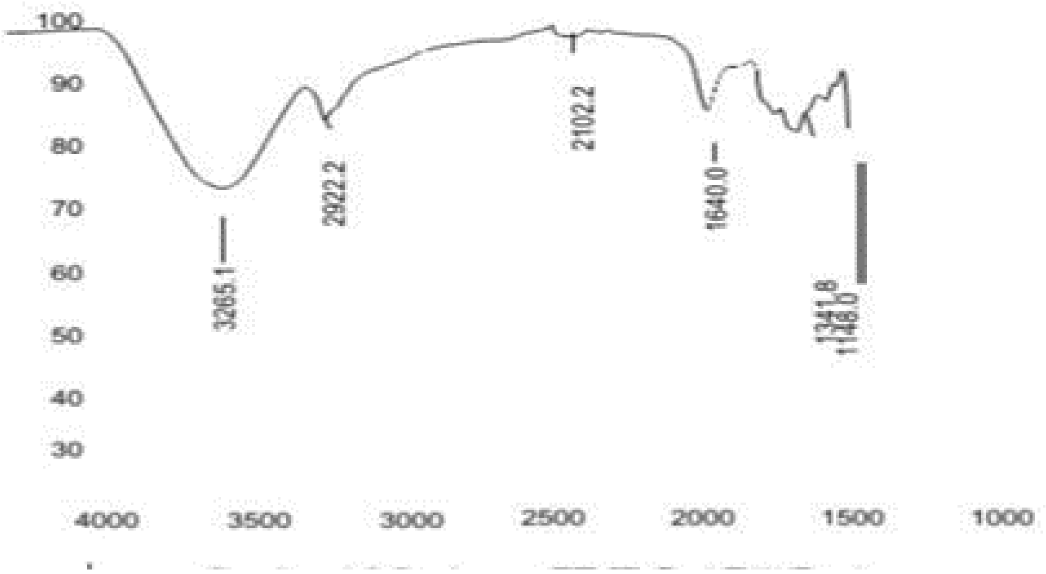

Figure 1. FT-IR spectrum of durian seed starch

3.2 Analysis Results Using FT-IR Spectrophotometer Superabsorbent

Superabsorbent is a mixture of gelatinized starch and CMC suspension. H_2SO_4 was added, the resulting solution is poured and spread on a baking sheet, and dried at 55°C until a film is formed. The obtained results were analyzed using FT-IR spectroscopy. The FT-IR spectrum results show vibrations at wave numbers 3303-3333 cm^{-1}, 2907-2922 cm^{-1}, 1722-1729 cm^{-1}, and 998-1021 cm^{-1} [9].

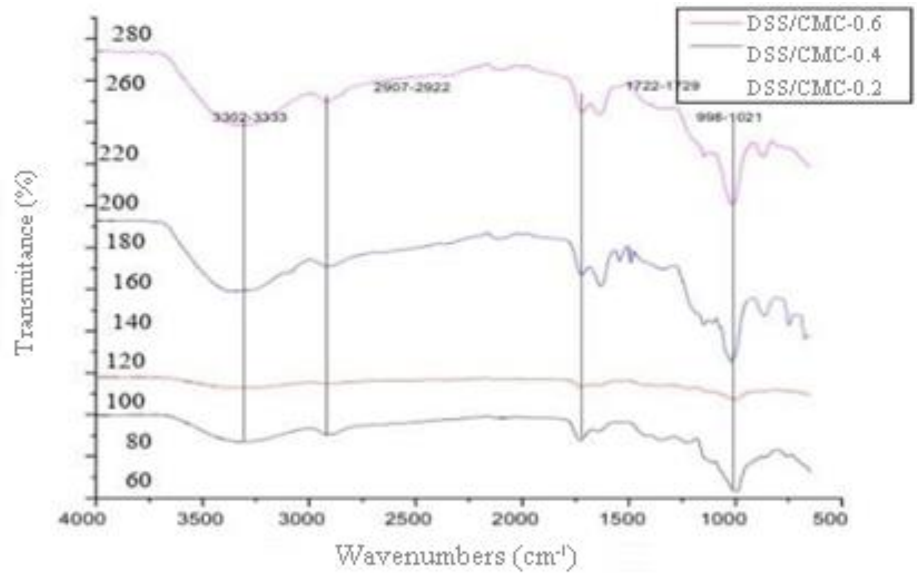

Figure 2. FT-IR spectrum of superabsorbent

3.3 Scanning Electron Microscope Analysis Results

The SEM testing was conducted on Superabsorbent with variations of citric acid at 0.0 grams, 0.2 grams, 0.4 grams, and 0.6 grams, as shown in the following image:

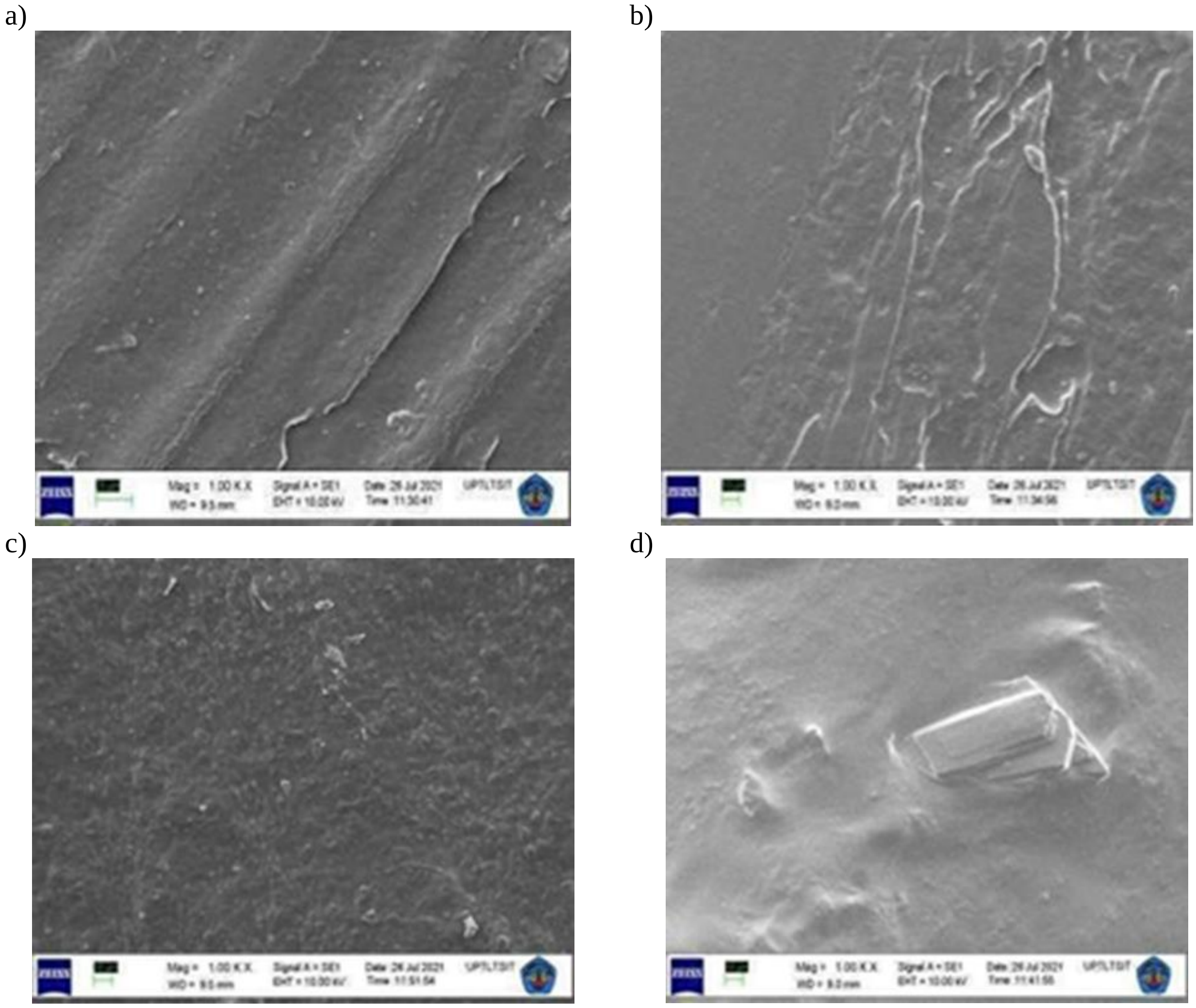

Figure 3. Results of scanning electron microscopy of superabsorbent with citric acid variations

3.4 Results of Determination of Cross-Bound Degree

The following is a table of the results of determining the Cross-Band Degree value, as seen in Table 1.

Table 1. Data value results of determination of cross-bound degree of DSS/CMC

Sample	Absorption Time (hours)	DC (%)
DSS/CMC-0.0	24	30
DSS/CMC-0.2	24	31
DSS/CMC-0.4	24	34
DSS/CMC-0.6	24	41

The crosslinking test results yielded percentages of approximately 30%, 31%, 34%, and 41%. These percentages indicate relatively low values, which is consistent with the water absorption capacity (WAC) percentages. If the crosslinking degree percentage is high, the water absorption result is also high. It's possible that non-sterile experiments led to the low percentage value of the crosslinking degree. This meant that other compounds could soak in and stop citric acid from attaching to the polymer [10].

3.5 Results of Determination of Water Absorption Capacity (WAC) Value

The water absorption capacity based on Table 2 shows the highest value in the superabsorbent using 0.4 grams of citric acid (DSS/CMC-0.4), which is 4130% or 41.3 g/g with an absorption process time of 24 hours. Meanwhile, in a previous study by Nnadi and Brave in 2011, a superabsorbent was produced with a water absorption capacity of 73 times its dry weight. The high water absorption value is due to the increased molecular bonding that forms a three-dimensional polymer network, resulting in a superabsorbent. In addition, at the same absorption time, the lowest capacity in water absorption is DSS/CMC-0, indicating that the addition of citric acid shows a significant change [11].

Table 2. PBD/CMC/AS water absorption capacity result value data

Sample	Absorption Time (hours)	WAC (%)
DSS/CMC-0.0	24	3680
DSS/CMC-0.2	24	2760
DSS/CMC-0.4	24	3820
DSS/CMC-0.6	24	4130

4. Conclusion

The characteristics of the superabsorbent show that the FT-IR spectrum supports the formation of superabsorbent polymers carried out with the presence of carboxyl groups from CMC at wave numbers around 1640 cm^{-1}. In the DSS/CMC mixing spectrum, there are -OH and -CH groups at wave numbers around 3302-3333 cm^{-1} and 2907-2922 cm^{-1}. SEM test characteristics reveal the superabsorbent's rough surface and high density, as evidenced by the tea bag method's water absorption capacity, which reaches a maximum WAC characterization of 4130%. It can also absorb water up to 41.3 g/g, with a maximum cross-linking degree of 41%.

5. Acknowledgements

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6. Conflict of Interest

Authors declare no conflicts of interest

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