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The Release of Fertilizer on Corncob Cellulose – Based Acid-Acrylamide Hydrogel Prepared by Chemical Cross-Binding Method

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Abstract

Farmers' knowledge of the amount and dosage of fertilizers recommended for a practical fertilization system is low. As the result, the plant does not fully absorb the given fertilizer. Some fertilizers are leached by the flowing water and wasted into the soil layer. In long term basis, this practice can cause environmental pollution, especially on the land, water and air. Due to this fertilization problem in agriculture practice, a material with a high-water absorption capacity, which further releases it together with the fertilizer over a desirable period of time, is needed. One way to effectively provide water and nutrients to the plants and improve the physical and chemical properties of the fertilizzer is by the application of hydrogel. In this work, the release of urea fertilizer in a hydrogel-based on corncob cellulose was prepared using N, N'-Methylene Bis-acrylamide (MBA) as a crosslinker was studied. This research aims to produce a hydrogel with good physical and mechanical properties using acrylamide based on corn cobs cellulose and can be applied as a fertilizer carrier matrix whose structure can regulate fertilizer release. The treatments tested were MBA concentrations of 0%, 1%, and 2%, while the ratio of cellulose: solvent was 1: 2 and the addition of urea fertilizer with a concentration of 5%. The results showed that the swelling value increased with increasing acrylamide in the treatment ratio of the concentration of cellulose: acrylamide-acrylamide (NS: AAm). Fertilizer factors also gave a good swelling value. This shows that the addition of fertilizers gives maximum results. The hydrogel with the best treatment, namely the concentration ratio of 1% MBA, produced a swelling value of 7633.3%, a gel fraction of 76.51%, 1.73 miligram fertilizer loading, fertilizer release by 2.9%, a hardness of 7,865 N, with the morphology showing urea crystals in the form of white spots and showing the results of a slow but optimum release rate of fertilizer so that it can be applied for agriculture that requires a lot of nutrients at the beginning of growth.

Keywords: *corncorb; cellulose; hydrogel; N',N'-Methylene Bis-acrylamide (MBA), slow released fertilizer*

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INTRODUCTION

The fertilization system implemented by farmers often does not follow the recommended amount and dose of fertilizer. Farmers generally use high dose of fertilizer without adequate awareness its danger to the environment. Such fertilization systems are less effective. The plant does not fully absorb the given fertilizers. Some fertilizer is leached out by water and wasted into the soil. This will cause environmental pollution, especially on land, water, and air, in the long run. In a study (Serpil 2012), the nitrate content in the drinking water increased. The nitrate content in the well of the nearby community exceeded the NO_3-N concentration limit. On the land, 85% of the soil in this area became more acidic (low pH value) and considered critical. In addition, improper use of fertilizers will cause air pollution due to the emission of nitrogen oxides (NO, N_2O, NO_2). This gas significantly promotes the greenhouse effect. Globally, the amount of N_2O in the atmosphere increases from 0.2% to 0.3% every year. In addition, in the case of excessive use of nitrogen fertilizers, especially the content of plant nitrates will threaten human health and the consumption of leafy vegetables. Due to fertilizer issues in agriculture, a material that has water absorption capacity can be combined with fertilizer for long-term fertilizer release is required. One way to effectively provide plants with water and nutrients and improve physical and chemical properties is by the application of hydrogels. Hydrogel is a soil amendment that have the capacity to retain moisture and nutrients and supports plant growth by improving soil properties (Poormeidany et al. 2006). The use of hydrogel and fertilizer can improve bulk density, porosity, number of holes, field water holding capacity, moisture content, permanent wilting point water content, effective moisture, chemical properties and physical properties of soil biology. According to Bhaskar et al. (2013), a good hydrogel should demonstrate several advantageous criteria, including the ability to bear large amounts of fertilizer, periodically release fertilizer, hold fertilizer for a long time, and ability to hold soil moisture and control soil erosion. Therefore, hydrogels' importance and prospective application in controlled release culture systems and water absorption systems have received extensive attention.

Both natural and synthetic polymers can be used to synthesize hydrogels. It is recommended to use natural polymers because of they are abundantly available, non-toxic, have low production cost but low water absorption capacity, while synthetic polymers have high swelling properties. Most hydrogels overuse synthetic polymers. Because hydrogels are not environmentally friendly and non-toxic, they are difficult to undergo natural decomposition (Ahmed 2015). The advantages and disadvantages of each polymer determine the properties and characteristics of the hydrogel. These two characteristics are combined to produce a hydrogel with good swelling ability and physical properties. An alternative to natural polymers used for hydrogel synthesis is cellulose (Chang and Zhang 2011). Cellulose is a natural polymer that is found in many plants and is very abundant. Corn cobs are a material that has a high cellulose content. According to BPS (2019) data, Indonesia produces approximately 19.6 million tons of corncob waste each year. Therefore, it is necessary to optimize the use of these polymers. Natural polymers need to be chemically modified to improve their mechanical stability to approach the synthetic properties. In addition to increasing water absorption, the modification of natural polymers into hydrogels can also be used as a fertilizer carrier matrix, so its effectiveness is higher.

MATERIALS AND METHOD

Materials

The raw material used in this research was milled corn cobs powder of 60 mesh particle diameter. The chemicals used include sodium hypochlorite (NaClO), 8% NaOH, urea, distilled water, acrylic-acrylamide, KOH, ammonium persulfate (APS) initiator, and N, N'-Methylene Bis-Acrylamide (MBA) as a crosslinker.

Hydrogel Preparation

Cellulose extraction refered to the study of Shogren et al. (2011). The crushing process of corncob flour was treated in an autoclave at 80°C for 12 hours, and then treated with 8% NaOH in a delignification process at 15°C for 30 minutes. The sample was rinsed under running water. The bleaching process was carried out by using15 liters of NaClO for 1 hour at room temperature and followed by rinsing with running water until the rinsing water was neutral.

An ultrafine pulverizer (Masuko Corp, Japan) was used to reduce the fiber size, and the cellulose was dissolved in distilled water at a concentration of 2% (dry matter) (Iriani et al., 2015) and subsequently followed by sonication using an ultrasonic processor to process up to 300 ml of cellulose solution for 1 hour. The solution was stored at -4°C.

Hydrogel synthesis began with the neutralization of acrylic acid (by addition of KOH solution). Furthermore, as much as 10% wt acrylamide powder is dissolved in aquadest. Then, the neutralized acrylic acid was mixed with acrylamide solution into a beaker glass with a mixing ratio of 1: 1. This process was a modification that refers to the research of Rinawita (2011), Salim (2009) and Akalin (2019). Cellulose solution was added with APS solution as an initiator. The APS solution was prepared by dissolving 2% wt ammonium persulfate in distilled water. Then, 1% wt) of APS solution was added to the mixture. After 15 minutes of mixing, the cellulose (NS) solution was gradually added to the acrylic acid – acrylamide (AAm) solution until it reaches the specified composition 1: 2. The grafting reaction occurred for 45 minutes at a temperature of 45 ° C. After that, MBA as a cross-link agent was added gradually with variations in the concentration of the total cellulose-acrylamide acrylamide solution by 0, 1, 2% wt. The temperature was then raised to 70°C and kept constant for 3 hours. Then, the solution was introduced into a test tube and placed it at room temperature for 24 hours. After drying, the hydrogel was cut into 0.5 cm thick pieces and washed several times with distilled water to remove unreacted chemicals. Then, the hydrogel was first dried in air and dried in a vacuum oven at 40°C.

Determination of Gel Fraction

Dry the hydrogel in the optimal swelling state (no more increase) to see the remaining fraction. The number of undissolved parts indicates the number of cross-linking bonds formed during the cross-linking mechanism, which is performed by a chemical cross-linking method. Put 0.1 g of hydrogel in a tea bag (m_0), immerse it in 100 mL of the optimal aqueous solution for 24 hours, and then dry it in an oven at 60°C for ± 48 hours until constant weight. Then weigh (m1). The test was carried out three times. The gel fraction was calculated using the following equation (Erizal et al., 2015):

$$\text{Gel Content}\ (\%) = \frac{m1}{m0} \times 100\ \% \qquad (1)$$

where, m_1 = mass of dry hydrogel after soaking/swelling (g) and m_0 = initial mass of dry hydrogel (g)

Swelling Ratio

Swelling ratio determination was performed to find the hydrogel's optimum swelling or expansion ability to absorb water. Dry hydrogel (m_0) was immersed in water until there was no increase in weight then weighed (m_s). The swelling ratio was calculated using the following equation (Essawy et al. 2016):

$$\text{Swelling Ratio}\ (\%) = \frac{ms - m0}{m0} \times 100\ \% \qquad (2)$$

where, m_s = hydrogel mass when immersed for seven days (g) and m_0 = initial mass of dry hydrogel (g)

Swelling Kinetics and Diffusion Coefficient

The hydrogel was soaked in deionized water to achieve optimum swelling. The weight of the hydrogel was measured every day but on the first day, the weight of the hydrogel was measured every hour for five hours and the swelling value was calculated every hour and then the swelling kinetics of each hydrogel was obtained every one hour.

Kinetic analysis of swelling results was applied to Fick's law (El-Arnaouty et al 2015). Several parameters were calculated using the following equation:

$$F = \frac{M_t}{M_\infty} = Kt^n \qquad (3)$$

Mt was the water adsorbed at time t, and M_∞ is the water adsorbed at the optimum swelling, K was the swelling constant (h^{-1}) and n was the swelling exponent. The values of n and K can be calculated from the slope and intercept of the equation of the plot line (ln F) against (ln t). Fick's first and second laws describe most of the diffusion processes which were shown in the following equation (Ghaffar et al 2016):

$$F = \frac{M_t}{M_\infty} = 4\left(\frac{Dt}{\Pi L^2}\right)^{0.5} \qquad (4)$$

The slope of the linear plot between ($Mt/M\infty$) and $t^{1/2}$ gave the coefficient of diffusivity per unit area, D(m^2 h^{-1}).

Loading and Slow-Release Fertilizer

The process of loading urea fertilizer was carried out by immersing a pre determined weight of dried hydrogel sample in the urea solution. The concentration of urea 5 % wt. The weight ratio between dry hydrogel and urea solution was made to be 1: 1. The hydrogel was immersed in the urea solution for 24 hours, where it was expected that all of the urea solutions was absorbed into the hydrogel. This process refers to loading urea into the hydrogel by Ibrahim et al (2016).

The release of urea was measured by spectrophotometry at a wavelength of 689.5 nm. The prepared urea-rich hydrogel was introduced into a tea bag and was immersed in 100 mL of water. At different times (every 12 hours), approximately 0.5 mL of liquid samples were taken from the medium, and the absorbance was measured using a UV-Vis spectrophotometer for a maximum of 8 days, for a total of 4 days. The difference in sample volume from 0 to 8 is only 4 mL, so the volume can be considered constant (Pulat 2018). A standard curve was prepared for accurate use of an ultraviolet spectrophotometer. The standard curve was prepared by dissolving 1000 mg of urea in 1 l of raw water (1000 ppm). Dilute the solution to various concentrations, such as 500, 250, 125, 75, 37.5, 18.75, and 9,375. A UV spectrophotometer was used to measure each concentration of the spectrophotometer at a wavelength of 689.5 nm. After obtaining the absorbance, plot it as a graph of the relationship between absorbance and concentration.

Determination of the Concentration of Urea Release

Measurement of the weight of released urea from the hydrogel was calculated based on the linear correlation between the absorbance of the sampleand the urea standard curve. For each extraction, the concentration is obtained and with the formula below, the measured urea mass is obtained and can be associated with the urea release formula above:

$$\text{Concentration} = \frac{\text{mass of urea}}{\text{volume of solvent}} \quad (5)$$

where, the concentration of urea in parts per million (ppm), the mass of ammonium nitrate in mg (mt), and the volume of solvent in liters. (Pulat 2018). The cumulative release of urea was calculated using the equation given below:

$$\text{Release (\%)} = \frac{m_t}{m_{total}} \times 100\% \quad (6)$$

where m_t is the mass of urea released in the medium over time, and m_{total} represents the initial total mass of urea trapped into the hydrogel system (52 mg). (Pulat 2018)

Texture Analysis

A texture analyzer was employed to analyze the texture of the swollen hydrogel. The dried hydrogel was soaked in water for 24 hours. Then, the hydrogel was drained and placed in a container. The sample probe of the texture analyzer was use to press the sample to obtain the sample strength values are in the form of hardness (mJ), adhesion (mJ), cohesion and elasticity (mm).

Surface Morphology

The dried hydrogel was cut into thin slices, and then the slices were dissolved with a fertilizer solution for morphological observation. A scanning electron microscope (SEM) was used to characterize the surface morphology by employing 500 × and 2000 × magnifications, respectively.

RESULTS AND DISCUSSION

Gel Fraction

The degree of cross-linking in the hydrogel is represented by the value of the gel fraction, which also reflects the density of cross-linking that occurs between polymers. Such increase in the MBA concentration caused an increased value of the gel fraction to some extent and then decreased as shown in Figure 1. The MBA as, a cross-linking agent contains two reactive double bonds, it can easily polymerize and form cross-linking bonds (Salim and Suwardi 2009). The highest fraction value was in the concentration with 1% wt MBA at 76.51%. Addition of 1% MBA caused the degradation of the long chain of natural polymers to form radical compounds leading to the improvement of the gel fraction (Erizal et al. 2011). A higher gel fraction indicates a higher degree of cross-linkage.

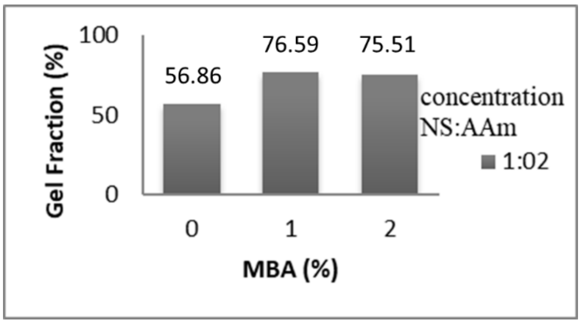


Figure 1 Value of hydrogel gel fraction on MBA treatment

Swelling Ratio

Swelling is an important parameter and directly affects the release of fertilizer. The amount of fertilizer in a concentration will cause the hydrogel to better hold water in it. The hydrogel swelling characteristic of 5% urea is shown in Figure 2a.

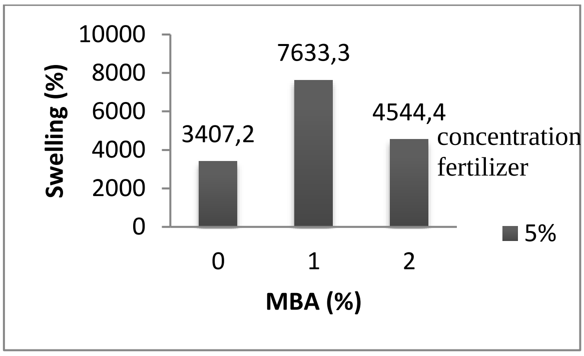


Figure 2a. The value of hydrogel swelling in fertilizer treatment of MBA

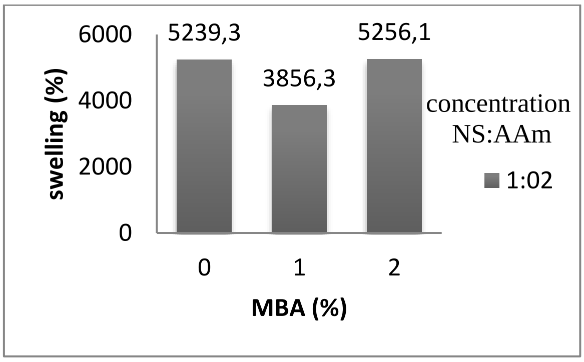


Figure 2b. Hydrogel swelling value in the treatment of the concentration of NS: AAm against MBA

Figure 2a shows that the highest swelling value (7633.3 %) was achieved at 1% wt MBA, but a higher MBA concentration induced the reduction of swelling value. This is because the coating at a concentration of 1% wt MBA made the hydrogel was able to retain more water.

The treatment of increasing MBA concentration in Figure 2b shows an increased swelling value. An MBA added as a cross-linking at polymerization will produce polymer networks (which can absorb water) (Astrini et al 2016). The ability of a polymer to absorb water depends on the degree of cross-linking. The highest average swelling results were obtained from 2% wt MBA concentration treatment. The addition of MBA concentrations at 0 % and 2% wt, cause the structure of the hydrogels does not dissolve easily on contact with water due to the cross-linking with the polymer network.

Swelling Kinetics and Diffusion Coefficient

Figure 3 shows that the longer the immersion time, the more the hydrogel absorbs fertilizer because a diffusion process occurs at a certain rate so that the swelling value increases. The optimum swelling value at 12 hours was used in the analysis to determine several parameters including the swelling exponent (n), swelling constant (K) and diffusion coefficient (D). The values of n and K can be calculated from the slope and intercept of the equation of the line plot (ln F) against (ln t) while the diffusion coefficient (D) is the slope of the linear plot between ($M_t/M\infty$) and $t^{1/2}$.

Based on the swelling kinetics analysis shown in Table 1, each hydrogel concentration ratio of the MBA solution is included in the first diffusion type, namely Fickian because the value of n was 0.5. This indicates that the rate of diffusion is lower than relaxation. Therefore, the swelling kinetics shown in Figure shows that the hydrogel in each comparison of the MBA concentration has a swelling value that is almost the same as the other concentrations. The rate of diffusion is directly proportional to the swelling value which indicates the absorption of fertilizer in water.

Table 1. Hydrogel swelling kinetics analysis

NS:AAm ratio	MBA (%)	Urea (%)	n	K (h^{-1})	D ($m^2 h^{-1}$)
1:2	0	5	0.2821	0.1892	0.7011
1:2	1	5	0.2702	0.1711	0.7385
1:2	2	5	0.2721	0.1075	0.6860

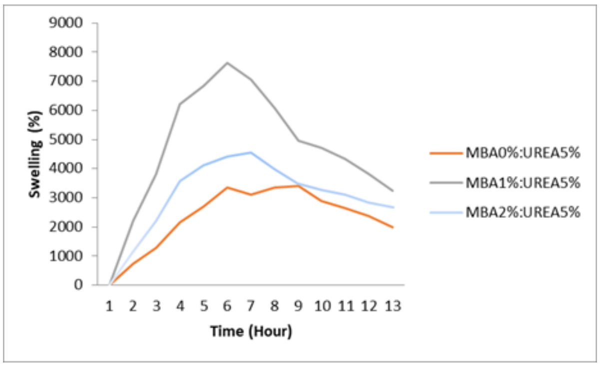


Figure 3 Swelling kinetics of hydrogel against immersion time in the variation of MBA

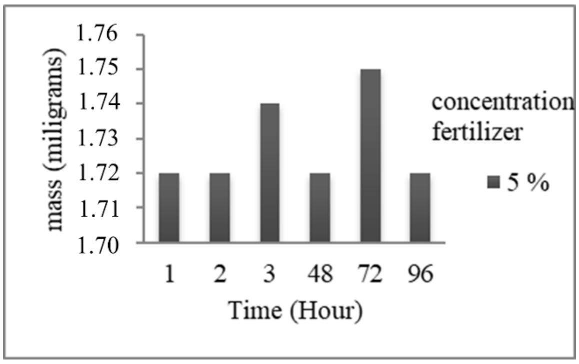


Figure 4 The Fertilizer release from hydrogels

The swelling constant, K shown in Table 1 was obtained from the intercept of the equation of the plot line (ln F) against (ln t) which results in a value of $F < 1$. Therefore, the values of (ln F) and (ln K) will be negative. The negative sign can be ignored, meaning that the negative sign did not affect the characteristics of the swelling constant (K) on swelling, but the negative sign indicates the flow of the diffusion process from high concentration to low concentration based on Fick's law. The value of the swelling constant (K) and the diffusion coefficient (D) have a positive correlation with the swelling value of the hydrogel (El-Arnaouty et al. 2015). Therefore, higher swelling values also indicated the higher K and D values.

Fertilizer Release

The release of the fertilizer achieved using a hydrogel weight soaked with urea fertilizer was tested for its absorbance value using a UV-vis spectrophotometer.

Figure 4 shows the relationship between the mass of urea released from the hydrogel and the function of time in the hour scale. In the Figure, the release of urea increases until the 72nd hour and decreases at the 96th hour, this rapid fertilizer release is accompanied by a decrease in the fertilizer in the following hour. The release of fertilizers continued to increase until the time of the 96th hour. The slow release of fertilizer is due to the absorption of the MBA cross-linking agent in the hydrogel network, resulting in a reduction in fertilizer release. The results showed that the fertilizer release rate followed the slow but optimum release concept. The concentration of fertilizer will regulate the solubility of nutrients so that the measured release of nutrients will be gradual. Slow release of fertilizer ranged from 2.9% of the total mass 60 mg. Urea in the hydrogel network will experience swelling when dissolved with water and fertilizer so that it will slowly release due to the free exchange of water with hydrogels and fertilizers so that it can be applied to agriculture that requires a lot of nutrients at the beginning of growth (Ahmed 2017). The main advantages of natural-based materials for controlled release via synthetic polymers are that they are environmentally friendly, inexpensive, readily available, and biodegradable. In addition, in the hydrogel, the presence of this material is very important in increasing the water resistance capacity in the soil.

Texture Analysis

Based on Table 2, it can be seen that the hydrogel containing fertilizer increases hardness, cohesion and elasticity, and reduces adhesion. According to Ghanbarzadeh et al. (2011), the cross-linking reaction in the hydrogel leads to a tighter structure. This causes the strength of the hydrogel to increase with the increase of the concentration of the cross-linking agent. In Table 2, the hydrogel hardness values are given by the cross-linking agent (MBA) with concentrations of 0%, 1%, and 2% in the concentration ratio of the NS: AAm solution (1: 2) had the best swelling value. This indicates that providing the cross-linking agent in the form of MBA can increase the hardness of the formed hydrogel, thereby increasing the ability of the hydrogel to withstand external pressure.

Table 2 Results of Hydrogel Texture Analysis on the MBA Treatment

Treatment	Hardness (N)	Adhesion (mJ)	cohesion	elasticity (mm)
S2M0P5	9.047	0.02	0.93	4.9
S2M1P5	7.865	0.04	1	4.71
S2M2P5	7.414	0.16	0.92	5.55

aA: Comparison of the concentration of NS: Aam (S2: 1: 2), M0: MBA concentration 0%, M1: MBA 1%, M2: MBA 2%, P5: fertilizer concentration 5%

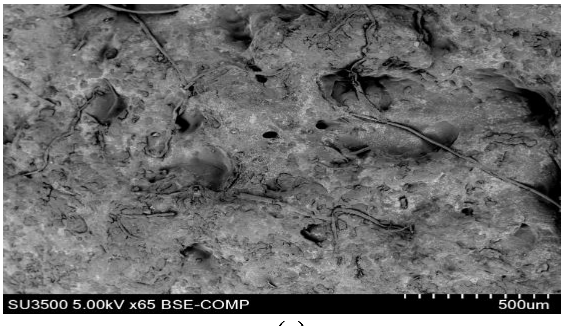
Adhesion is a measure that describes the force between different types of molecules. The higher the adhesion value, the material will have a texture that is easy to adhere to the media. According to Table 2, it can be seen that the control treatment (MBA 0% wt) has a high adhesion value compared to the hydrogel that has undergone cross-linking and has a fertilizer concentration of 5% wt. The high adhesiveness causes the control sample to have a sticky structure like glue. This overly sticky structure is certainly not suitable for hydrogels, because later this hydrogel will be used as a garden medium on sandy land. Therefore, cross-linking treatment is necessary to produce a hydrogel suitable for its application as a growing medium. In addition, the higher the adhesiveness value will cause the hydrogel to be more easily degraded. The adhesiveness value at the fertilizer concentration indicated that the addition of urea was suitable as a hydrogel application.

Table 2 shows that giving cross-linking treatment using a chemical cross-linking agent (MBA) can significantly reduce adhesiveness and increase cohesiveness. According to Table 2, it was found that cross-linking resulted in the lowest adhesion (MBA 1% wt). The low adhesion value indicates that the hydrogel structure is mechanically generated. At the same time, as shown in the figure, cohesion and adhesion are inversely proportional. The lower the hydrogel adhesion value, the higher the cohesion of the hydrogel. Cohesion is a measure of the strength of interaction between similar molecules. The higher the cohesion value, the better the mechanical stability.

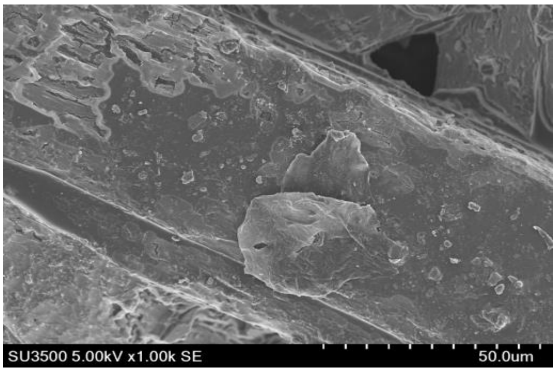
Elasticity is a measure of the elasticity of a material when a load is applied. This value can be measured by measuring the restoring force of the material after the compression force is applied. According to Table 2, the hydrogel with the highest elasticity value has an MBA content of 2%. This indicates that the hydrogel has the highest level of elasticity compared to other treatments. However, on the whole, under the same cross-linking treatment, the addition of 1% hydrogel of fertilizer on the MBA reduced the elasticity value. This may be due to the fact that the hydrogel has a harder structure due to the tightly formed cross-links, which makes the ability to recover its original shape after being subjected to a load lower.

Surface Morphology

Figure 5 shows the observation of the surface morphology of the hydrogel sample when a solution with a concentration of NS:Aam (1:2) was treated with 1% wt of MBA. Figure a) hydrogel without fertilizer addtion, that is, the shape of the material has a highly porous structure when swelled in water, showed the shape of material has a highly porous structure when swelled in water, so it shows a rod-like shape. Compared with Figure 5 (a), a rough and smooth surface is observed in Figure 5 (b). Urea crystals in the form of white spots are scattered on the surface, and the pore size is reduced due to the hydrogel absorption of the fertilizer according to the deposited fertilizer.



(a)



(b)

Figure 5 Hydrogel surface morphology using SEM a) NS: AAm (1: 2) MBA 1% without fertilizer b) NS:AAm (1: 2) MBA 1% fertilizer 5%

CONCLUSION

Hydrogels can be synthesized using a combination of cellulose solution from corncobs and acrylic-acrylamide acid using the chemical cross-linking method. Hydrogel treatment of cellulose (NS) solution to acrylamide (AAm) by chemical cross-linking method showed a swelling value between 444-5256.1% in the MBA treatment.

The hydrogel showed optimum results in the chemical cross-linking method, namely at 1% wt MBA concentration with a swelling value of 7633.3%, 76.5957% gel fraction, 1.73 miligram fertilizer loading, fertilizer release by 2.9% and 7.865 N hardness mechanical properties. 500 × shows the fertilizer-free treatment, which exhibited a stem-like shape. The surface of the hydrogel was rough, but it became smooth when it was treated with fertilizer. Urea crystals in the form of white spots were scattered on the hydrogel surface leading to the reduction of pores size. The swelling ability of the hydrogel can be considered for further application in various fields, including diapers, soil conditioners, wound dressings, drug delivery, etc.

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