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PRODUCTION OF SUPERABSORBENT HYDROGEL FROM COPOLYMERIZATION POLYACRYLIC ACID BASED CARBOXYMETHYLCELLULOSE (NaCMC) WITH ADDITION POLYMERIZATION

Prahady Susmanto✉, Tuti Indah Sari, Elfa Armelia and Stevany Vanesya Syeva Maharani

Department of Chemical Engineering, Universitas Sriwijaya, Palembang-30862, Indonesia
✉Corresponding Author: prahady.susmanto@ft.unsri.ac.id

ABSTRACT

In this study, a polymerization process was conducted using additional polymerization with a crosslinking agent in the form of MBA, on the main chain (backbone), namely polyacrylic acid (PAA), and the side chain, namely cellulose from NaCMC, supported by an initiator in the form of ammonium persulfate (APS). The highest absorption capacity of the superabsorbent hydrogel obtained was 525%, with an APS variation of 0.2 grams and an MBA of 0.28. The best swelling ratio in the NaCl solution based on research data is sample C2 at 517% with APS initiator variation of 3% and MBA of 4%. Meanwhile, in the urea solution, the best swelling ratio is sample B2 at 550% with APS initiator variation of 2.5% and MBA of 4%. Based on FTIR analysis, sample C2 shows a broken C=C bond, where the peak at 1654 cm^{-1} indicates the absence of this bond, illustrating the successful graft copolymerization reaction between cellulose and acrylic acid, where C=C occurs in PAA.

Keywords: Crosslink Agent, Superabsorbent Hydrogel, Initiator, Addition Polymerization

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INTRODUCTION

The goal of superabsorbent modification is to create a superabsorbent formulation that is optimal in terms of quality, cost of production, and environmental effect. Hydrogels are gels that have undergone mechanical or chemical cross-linking; polar materials are typically used to make them. The structures are connected in the first case by physical forces such as hydrophobic, hydrogen bonding, or ionic forces; in the second case, the gel has networks that are covalently coupled.[1] Superabsorbent hydrogels have several times more absorption capacity than weight, which is one of its benefits. Superabsorbents have been modified in numerous earlier investigations to strengthen their shortcomings and enhance their qualities. Utilizing crosslinkers like sodium trimethaphosphate (STMP) and aluminum sulfate (AIS) in conjunction with starch allows for the production of superabsorbents with excellent flexibility and good absorption that are biodegradable.[2] The hydrogel containing 10% Citric Acid, CMC (Mw=250,000)/PEG10, and high absorbency (3200%), moderate degradation (54%), stiffness (1.39 ± 0.19 GPa), cell survival (94.8 ± 1.3%), and strong cell proliferation demonstrated acceptable properties. SAH with tunable structural, chemical, and biocompatibility with the modification of the CMC and PEG concentrations used.[3] Superabsorbent hydrogels made of various materials have been created through numerous investigations. It has been discovered that chemical procedures are more economical than physical ones. In addition, cellulose is easily accessible and abundant in comparison to other natural sources like starch and chitosan.[4] Three categories of superabsorbents exist natural, synthetic polymers, and semi-artificial (cellulose derivatives). Carboxamides, carboxylic acids, carboxylate salts, and partially neutralized carboxyl groups are commonly found in superabsorbents. These characterize the environment in which water diffuses into the network while cross-links in the superabsorbent's backbone chain keep it structurally intact. As a result, the network of polymers swells and grows, and the covalent cross-links keep the network from dissolving in water. Superabsorbents have the ability to absorb water up to 1000–1500 times their dry weight without disintegrating. Superabsorbents are made for a wide range of applications to meet a variety of purposes because of their notable variances in characteristics. Polyacrylic acid has ionized groups on every repeating unit (-COOH) in its structure. The flexibility and swelling capacity of a superabsorbent can be regulated by

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the kind and quantity of cross-linker employed in a number of superabsorbent manufacturing techniques.[5] The use of different initiators and crosslink agents can provide new characteristics with the previous superabsorbent. The use of initiator and crosslink agent variations aims to determine the optimal formulation to produce superabsorbent with the best quality, economical, and utilize safe material.

EXPERIMENITAL

Material and Methods

Ammonium persulfate (APS) as initiator from Merck, N, N'-methylene bisacrylamide (MBA) as crosslinkers from Merck, Sodium Carboxymethylcellulose (NaCMC) from Sigma Alrich, Aquadest, Polyacrylic Acid from Merck, Urea, and NaCl. 3 grams of NaCMC and 8 mL of PAA are the concentrations. Variation in the number of initiators is 0,16(2%); 0,20(2,5%); 0,24(3%); and 0,28(3,5%) gram amounts the monomer while the variation in the number of crosslinkers is 0,28(3,5); 0,32(4%); and 0,36 grams (4%) amounts the monomer.

Superabsorbent Polymer Synthesis Stage

NaCMC as much as 3 grams was dissolved into 50 mL of distilled water accompanied by constant stirring for about 1-2 hours until homogeneous. The stirring process is carried out on a hotplate using a magnetic stirrer at a temperature of 20-30 ℃. Ammonium persulfate initiator was added with variations of 2%; 2.5%, 3%; and 3.5% of the weight of the monomer used and then stirred and heated at a temperature of 60-65 ° C for 30 minutes. Polyacrylic Acid (PAA) was then neutralized with 10 M NaOH. The neutralization process is carried out where PAA is dripped with 10 M NaOH little by little until it approaches a neutral pH by testing using a universal indicator. After stirring for 30 minutes, 8 grams of Polyacrylic Acid (PAA) and N, N'-methylene-bisacrylamide as a crosslinking agent were added to the suspense with variations (3.5; 4%, and 4.5%). The polymerization reaction was carried out at 70°C for 4 hours to react. The thickened sample was then poured into a petri dish and placed in an oven at 50-55℃. This research is based on Klinpiktusa and Kosaiyakanon (2017) and Pourjavadi, *et.al* (2010).[6,7]

Absorption Capacity Testing of Superabsorbent Hydrogel

The polymerization of the superabsorbent hydrogel yielded products with a maximum weight of 0.1 grams. After it's inside the sample container, pour 6 milliliters of distilled water in and let it sit for 15 minutes at room temperature. The liquid that is not absorbed is removed and left to stand until it no longer drips. Analyze the superabsorbent's absorption capacity using the water absorption test, following ASTM D570-98. [8] The following formula was used to determine the absorption capacity.

$$Q = \frac{m_2 - m_1}{m_1} \times 100\ \%$$

Where: Q = Absorption capacity, m_1= Weight of the dry sample before immersion and m_2= Weight after the sample expands

Swelling Ratio Testing of Superabsorbent Hydrogel in Urea and NaCl Solution

The swelling capacity of the superabsorbent is conducted using the method [9]. The superabsorbent hydrogel's polymerization produced results that weighed up to 0.1 grams. After that, place into the sample container and let 6 mL of 5% urea solution sit at room temperature for 15 minutes. The liquid that cannot be absorbed is taken out and left to stand until it stops pouring. After swelling, superabsorbent hydrogel was weighed as a mass. For the 0.9% NaCl solution, the prior procedure was carried out again. The following formula was used to get the swelling ratio:

$$\text{Swelling ratio} = \frac{W_2 - W_1}{W_1} \times 100\ \%$$

Where: W_1= Weight of the superabsorbent hydrogel polymer in the swelling state and W_2 = Weight of the superabsorbent hydrogel polymer in dry state

FTIR Analysis

The material's and the superabsorbent hydrogel products' functional groups are determined by this analysis. After combining the SAPs with KBr, they were compacted onto a plate and subjected to spectroscopic evaluations at 400 cm^{-1} to 4000 cm^{-1} in ambient circumstances. From each spectr, a background spectrum with no samples was subtracted. A Zeiss Supra 40 (Germany) equipped with a 5 kV acceleration voltage was used to take SEM pictures.[5]

SEM Analysis

The morphology of hydrogel after water absorption and swelling was observed by SEM. Commonly, the samples were gold-coated by sputtering technique before analyses.[10]

RESULTS AND DISCUSSION

The backbone of this study is PAA polymer, the daughter chain is cellulose derived from NaCMC, the initiator is APS, and the crosslinking agent is MBA. The amount of initiator employed in the hydrogel preparation varied as much as 0.16, 0.20, 0.24, and 0.28 grams, and the amount of crosslink agent varied as much as 0.28, 0.32, and 0.36 grams. The hydrogel formation process was carried out through the chemical crosslinking method with the help of initiators as chemical reagents, using the cast-drying method technique. With no further chemical reagents needed for drying, a clear thin-film hydrogel was produced. The superabsorbent hydrogel product was then kept in a desiccator to shield it from the effects of ambient humidity after the hydrogel dried.

Addition Polymerization Process in the Preparation of Superabsorbent Hydrogel

The mechanism of synthesis and reaction in the research is the reaction of monomer free radical polymerization with synthetic cellulose and crosslinking polymerization consisting of initiation, propagation, and termination reactions according to the estimation of the reaction based on Klinpiktusa and Kosaiyakanon (2017), and Rakhmawati *et al* (2019). Addition polymerization is the type of polymerization used in this study involving a chain reaction, which results from the presence of pineapple radicals (reactive particles containing no unpaired electrons) or ions.[6,11] The following is the reaction mechanism.

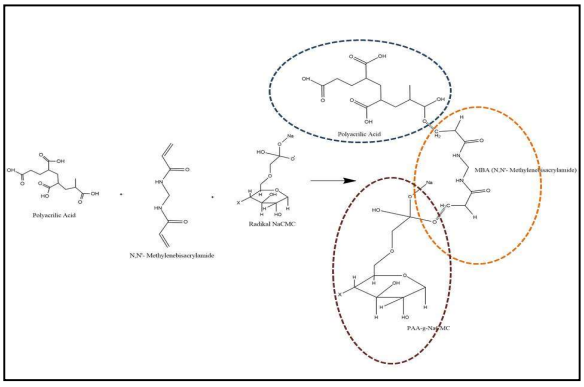


Fig.-1: Polymerization of PAA-g-NaCMC

The formation reaction of free radicals is the initial process of the radical polymerization reaction in the synthesis. The APS initiator was added when the temperature of the NaCMC suspense solution reached 65°C. SO4- radicals derived from APS will be formed when heating takes place. The hydroxyl radicals then initiate the double bonds of NaCMC to trigger the next stage of propagation in the polymerization process. NaCMC is one type of carboxymethyl group derivative that is environmentally friendly and functional as well as biodegradable and renewable. The hydroxyl radical triggers the acrylic acid monomer for radical-initiated polymerization.[3] Reactive NaCMC macroradicals are created when the hydroxyl radicals further abstract the NaCMC molecules. Next, the macroradicals start the monomer and MBA graft copolymerization process, which results in the creation of NaCMC-G-PAA crosslinks. MBA creates a water-soluble, superabsorbent hydrogel by acting as a crosslinker between the PAA chains. After the addition of MBA, the viscosity increases and the temperature increases to 70-75°C. MBA contains two double bonds which are reactive and can combine with two different chains. This c[12]ondition must be maintained to achieve stability until the polymer growth stops or the final stage.

Impact of Different Initiator and Crosslinking Agent Combinations on Absorption Capacity Value of Superabsorbent

The addition of MBA increases the absorption capacity but the addition of MBA with too much amount can give poor swelling. The increase in tissue density also causes an increase in physical attachment and management of swelling properties.[5] The absorption capacity analysis is in Fig.-2.

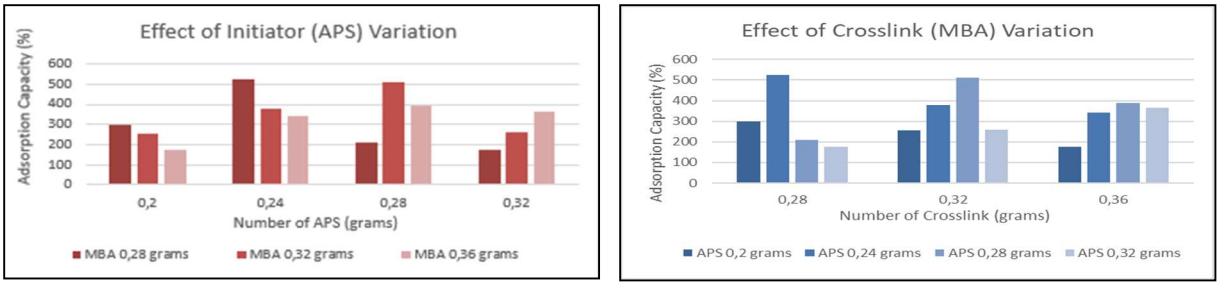


Fig.-2: Results of Absorption Capacity Analysis with (a)APS Variations and (b) MBA variations

Figure-2(a) illustrates the optimum point of absorption capacity of superabsorbent formed is 525%, with variations of APS as much as 2% (0,16 grams) and MBA as much as 3.5% (0,28 grams). The increase in absorption capacity value is at the addition of the amount of initiator by 2.5-3% (0,28-0,32 grams). However, the analysis results showed a decrease for the addition of MBA with variations of 3% (0,32 grams) and 3.5% (0,36 grams). This shows that there is an optimum point in the addition of initiator where if the addition of APS is too much, the water absorption becomes worse than before. The excessive amount of initiator causes the swelling value to decrease due to the shorter molecular chain. The length of the hydrogel's molecular chain is altered to alter the impact of the amount of initiator injected on absorption. An insufficient quantity of initiators leads to a reduced ability of micro/nano cellulose to generate free radicals, hence impeding the successful formation of a three-dimensional macromolecular network. However, because the active center is too slow to stop polymerization, the process finishes sooner when there is an excessive amount of initiator present. Polymerization of AA is triggered to generate polymers with lower molecular weights and increase the density of crosslinking, resulting in a decrease in absorption capacity[13]. The impact of crosslinker modifications on the absorption capacity analysis results is shown in Fig.-2(b), where sample B1 (APS 0,2 grams and MBA 0,28 grams) achieved the greatest value, with a result of 525%. Based on the figure above, it can be concluded that at a fixed APS concentration, the more crosslink agents, the higher the absorption capacity. However, after passing the respective optimum point, the results will adjust to the variation of initiators and other materials where the addition of excess MBA tends to cause a decrease in the absorption capacity. This is because the network structure of the product formed will be even more. This will cause the pores to become smaller and the product is difficult to absorb water. The addition of too little MBA causes less tightness of the connective tissue formed in the product. Based on previous research, water absorption can also be influenced by the formation of additional cross points where the -OH on the surface will react with the monomer which causes the flexibility of the copolymer chain to be reduced, a decrease in the hydrophilic part of the composite and a chain transfer mechanism that shows an obstacle in the growth of the chain of a polymer product.[14]

Impact of Different Initiator and Crosslinking Agent Combinations on the Swelling Ratio of Superabsorbent

The swelling ratio in this study was analyzed using 0.9% NaCl solution and 5% urea solution. The results of the graphical presentation in Fig.-3(a) and Fig.-3(b) show that in the NaCl solution, the swelling produced by some samples decreased. The decrease or increase in the swelling ratio is in line with the addition of the initiator and crosslink agent. The smaller the value of the addition of initiator in NaCl solution up to 2.5% (0,2 grams), the swelling ratio of the polymer decreased. However, if more initiator is added around 3-3.5% (0,24-0,28 grams), the swelling ratio increases as shown in Fig.-3(c) and 3(d). It can be seen in the data that the water absorption increases with increasing mass ratio of APS, and then, decreases with increasing mass ratio of APS. When the amount of APS as an initiator is low, it will produce a small amount of free radicals, resulting in low reaction activity, therefore, it is difficult to form an effective network[15]. The following is a sample graph of the swelling capacity analysis.

On the other hand, the grafted chain's length shortens and the absorption capacity drops when an excessive amount or variety of APS is added. This happens at the concentrations shown in Fig.-3(c) and 3(d) using urea solution. One important component of water absorption in superabsorbent hydrogels is the crosslinker content of MBA. This is because a higher cross-linker content will result in a denser polymer network,

which stiffens the structure. It will be challenging to access the superabsorbent's tiny pores and room for the absorption of water molecules[16]. The best swelling value in NaCl solution based on the research data is sample C2 of 517% with 0,24 grams APS initiator variation and 0,32 grams MBA. In urea solution, the best swelling value is sample B2 of 550% with a variation of APS initiator 0,28 grams and MBA 0,32 grams. When compared to the swelling results, the urea solution has the best swelling capacity value.

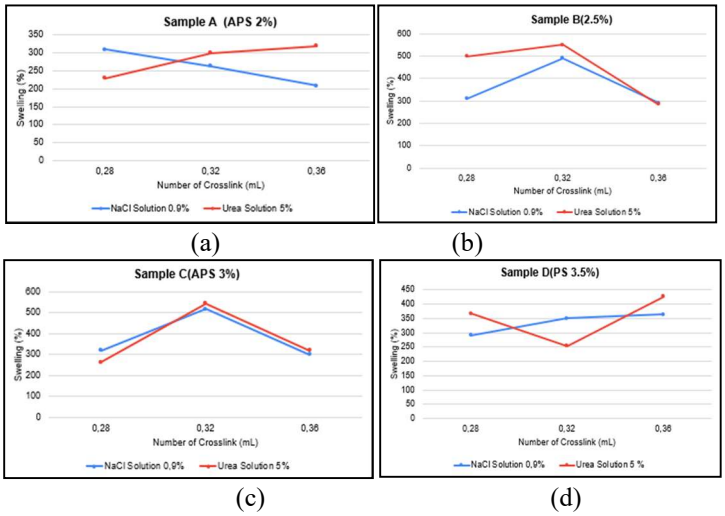


Fig.-3: Relationship between the Effect of Initiator and Crosslink Variations on Swelling

This is because the protective effect of cations in the salt solution is a supporting factor for the decrease. Another factor is also caused by excess cations in the solution with higher concentration. However, it is inversely proportional to the urea solution in the superabsorbent which has increased swelling capacity. This is due to the hydrophilic nature of the urea group[17]. The availability of bigger monomer molecules in the vicinity of the NaCMC macromolecular chain propagation site may account for the influence of PAA and NaCMC on the initial swelling capacity. Because there are more counter ions in NaCMC, which can improve the hydrogel's propensity to swell, these ionic groups tend to raise the osmotic pressure[18] The more NaCMC was used, the tighter the gel structure formed. The process occurs when water is present, Na^+ will be released and replaced with H+ ions to form HCMC, and increase viscosity. In addition, the higher content of acrylic acid increases the hydrophilicity in the superabsorbent and leads to stronger solvation.

FTIR Characterization of NaCMC, Initiator, Crosslink Agent, and Best Sample

The peak that shows the presence of CH_2 in the results of FTIR analysis of NaCMC can be seen at 1425-1421 cm^{-1} [19]. The two primary functional groups found in cellulose are O-H and C-H. Strong absorption bands are thought to exist at wave numbers of approximately 1600–1640 cm^{-1} and 1400–1450 cm^{-1}. The -COO ionization group and its salts exhibit both symmetric and asymmetric vibrations, indicating this. This demonstrates that when the carboxymethylation process takes place, carboxyl groups are substituted in the cellulose series.[20] Based on swelling analysis data in 0.9% NaCl solution and 5% urea solution, the maximum values were 517% and 544%, respectively, and sample C2 was selected as the sample with the best initiator variation. The existence of many functional groups possessed by APS, including C-O, O-H, C-O-C, C=C, C-C (aromatic), and C-H, is demonstrated by the addition of APS to this polymerization. The AA hydrogel's spectrum reveals that the peak at 1634 cm^{-1} lacks C=C bonds, a sign that the polymer chains were successfully produced and the C=C bonds were broken. Meanwhile, the peak corresponding to the COOH group is still present in all spectra[21]. Based on the results of FTIR analysis of sample C2, it can be seen that the C=C bond is broken where the peak of 1654 cm^{-1} shows the absence of the bond. This shows the copolymerization graft reaction of cellulose and acrylic acid was successfully formed where the C=C occurred in PAA. The results of FTIR analysis can be seen in Fig.-4.

The selection of sample D3 as a sample with the best MBA variation considering that the results of the absorption capacity of this product are considered quite good at 390% while the value of swelling analysis in 0.9% NaCl solution and 5% urea solution is 491% and 550%, respectively. The addition of MBA can be

seen from the joining of several typical MBA functional groups such as -NH and C-O in the analysis of sample D3 which are in the range of 3302.4 cm^{-1} and 1543.1 cm^{-1}, respectively.

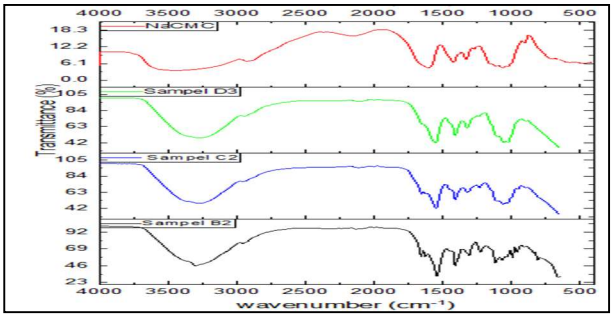
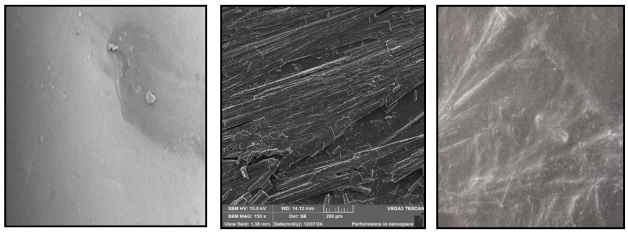


Fig.-4: FTIR Analysis Results of NaCMC and Superabsorbent Hydrogel Samples

Sample B2 was chosen as the best sample because based on the criteria of absorption and swelling analysis results showed good results. This sample has absorption capacity, and swelling ratio in NaCl solution, and urea solution of 380%, 490%, and 550%, respectively. Based on Fig.-4, when compared with NaCMC cellulose, there are some differences in the functional groups on the wave peaks such as -OH, CH2, -COO, and other groups while in the hydrogel product more prominent reading of C-O-C groups from NaCMC cellulose, -NH and C=O from MBA as a crosslinking agent and C-O and C-H from PAA. The presence of -NH bonds around 3266.1-3302.4 cm^{-1} on the FTIR graph reading on the superabsorbent hydrogel indicates that crosslinking has successfully formed on the polymer, followed by C=O bonds around 1543.1-1550.6 cm^{-1} from MBA. The C-O-C group seen in the FTIR spectrum is derived from the main cellulose of this hydrogel while some other groups are derived from the polymerization of additional ingredients, especially from PAA and MBA. According to earlier studies, the FTIR spectra for acrylic acid revealed the presence of the OH group at wave number 2987.92 cm^{-1}, the C=O group at wave number 1694.07 cm^{-1}, and the C=C group at wave number 1634.46 cm^{-1}. The FTIR spectra of MBA revealed the existence of the N-H group at wave number 3302.56 cm^{-1}, the C=O group at wave number 1655.79 cm^{-1}, and the C=C group at wave number 1538.23 cm^{-1}.[22]

Characterization of Superabsorbent Hydrogel Surface Morphology Structure on SEM Analysis

NaCMC was chosen due to the presence of a complex network of hydrogen bonds, which will stabilize the napkin after absorbing the liquid.[23] Morphological analysis using SEM in Fig.-5(a) for NaCMC and image,[24] (b) sample B2 as the best product, and (c) sample product of the best MBA variation.



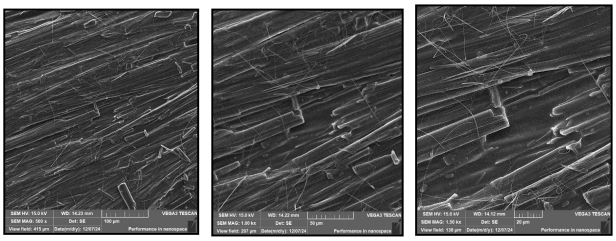
(a) (b) (c)

Fig.-5: (a)150x SEM of NaCMC; (b) 150x SEM of Sample B2 (c) 150x SEM of Sample D3

NaCMC has uniformly dispersed, fine fibers, as seen in Fig.-(a). The reason behind this morphology is the intense ionic crosslinking that takes place on the surface. The surface takes on a granular look due to the polymer chains' numerous places of bonding with the crosslinker. On the other hand, because of the high absorption percentage, picture (b) appears to have the best sample structure. The image's morphology is a combination of the NaCMC structure from the earlier image. The superabsorbent's surface structure with 3.5% APS and 4% MBA variation is depicted in Fig.-(c). A molecular bond of a crystal-like structure is formed by the initial NaCMC backbone structure.

The results of the surface structure at 500x, 1000x, and 1500x magnification are described based on Fig.-6. The material generated contains more pores with a bigger diameter than before the production of the superabsorbent, as explained by the micrograph results. In contrast to before the creation of superabsorbent

with weak structural qualities, the surface becomes dense, robust, and elastic. When the superabsorbent is created, the pore width increases from ±6 nm for polyacrylic acid to ±12 nm, indicating the success of the product.



(a) (b) (c)

Fig.-6: SEM Micrographs of Sample B2 (a) 500x; (b) 1000x; and (c) 1500x Magnification

The grafting process and the two materials' interfacial contact were successfully demonstrated by the alteration in surface structure. The abundance of -OH groups, which are the primary functional groups of NaCMC, led to an increase in the pores that formed on the surface. Such super absorbent (NaCMC-g-PAA) have a granule-shaped structure and a long backbone found due to cross-linked polyacrylates incorporated into the NaCMC molecule[25]. The surface of the MBA variation is held in place by a dense and rigid polymer network with a homogeneous skeletal structure, without showing obvious macropores. This may be due to the shrinkage of the matrix at higher levels of crosslinking density, affecting a rigid matrix structure with almost no porous surface. However, this rough and bumpy surface can help water penetrate the polymer network, thus improving water absorption.[26]

CONCLUSION

The addition of APS as initiator and MBA as crosslink agent can increase the absorption capacity but the addition of too much amount can give poor swelling. Increased tissue density also leads to improved physical adhesion and management of swelling properties. The best swelling value in NaCl solution is sample C2 by 517% while in urea solution is sample B2 by 550%. These results are influenced by the addition of MBA and APS in the polymerization where free radicals will be formed. The results of FTIR analysis of sample C2 showed the presence of a broken C=C bond at 1654 cm-1 peak which illustrates that the copolymerization graft reaction of cellulose and acrylic acid was successfully formed where C=C occurred in PAA. When the superabsorbent is generated, the pore width increases from ±6 nm for polyacrylic acid to ±12 nm, which indicates the success of the product.

CONFLICT OF INTERESTS

The authors whose names are listed certify that they have NO affiliations with or involvement in any organization or entity with any financial interest (such as honoraria; educational grants; participation in speakers' bureaus; membership, employment, consultancies, stock ownership, or other equity interest; and expert testimony or patent-licensing arrangements), or non-financial interest (such as personal or professional relationships, affiliations, knowledge or beliefs) in the materials discussed in this manuscript.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

Prahady Susmanto https://orcid.org/0000-0003-2219-3782
Tuti Indah Sari https://orcid.org/0000-0001-6426-7482
Elfa Armelia https://orcid.org/0009-0004-2318-3353
Stevany Vanesya Syeva Maharani https://orcid.org/0009-0000-5471-8411

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