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SYNTHESIS OF SUSTAINABLE SUPERABSORBENT BIOPOLYMER: MODIFIED RICE STRAW CELLULOSE WITH INITIATOR AND CROSSLINK AGENT

Abstract: Superabsorbent biopolymers are polymer-based materials that have high absorption capabilities made from synthetic monomers modified with natural monomers such as cellulose. The addition of cellulose as a natural monomer is to produce a product with superabsorbent properties biodegradable so as not to have a negative impact to the environment. The aim of this research was to determine the potential of rice straw cellulose to be used as a natural monomer in the manufacture of superabsorbent biopolymers. The preparation of this superabsorbent biopolymer was carried out by varying the concentration of the initiator and crosslink agent. Initiator variation potassium persulfate (KPS) namely 0.2 %; 0.4 %; 0.6 %; and 0.8 %; meanwhile, variation crosslink agent in the form of citric acid was 0 g, 2 g, 4 g, 6 g, and 8 g with the addition of 20 g acrylamide. Functional group analysis was carried out by testing Fourier Transform Infrared (FTIR) indicating the presence of -OH, C-H, and C=C functional groups in rice straw cellulose that shows the potential to be the material for producing superabsorbent polymer. The Scanning Electron Microscope (SEM) analysis showed that the morphology of superabsorbent, the surface of the superabsorbent biopolymer looks denser, this indicates that there has been a bond formation between the monomer and crosslink agent. The results showed that the superabsorbent biopolymer produced with variations in initiator concentrations of 0.6 % and crosslink agent 6 g has a water absorption capacity and swelling in solution with buffer acetate the best were 1304.3 % and 10.26 w/w respectively.

Keywords: superabsorbent biopolymer, rice straw cellulose, acrylamide, initiator, crosslink agent

Introduction

A polymer is a molecule composed of hundreds or even thousands of monomers and is widely used in everyday life. The role of polymers has also spread widely in various fields, and the types of polymers that we often encounter in everyday life include plastic, cloth, and rubber. Generally, polymers are made using synthetic materials and produce products that are less environmentally friendly due to their properties non-biodegradable which can cause environmental pollution and are toxic to humans. The development of modified polymers with the addition of natural materials has begun to be applied in the manufacture of superabsorbent biopolymers. Natural polymers with functional groups enable modification via crosslinking, simple blending process and grafting [1]. The physicochemical properties of a polymer can be further modified by forming a copolymer with another polymer or by grafting. Grafting polymers is incredibly important since it keeps the extended conjugated structure in the major chain while attempting to

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introduce and assimilate the grafted materials properties [2]. The chemical structure of hydrogel polymers influences the swelling characteristic of hydrogel, with hydrophilic functional groups having greater swelling capability than hydrophobic functional groups [3].

The use of superabsorbent has a wide scope, such as water storage media for agricultural areas, to catalyst urine absorption in baby diapers or sanitary napkins. The manufacture of synthetic polymer-based superabsorbent produces products with capacity swelling low, so it is necessary to add other substances and grafts with natural polymers [4]. One of the natural polymers that has the potential to be used in the manufacture of environmentally friendly superabsorbent is cellulose [5]. Compared to the using of only synthetic counterparts, the innovation of combine the synthetic and natural polymers gave its possibility of greater control of degradation rate [6]. The modifications of natural-based superabsorbent with chemical may affect environmental benefits compared to the synthetic counterparts, also has the lower carbon footprint, biodegradable, and could be promotes the use of renewable resources [7]. The selection of materials used in the manufacture of superabsorbent will affect the quality of the superabsorbent. Maintaining consistent quality while increasing production capacity is a significant challenge. Biopolymers must demonstrate consistent superabsorbent performance under a wide range of environmental conditions, including changes in temperature, humidity, and pH.

Superabsorbent polymers (SAPs) are built using acrylamide, which offers the advantage of excellent salt resistance in terms of raw material performance [8]. Acrylamide is a synthetic monomer that has several hydrophilic functional groups in the form of $-NH_2$, $-CH$, and $-C=O$. Rice plants belong to the genus *Oryza L.* and many are used as a source of staple food in several countries around the world, especially Asia. Indonesia is one of the largest rice producers in the world with a total harvest of 30,634,846 t/year ($t = \text{tonne} = 10^6 \text{ g}$) [9]. The high amount of rice production will certainly cause the by-product of the production process in the form of rice-straw to increase. Rice straw waste is estimated to contribute as much as 43 % of agricultural waste in Indonesia, with 31 % - 32 % used in making fertilizer, 7 % - 8 % used as raw material for the pulp and paper industry, while the rest is not utilized optimally [10]. Waste in the form of rice straw has potential as a raw material in the manufacture of superabsorbent and offers environmentally friendly for environmental cleanup, also to emphasize the repurposing waste. Utilizing rice straw cellulose can potentially be cost-effective as it takes advantage of a waste product that would otherwise require disposal. This could result in lower raw material costs compared to synthetic alternatives that require specialized production processes. With its enormous output and area, rice has a fantastic source of renewable energy. The cell wall of straw typically contains 35 % - 50 % cellulose, 20 % - 35 % hemicellulose, and 10 % - 25 % lignin as its biomass [11]. With cellulose from rice straw as a property of the materials, the resulting polymer can be used as bioplastic, even more the plastic material could be decomposed through microorganisms, became an environmentally friendly compound [12].

The content of cellulose in rice straw is high enough that it can be used as a raw material in the manufacture of superabsorbent. The addition of a crosslinking agent (*crosslink agent*) is also required in the process of grafting in the manufacture of superabsorbent to help improve the thermal properties of superabsorbent and produce polymer networks that can absorb water. Cellulose has a potential to be used as the material of hydrogel, because it is forming by the cellulose microfibrils and water with

an interconnected porous structure [13]. Citric acid is a material that has the potential as a crosslinking agent because it has a carboxyl group that can form bonds with the hydroxyl groups of cellulose. The concentration of initiators used is another important parameter in the preparation of superabsorbent that will affect the characteristics of the superabsorbent produced. Optimizing parameters such as temperature, pressure, mixing speed, and duration can significantly impact the properties of the biopolymer. Besides that, techniques like annealing, quenching, drying, or curing may further enhance the properties of the biopolymer. The initiator has the function of forming free radicals in cellulose because the initiator is related to the number of free radicals that will be formed. Potassium persulfate (KPS) is the initiator used in this process. The composition of the surfactants obtained from the reaction using the KPS initiator is denser because the structure of the potassium cation is smaller than ammonium peroxydisulfate (APS), so the repulsive force generated will be smaller. This study aims to analyse the effect of adding cellulose fibre to rice straw and additives in the form of a crosslink agent and the initiator of the quality of the resulting superabsorbent. The addition of cellulose fibre and rice straw crosslink agent in the form of citric acid is expected to produce superabsorbent biopolymers with the ability to absorb and swell against high water.

Materials and methods

The raw materials and chemicals used in this study were rice straw, acrylamide, potassium persulfate (KPS), citric acid, aqua Dest, NaOCl, NaOH 5 %, H_2SO_4 1 N, H_2SO_4 72 %, nitrogen gas, solution buffer acetate pH = 6, 9, and 12. Rice straw was obtained from Martapura City, East Ogan Komering Ulu Regency, South Sumatra.

Raw material preparation

Rice straw waste is washed first to remove any remaining dirt. Then the rice straw that has been washed is cut to a size of 2 cm. After being cut, the rice straw is then mashed using a blender.

Production of cellulose

Rice straw fibre weighed as much as 50 g with 5 % NaOH solution and heated for three hours at 80 °C while continuously stirring with a magnetic stirrer. The delignification results were filtered, and the residue was washed using aqua Dest until neutral pH and then dried at room temperature. The straw fibre was then heated with NaOCl solution for one hour at a 60 °C with a magnetic stirrer. The residue is rinsed using aqua Dest until the pH becomes neutral, then filtered and dried in the oven at 100 °C for two hours. The rice straw cellulose was then sieved through a 50-mesh sieve.

Superabsorbent biopolymer synthesis

Cellulose with a concentration of 10 % dissolved with aqua Dest 200 mL was then put into a three-neck flask with a volume of 500 mL and stirred at a stirring speed of 250 rpm, then heated until the temperature reached 70 °C. The inside of the three-neck flask flowed with nitrogen gas for the first fifteen minutes, then KPS powder was added with a variation of 0.2 %; 0.4 %; 0.6 %; and 0.8 % which has been dissolved in 10 mL aqua Dest when the temperature drops to 60 °C - 65 °C for 15 minutes with a magnetic stirrer. Furthermore, acrylamide which has been dissolved with 40 mL aqua Dest, and citric acid with variations

of 0 g, 2 g, 4 g, 6 g, and 8 g were added to a three-neck flask and polymerization was carried out at 70 °C with reaction time of three hours. The polymerization results are then washed and dried in an oven to obtain a product in the form of a superabsorbent biopolymer.

Analysis

Method analysis chesson

A sample of 1 g of rice straw has been carried out pretreatment by delignification using NaOH and bleaching using NaOCl will be analysed using the method *Chesson Datta* to find out the cellulose content found in rice straw [14].

Fourier Transform InfraRed (FTIR) analysis

The best rice straw cellulose and superabsorbent samples will be analysed using Fourier Transform Infra-Red technology. The analysis was carried out to identify the functional groups in rice straw cellulose as a material and superabsorbent polymer as a product.

Scanning electron microscope (SEM) analysis

SEM testing is carried out to observe the surface of solid objects directly with a magnification of 500x - 5000x. Observation of morphology was carried out to observe the smoothness, pores, and cracks on the surface of the hydrogel. SEM analysis was carried out using a Tescan Vega 3 SEM tool.

Absorption capacity analysis

The polymerized superabsorbent biopolymer product was weighed at 0.1 g and then put into a 10 mL measuring cup and added with 6 mL aqua Dest at room temperature for 24 hours. The liquid that was not absorbed was then removed from the measuring cup and left in an inverted position until no more liquid dripped. The superabsorbent is then weighed as a mass after sorption. The absorption capacity is calculated using equations:

$$\text{Absorption capacity} = \frac{W2 - W1}{W1} \cdot 100 \% \quad (1)$$

where: W1 - initial mass of superabsorbent polymer before absorption capacity test, W2 - final mass of superabsorbent polymer after absorbing water.

Ratio analysis swelling

The polymerized superabsorbent biopolymer product was weighed as much as 0.1 g and then put into a 10 mL measuring cup and added 5 mL of the solution buffer acetate pH = 6 and soaked at room temperature for 24 hours. The liquid that was not absorbed was then removed from the measuring cup and left in an inverted position until no more liquid dripped. The superabsorbent is then weighed as the after-mass swelling. Repeat for the solution buffer acetate with pH = 9 and 12. Ratio swelling superabsorbent biopolymer is calculated using equation:

$$\text{Ratio swelling} = \frac{W2 - W1}{W1} \quad (2)$$

Result and discussion

FTIR characterization

Before reacting to the pure rice straw cellulose and becoming a superabsorbent, an FTIR test is carried out first to be able to determine the functional groups contained in the cellulose fibre of rice straw, shown in Figure 1.

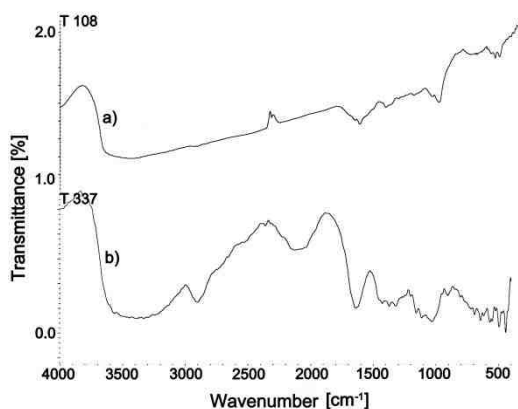


Fig. 1. FTIR results of: a - acrylamide-cellulose rice straw/citric acid 6 g/KPS 0.6 % biopolymer superabsorbent and b - cellulose from rice straw fibre

Based on Figure 1a, it is known that there are polyacrylamide groups that have hydrophilic properties. In the wave range of 3000 cm^{-1} - 3500 cm^{-1} there are -OH and -NH groups which indicate that acrylamide has been crosslinked on the backbone from cellulose. The -NH₂, -CH, and -C=O groups are formed by acrylamide monomers. In addition, there is an -OH group derived from citric acid as a crosslinking agent. The -OH group from the results of the FTIR analysis indicates that cross-links in the polymer are formed.

Percent transmittance on FTIR results of variation potassium persulfate 0.6 % and 6 g superabsorbent citric acid tended to decrease or decrease due to the presence of hydroxyl groups in the form of -OH groups which are reinforcing groups in the formation of superabsorbent. The -OH group is a group that is bonded to each other between monomers, crosslink agents, and cellulose.

FTIR graph of the occurrence of the superabsorbent formation process, namely in the early stages of initiation, the initiator will be decomposed to form free radicals. After that the propagation reaction occurs, and cellulose is added to form free radicals. Then proceed to the termination stage will bind to crosslink agents and monomers and -OH groups bonded together. It can be seen from the FTIR graph that the more -OH groups, the lower the transmittance percentage, thus the absorbance of the OH groups will be greater. It has been proven that the transmittance percentage of the -OH group is getting smaller, meaning that a termination bond is formed, and it succeeds in forming a good superabsorbent. Based on Figure 1a, the reading of the infrared spectrum from the results of the FTIR analysis of superabsorbent biopolymers can be seen in Table 1.

Table 1

Readout of biopolymer acrylamide-cellulose rice straw analysis results/citric acid 6 g/KPS 0.6 %

Wavenumber [cm^{-1}]	Functional groups	Information
3447.28	-OH, -NH ₂	From citric acid (<i>crosslink</i>) and acrylamide
2284.90	-CH	From cellulose
1660.33	-C=O	From acrylamide and citric acid
1455.66	-C-N	From acrylamide and cellulose
1040.41	-C-O	From acrylamide and cellulose

Based on Figure 1b, the results of the FTIR analysis were carried out at wave numbers of 400 cm^{-1} - 4000 cm^{-1} . Then at 2500 cm^{-1} - 4000 cm^{-1} the cellulose consists of three main groups namely O-H, N-H, and C-H groups. FTIR spectrum readings on rice straw fibre cellulose are shown in Table 2.

Table 2

Cellulose FTIR result reading

Number of waves [cm^{-1}]	Functional group	Information
3329.39	O-H	The hydroxyl group of cellulose
2900.50-2127.87	C-H	C-H <i>stretching</i> on CH ₂ from the oxidation of the CH group ₂ OH
1639.95	C=C	From hemicellulose and lignin
1372.55	C-H	C-H <i>bending</i> from lignin
1028.66	C-O-C	from lignin
902.35	C-H	There is bond absorption β -glycosidic

The result of the reading of the FTIR spectrum graph for cellulose derived from rice straw is the presence of -OH groups. Table 2 shows the infrared absorption range of rice straw fibre. In the alkalization process, hydroxyl groups are removed through reaction with sodium hydroxide, thereby reducing hydrogen bonding. As a result of the alkalization process, the -OH concentration increases. This OH group is indicated by the presence of absorption at wave number 3329.39 cm^{-1} . The reaction that occurs in the alkalization process is that the NaOH solution dissociates into Na⁺ and OH⁻ and reacts with lignin. OH ions react with the H groups of lignin to form H₂O. This causes the O group to become a free radical and react with C to form C-O-C. Group C already has four "arms", hence many groups separate from group O. This reaction produces two separate benzene rings, each ring containing a reactive O group. These reactive O groups react with Na⁺ and dissolve alkaline solutions, resulting in lignin being lost during the rinsing process.

Additionally, H₂O is also produced during this reaction. Because cellulose is hydrophilic, H₂O binds to cellulose, and caused the increasing concentration of O-H bonds. The number of waves 2900.50 cm^{-1} showed the presence of cellulose compounds, when heated with alkali the components of these cellulose compounds did not disappear. At the number of 1639.95 cm^{-1} vibration appears with a C=C spectrum, this indicates that there is a delignified lignin compound. The number of waves 1372.55 cm^{-1} showed the presence of C-H bending [15]. Next the number 902.35 cm^{-1} shows absorption β -glycosidic [16]. β -glycosidic is the original absorption connected from glucose to cellulose.

Surface structure morphology of cellulose and superabsorbent in testing scanning electron microscope (SEM)

The surface structure of rice straw cellulose and superabsorbent were analysed using SEM with image magnification 1000x (Fig. 2). Figure 2a shows the surface morphology of rice straw cellulose fibre which visually looks a bit rough and irregular.

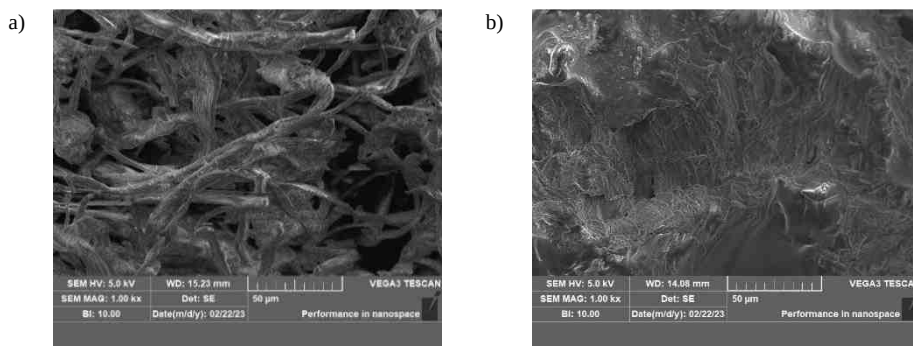


Fig. 2. a) SEM results of rice straw cellulose with magnification 1000x, b) SEM results of superabsorbent polymer with magnification 1000 x (KPS: citric acid = 0.6 % : 6 g)

The alkaline treatment during the delignification process causes the cellulose fibres to shrink and the outer layer to roughen and crack in certain parts of the inner structure showing fibril strands. This fibril structure will then decrease in size due to the process of bleaching [17]. The appearance of crystalline cellulose is caused by the formation of inter- and intramolecular H bonds between hydroxyl groups. The H bonds will hinder the free movement of the cellulose chains and are bound in the network structure [18]. Porosity in rice straw cellulose ranges from 66.8 %. Figure 2b shows the surface morphology of the superabsorbent biopolymer with the formulation of acrylamide-cellulose rice straw/citric acid 6 g/KPS 0.6 %. Visually, there are many concavities with a shell-like polymer coating. The thickness surface of crosslinked shell allows SAP to have a high absorption capacity because it prevents the diffusion of dissolved particles in the polymer [19]. In addition, this morphology also prevents the deformation of the swollen particles under load, demonstrating the extraordinary absorption ability of superabsorbent biopolymers.

The surface of the superabsorbent biopolymer looks denser, this indicates that there has been a bond formation between the monomer and crosslink agent. Meanwhile, the surface that was visible when it was still in the form of cellulose still had many gaps. In addition, the shape of the superabsorbent material no longer looks like fibre but fine sheets with a tight porosity of 62.8 %. To improve soil water retention, SAPs are employed in agricultural and environmental engineering. In these conditions, the SAP in the soil swells, the water will be trapped in the pores of SAP which affects the SAP's capacity to absorb water [20]. The characteristic that builds is the main factor of the polymeric material that is formed to act as a superabsorbent with high absorption capacity and swelling ratio [21]. The good structural integrity of the hydrogel is shown by the smooth surface of the hydrogel and the homogeneous hydrogel matrix with less pores on its surfaces [22].

Effect of initiator and concentration crosslink agent on water absorption capacity

Superabsorbent polymer is a polymer network that can absorb and absorb or retain a large amount of liquid that is much heavier than its initial weight in a short time [23]. This material can be in the form of polymers dissolved in water using radiation or a crosslink agent. Cross-linking during reaction polymerization takes place is important for can produce a network of polymers that can absorb water. The ability of hydrogel to absorb water will be affected by the free functional groups in the structural network molecules that can bind air. Superabsorbent polymer is made using rice straw cellulose and acrylamide, both materials will increase the water absorption ability of the superabsorbent. This is because these two materials have hydrophilic molecules (binding water) and a high affinity for water. The effect of the addition of citric acid and potassium persulfate on the water absorption capacity can be seen in Figure 3.

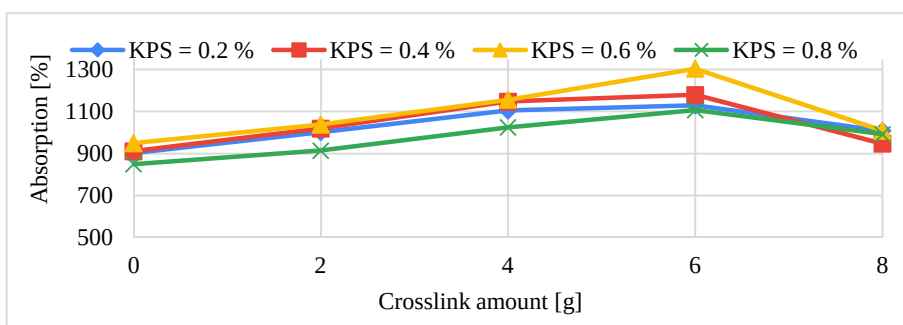


Fig. 3. Results of water absorption capacity in superabsorbent products, with the concentration of: KPS = 0.2 %; 0.4 %; 0.6 %; 0.8 %

At concentrations crosslink is low, the absorption capacity of the resulting polymer superabsorbent will also be low [24]. This matter is due to the number of crosslink agents which is little or low when forming superabsorbent polymers. Super absorbents can absorb water up to 1500 g/g dry SAP polymer exceeds the ability of traditional absorbent materials such as charcoal and clay [25]. Polymer without the crosslink will dissolve in water, then will not be counted in the absorption capacity figure. If the concentration of crosslink height, capacity absorption will be high because crosslinks which is formed in large quantities so that the polymer does not dissolve in water and has a high absorption capacity. Furthermore, when the concentration crosslinks large, the absorption capacity will decrease. The best results are seen in concentration crosslink which depends on concentration of potassium persulfate inside the polymer. The further addition of the KPS initiator of 0.8 % will cause the absorption of the superabsorbent to decrease or the polymer chain to become shorter. This is because the number of polymer chains will not form a polymer network and crosslink. As the number of initiators increases, the degree of superabsorbent swelling decreases. Increasing the number of initiators will shorten the chain, resulting in SAP being lighter. Also, an excessive increase in the number of initiators and cross link agents can also cause swelling and absorption capabilities to decrease [26].

This is because too many free radicals will cause chain-breaking reactions so the polymer graft chains become shorter and there is a dense network structure that will be difficult to form [27]. Based on Figure 3, the optimum point is achieved with an initiator

variation of 0.6 % and an additional crosslink of 6 g with a capacity of 1304.3 %. At this point, it shows a relatively higher water absorption ability when compared to initiator concentrations of 0.2 %, 0.4 % and 0.8 % with variations of crosslinking agents 0 g, 2 g, 4 g and 8 g. This is due to the addition of the initiator KPS can affect the water absorption capacity of the superabsorbent.

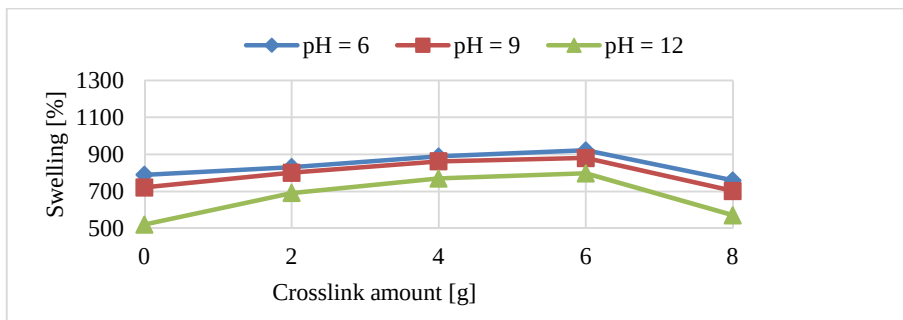
Effect of initiator and concentration crosslink agent on swelling ability

In this study, a test was carried out to determine the effect of absorption of liquid solution buffer acetate pH = 6, 9, and 12 by soaking for 24 hours. This test uses a solution buffer acetate by mixing (CH_3COOH) into its salt solution (CH_3COONa) with pH = 6, 9, and 12. Test using a solution buffer done since the solution buffer has ionic strength (extra ionic) and helps stabilize the pH. Figure 4 shows the higher the pH of the solution, the capacity value swelling produced will below.

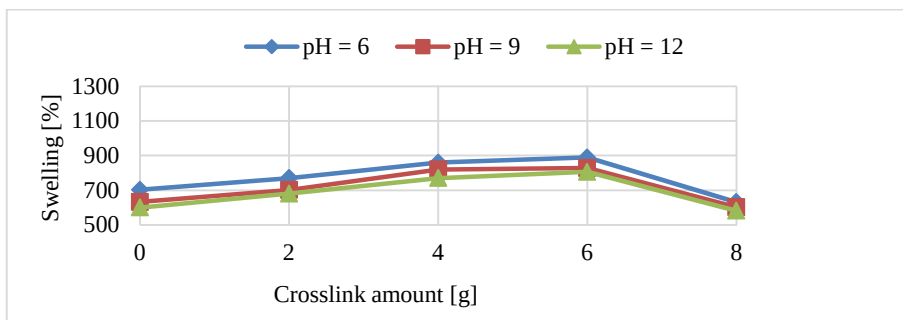
Increased pH differently influenced the swelling degree in the case of the hydrogel series in which the concentrations of the monomer and crosslinking agent were varied [28]. Based on Figure 4 the optimum point is achieved with an initiator variation of 0.6 % (Fig. 4c) and an additional crosslink of 6 g with a capacity of 10.26 (w/w). At this point, it shows a relatively higher water absorption ability when compared to initiator concentrations of 0.2 %, 0.4 % and 0.8 % with variations of crosslinking agents 0 g, 2 g, 4 g, and 8 g. Superabsorbent products with value swelling the highest occurred in a solution of pH = 6. This is due to the influence of the crosslink *agent*, the carboxylate found in citric acid can cross-link with the amide group found in acrylamide so that it can improve the physical properties of the superabsorbent. Citric acid as a crosslink can exchange ether reactions with hydroxyl groups on super absorbents in acidic conditions. Crosslinked citric acid and cellulose in superabsorbent appeared as a swelling hydrogel [29]. The more crosslinking agents added to the reaction, the value swelling will decrease.

The main chain in this polymerization reaction is acrylamide with a pH range of 6.5. At a pH value of 6, the amide group of acrylamides hydrolysed into a carboxylic acid group. Thus, the increase in anionic carboxylic groups promotes electrostatic forces in the polymer network resulting in increased solubility absorption of the hydrogel. Acrylamide has the NH_2^+ amide functional group which can release H^+ . This reaction is a weak acid hydrolysis which will make the superabsorbent, including the acid solution, protonated to form cation bonds and make the capacity value swelling good [30].

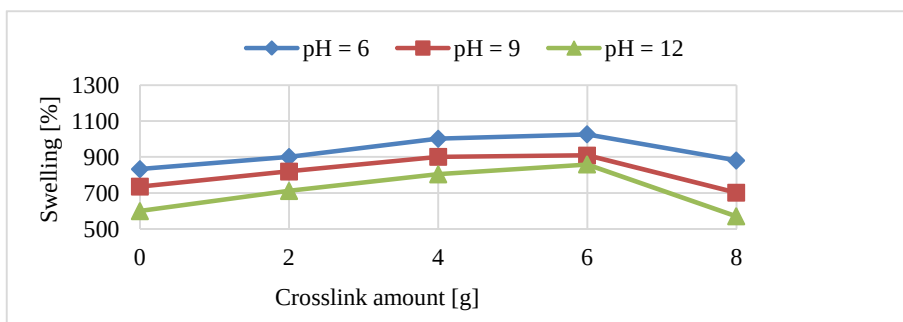
a)



b)



c)



d)

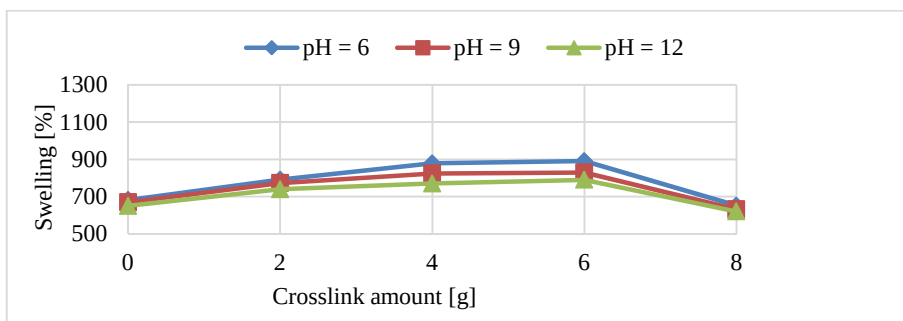


Fig. 4. Relation between initiator and crosslink agent on the effect of swelling in pH = 6; pH = 9; and pH = 12 with initiator amount of: a) 0.2 %; b) 0.4 %; c) 0.6 %; d) 0.8 %

Conclusion

Rice straw cellulose has the potential to be used as a natural monomer in the manufacture of superabsorbent biopolymers because it contains 49.59 % cellulose. The highest absorption capacity and value swelling was found in the addition of 0.6 % initiator and crosslink agent 6 g with an absorption capacity of 1304.3 % and a value of swelling 10.26 (w/w). With the addition of initiator concentration and crosslink agent more than the optimum point, there will be a decrease in absorption capacity and value swelling due to the role of the initiator and crosslink agent no longer functioning, causing damage to the material properties of the superabsorbent biopolymer. The porosity value of rice straw

cellulose was obtained at 66.8 % and superabsorbent at 62.8 %. This decrease in the porosity value indicates the occurrence of bonds between the monomer and crosslink agent which causes the volume of free space to be reduced.

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