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Improvement of PVA-Glucomanan-Acrylamide Hydrogel as Base Material of Immobilization

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Abstract

Hydrogel products are currently widely used in various fields, one of which is agriculture. Most hydrogels are made of synthetic polymers because they have good absorption but are not biodegradable. Glucomannan is a natural polymer that is able to absorb large amounts of water and biodegradable but it is difficult to maintain water content. One method to improve the mechanical properties of hydrogels is by modifying the hydrogels through the formation of an interpenetrating network (IPN) between natural polymers and synthetic polymers. In this study, the IPN hydrogel based on polyvinyl alcohol and glucomannan and acrylamide as a crosslinker was made by combining freeze-thaw and gamma irradiation techniques. The results showed that the hydrogel water absorption after immersion for 24 hours was 311.09% where the weight of glucomannan was 1 g and acrylamide was 0.75 g and the irradiation dose was 30 kGy. The result of cumulative release test of paraquat immobilized into the hydrogel was 12.00% within 10 days. This indicates that the PVA-glucomannan-acrylamide hydrogel can be used as a controlled paraquat release matrix so as to minimize the effect on the overuse of pesticides.

Keywords: Controlled release, Gamma irradiation, Glucomannan, IPN hydrogel.

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1. INTRODUCTION

Hydrogel products are a group of polymer materials with a hydrophilic structure that can hold a large amount of water in its three-dimensional network so that it is widely used in various fields (Cipriano et al., 2014). Most hydrogels are made from synthetic polymers such as polyvinyl alcohol and acrylamide because they have good absorption. However, synthetic polymers are not biodegradable and toxic, so they are not suitable for agriculture or any application related to human consumption. Therefore, developing safe and environmentally friendly hydrogel products is necessary to carry out (Demitri et al., 2013). Glucomannan is a natural polymer with a hydrophilic group, abundant, economic, easy to form gels and films, high viscosity, biodegradable, biocompatible, biofunctional, and bioabsorbable. Still, it is difficult to maintain water content (Zhang et al., 2014). One way to improve hydrogel mechanical properties is by modifying the hydrogel by forming an interpenetrating network (IPN) between synthetic and natural polymers. An interpenetrating network (IPN) combines two or more polymers with different characteristics, including a polymer network without any covalent bonds between networks (Wu et al., 2011). The IPN hydrogel can be prepared by mixing free radicals, gamma radiation, and UV radiation (Jayaramudu et al., 2017).

Li et al. (2015) reported a double network hydrogel of glucomannan crosslinked with two synthetic polymers, polyacrylamide, and polyvinyl alcohol, using a cycle freezing-thawing method obtained hydrogels with good

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strength and extensibility. Nevertheless, there are no data on the swelling properties of the hydrogel. One of the important characteristics of hydrogel is its swelling behaviour. The rate and degree of swelling hydrogel, solvent uptake, and desorption are essential parameters that affect the release patterns of solvents from these polymeric networks.

One of the techniques to improve this swelling property is the irradiation technique. Polymerization radiation has many advantages and conveniences over conventional chemical and photochemical methods. This method is an additive-free, environmentally friendly, and simple process, it can occur at room temperature, and the crosslinking level can be easily controlled by changing the irradiation conditions (Francis et al., 2009). Hydrogels formed by the irradiation method have potential biomedical applications due to the absence of foreign and toxic additives such as chemical initiators, cross-linkers, and others. Another advantage of this method is that the resulting products are sterilized simultaneously during the irradiation process (Karadağ et al. 2001). Therefore, in this study, a hydrogel based on polyvinyl alcohol, glucomannan, and acrylamide was synthesized to produce a hydrogel that is biodegradable, strong, and has high absorption. We know that the synthesis of hydrogel based on PVA, glucomannan, and acrylamide using gamma irradiation has not been reported. In this case, the ^{60}Co gamma irradiation technique was used.

Hydrogel has various benefits, one of which is in agriculture as a material for immobilizing pesticides. Immobilization is a technique to curb or trap active ingredients in a matrix where their release can be controlled. Generally, pesticide immobilization on hydrogels can be done post-loading and situ loading (Erizal, 2019). The advantages of controlled release are extending the release time of active ingredients, using fewer doses, reducing phytotoxicity, reducing environmental pollution, and easy handling (Rashidzadeh and Olad, 2015). Sun et al. (2016) have immobilized several types of pesticides in the hydrogel matrix based on lignin and poly (acrylic acid), showing that the amount of pesticides immobilized in the hydrogel matrix is quite high, and its release is also monitored relatively quickly. However, the synthesis process of this hydrogel uses a grafting method that uses a chemical initiator such as H_2O_2, which is known to be toxic and dangerous. In 2018, Erizal et al. also immobilized propranolol HCl with gamma irradiation on the PVA-alginate matrix, resulting in a decrease in the release of propranolol HCl due to an increase in the irradiation dose (Erizal et al., 2018).

In this study, hydrogel-based on PVA-glucomannan-acrylamide (PVA-GM-AAM) was synthesized with variations of the weight of glucomannan and acrylamide and gamma irradiation doses. Various concentrations of glucomannan as natural polymer and acrylamide as cross-linker and variation in irradiation doses were carried out to determine the optimum condition of the polymers composition to form a hydrogel with high absorption and can maintain water content in it. Therefore, this study aims to synthesize hydrogel from PVA-glucomannan-acrylamide solution and determine its ability to absorb water and active material such as pesticides. In application, pesticides on the PVA-GM-AAm matrix were irradiated at gamma doses of 10, 15, 20, and 30 kGy, and without gamma irradiation, were tested for their release. Their characteristics were carried out using Fourier Transform Infra-Red (FTIR) spectrophotometers and Scanning Electron Microscopy (SEM). The hydrogel is expected to be applied as environment-friendly material in agriculture as a material for immobilizing pesticides.

2. MATERIALS AND METHODS

Materials

The monomers used included poly(vinyl alcohol), acrylamide was produced from Merck, glucomannan was obtained from local industry, and distilled water was used to synthesize and determine the hydrogel. Paraquat dichloride was produced from Merck and used for the cumulative release test.

Procedures

Synthesis of PVA-GM Hydrogel

PVA-Glucomannan hydrogels were prepared using a combination freeze-thaw and gamma irradiation method. 10% (w/v) PVA solution was heated at 121 ºC using an autoclave. PVA solution was mixed with glucomannan with variation of the weight of 0.5; 1.0; 1.5; and 2.0 g. The mixture is stirred until homogeneous. Each mixture was packed in polypropylene (PP) plastic bag and sealed.

The packed mixtures was frozen in the freezer and then melted at room temperature. The freeze-thaw process was carried out for 3 cycles and then irradiated using gamma rays (PAIR-BATAN irradiator) at doses of 10, 15, 20 and 30 kGy/hour. The irradiated hydrogel is cut into small pieces and then dried in oven at 60 °C until dry. Furthermore, the dried hydrogel was tested for water absorption to obtain optimum concentration of glucomannan (Erizal and Abidin, 2011).

Synthesis of PVA-GM-AAm Hydrogel

The optimum weight of glucomannan was added to 10% (w/v) PVA solution, then acrylamide was added with variations in the weight of 0.25; 0.50; and 0.75 g. The mixture is stirred until homogeneous. Each mixture is packed in PP plastic bag, sealed, and then frozen in the freezer and melted at room temperature. The freeze-thawing process was carried out for 3 cycles and then irradiated using gamma rays at doses of 10, 15, 20, and 30 kGy/hour. The irradiated hydrogel was cut into small pieces and then dried in an oven at 60 °C until dry and milled to a size of 60-80 mesh for characterization using FTIR (Erizal et al., 2015).

Determination of Hydrogel

The composite hydrogel was evaluated for water absorption, gel fraction, chemical structure, and morphology properties. Water absorption and gel fraction of the hydrogel was determined using a hydrogel sample that had been dried for 24 hours and obtained a constant weight (W_0). The sample was immersed in distilled water, and the wet weight (W_t) was measured every 1 hour for 5 hours and continued at 24 hours to determine water absorption. Then, the water absorption capacity was calculated using equation 1.

$$\text{Water absorption } (\%) = \frac{W_t - W_o}{W_o} \times 100\% \qquad (1)$$

Furthermore, to determine gel fraction, the hydrogel, which had been soaked in distilled water for 24 hours, was dried in an oven at 60 °C to constant weight (W_1). Then, the gel fraction is calculated by equation 2. The test was repeated three times.

$$\text{Gel fraction } (\%) = \frac{W_1}{W_0} \times 100\% \qquad (2)$$

FTIR Shimadzu Prestige-21 model was employed to analyze the functional group of cross-linked polymers. Samples were prepared for FTIR analysis by preparing KBr powder (1:200) and then scanning the range of wavenumbers 4000-400 cm^{-1}. The microstructure of a dry hydrogel sample was investigated using a scanning electron microscope (SEM) Hitachi S3400.

Pesticide Immobilization in PVA-GM-AAm Hydrogel Matrix

The mixture solution of PVA-GM-AAm has been added with paraquat 25 ppm. A 17 mL glucomannan-PVA-acrylamide solution was added into the tube, and then added 10 mL paraquat. The mixture is stirred until homogeneous using a vortex. The mixture was frozen in the freezer, melted at room temperature for 3 cycles, and irradiated with gamma at various doses of 10, 15, 20, and 30 kGy/hour (Erizal et al., 2018).

Determination of Pesticide Cumulative Release

The cumulative release of paraquat was determined by immersing loaded paraquat hydrogel in 100 mL of distilled water at room temperature. Pesticide measurements were carried out every 1 hour for 8 hours and continued for 1; 4; 7; and 10 days. For each solution measurement, 5 mL of the sample were taken, and the solution was replaced with the same volume of 5 mL. The absorbance of the solution was measured using a UV-Vis spectrophotometer at the maximum wavelength. The cumulative percentage release (R) of paraquat from the hydrogel was calculated according to equation 3 (Sun et al., 2016).

$$R\ (\%) = \frac{(M_t + \sum M_{t-1})}{M_0} \times 100\% \qquad (3)$$

M_t = the released concentration of paraquat at time t (ppm)
M_{t-1} = concentration of paraquat at the end of time (ppm)
M_0 = initial concentration of paraquat (ppm)

3. RESULTS AND DISCUSSION

Effect of Adding Glucomannan

Glucomannan is a natural polymer obtained from the extraction of the konjac plant as Amorphophallus muelleri. The

addition of glucomannan is intended to make the hydrogel biodegradable (Alonso-Sande et al., 2009). The more glucomannan is added, its biodegradable properties would be better. The results of the PVA-Glucomannan hydrogel synthesis with various glucomannan weights show that the water absorption capacity has increased along with the increase in the weight of added glucomannan. The highest absorption at 24 hours was the addition of 1.0 gr of glucomannan with a percentage of absorption of 151.83% for hydrogel without irradiation, and hydrogel with irradiation doses of 10 kGy, 15 kGy, 20 kGy, and 30 kGy were 158.46%, 167.28%, 181.04%, and 195.90% respectively. The addition of glucomannan of more than1 gr decreased absorption. It is due to the addition of more than 1 g of glucomannan increase in hydrophilic groups, but the density of the distance between intramolecular and intermolecular in the hydrogel matrix also increases, resulting in difficult water entering the hydrogel matrix network so that water absorption decreases (Tomar et al. 2007)

Cross-linking by irradiation is relatively the same as the freeze-thawing process, which does not require an initiator or catalyst. The physical properties of hydrogels resulting from the irradiation process are relatively better than those of the freeze-thawing process. It is because, in the irradiation process, chemical cross-links are formed. Jayaramudu et al. (2017) argue that PVA is a semi-IPN structure-forming material because it has a good mechanical structure. The formation of semi-IPN occurs when exposed to gamma rays; PVA will first form cross-links (Figure 1) because it has a relatively smaller size than glucomannan and is sensitive to radiation.

Effect of Addition of Acrylamide

Acrylamide is a hydrophilic agent in polymer modification, one of which is hydrogels' manufacture. The irradiated PVA, glucomannan, and acrylamide solutions will form a three-dimensional network in which acrylamide will be a cross-linking agent. The synthesized PVA-Glucomannan-Acrylamide hydrogel was tested for its absorption capacity. The effect of adding acrylamide weight can be seen in Figure 2. The addition of acrylamide affects hydrogel absorption. Figure 1 shows that the increase in acrylamide weight increases the absorption of the hydrogel. The acrylamide weight of 0.75 g shows the highest absorption capacity in all the irradiation doses. It is because the addition of acrylamide increases its hydrophilicity. Polyvinyl alcohol and glucomannan contain -OH groups and acrylamide contains -$CONH_2$ groups, so the more acrylamide is added, the more hydrophilic groups in the polymer chain will increase and water absorption also increases. In addition, it can be seen that with increasing irradiation dose, water absorption also increases. After 24 hours, the hydrogel with an acrylamide weight of 0.75 g showed absorption with an average value of 162.35%, 211.17%, 233.99%, 271.87%, and 311.09% with irradiation doses of 0; 10; 15; 20; and 30 kGy, respectively. The increased absorption indicates that the diffusion process can occur in the hydrogel.

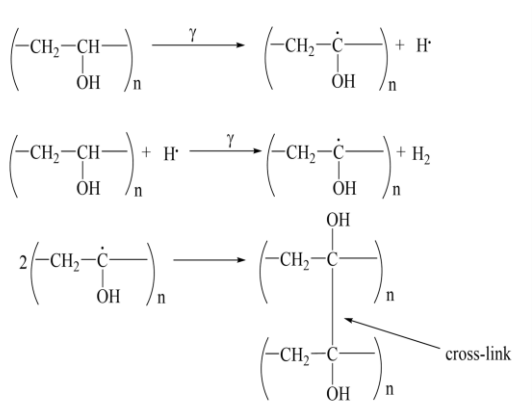

Figure 1. Scheme of cross-linking reaction when PVA irradiated with gamma rays (Sudrajat, 2009)

Gel fraction is one of the important parameters in research related to the hydrogel, which reflects how much (%) the starting material of the polymer or monomer is converted into a hydrogel in the polymerization process. In addition, the gel fraction also showed cross-linking density in the hydrogel (Lin and Gu, 2015). Gel fraction as the efficiency value of the hydrogel synthesis process depends on the sensitivity of the material to the irradiation dose being exposed. The gel fraction also shows the presence of cross-links formed due to gamma-ray irradiation of a polymer (Erizal E, 2019).

The average result of the determination of the gel fraction from the hydrogel with different acrylamide weights and irradiated with varying doses, namely 0; 10; 15; 20; and 30 kGy is presented in Figure 2. It can be seen that there is a decrease in the gel fraction due

to the increasing weight of acrylamide added. Likewise, with an increase in the irradiation dose, the value of the gel fraction also decreased. The gel fraction values with an irradiation dose of 0 kGy were 91.79%, 90.51%, and 87.77% for adding acrylamide weights 0.25 g, 0.5 g, and 0.75 g, respectively. The value of the gel fraction based on the acrylamide weight at all irradiation doses decreased, which was not much different. The cross-linking formed did insignificantly affect the process of releasing the immobilized active ingredient into the hydrogel matrix. The degradation of glucomannan also caused a decrease in the hydrogel gel fraction due to the higher irradiation dose. The degraded glucomannan causes less cross-linking (Kamoun et al., 2015).

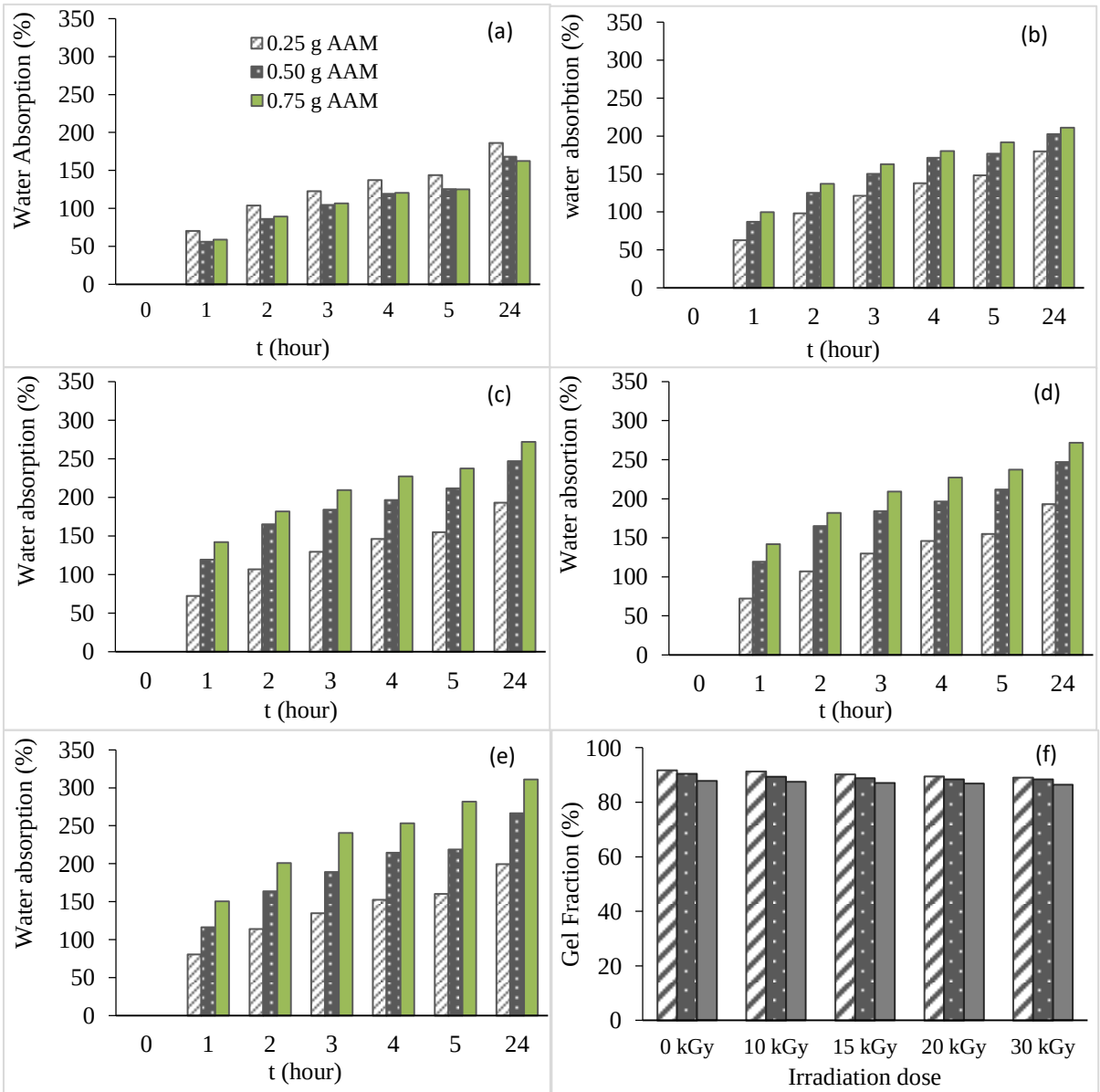

Figure 2. Water absorption of PVA-GM-AAm hydrogel with irradiation dose of 0 kGy (a), 10 kGy (b), 15 kGy (c), 20 kGy (d), 30 kGy (e) and gel fraction (f)

Paraquat Immobilization in PVA-Glucomannan-Acrylamide Hydrogel Matrix

The process of paraquat immobilization into the PVA-Glucomannan-Acrylamide hydrogel as a matrix is carried out by a freeze-thaw process where the paraquat will be trapped through cross-linking that occurs between the monomers of the hydrogel. The melting freeze is carried out for 3 cycles. It results in stronger physical cross-links and increasingly trapped paraquat in the matrix. In addition, this freeze-thaw process causes much water to freeze. When the irradiation process is carried out, the glucomannan in the matrix will be protected, and there will be no degradation (Erizal and Abidin, 2011). The release of paraquat from the PVA-GM-AAm matrix is controlled by paraquat diffusion through the matrix. The release of paraquat trapped in the PVA-GM-AAm matrix starts when the matrix is in contact with water. Then water penetrates the matrix so that the matrix swells. When the matrix swells, there is a diffusion process so that the paraquat leaves the matrix and dissolves in water. The released paraquat was measured using a UV-Vis spectrophotometer to determine the concentration.

Determination of the maximum wavelength is carried out to determine the maximum absorption of the solution. The maximum wavelength of paraquat in water solvents ranges from 256 to 258 nm (Sun et al., 2016; Rashidzadeh and Olad, 2015; Aouada et al., 2010). The maximum wavelength obtained is 256 nm. Standard curves of paraquat were used to determine the concentration of paraquat released. The concentration of paraquat released in water was obtained from absorbance measurements using a UV-Vis spectrophotometer. The standard paraquat curve used has the equation $y = 0.8260x + 0.0346$ with a correlation coefficient value of 0.9955.

Figure 3 shows the relationship between time and the cumulative paraquat released when immersed in water. The ability of the paraquat released in the first hour of the five hydrogels had a relatively small cumulative release of paraquat. It indicates that all paraquat is trapped in the hydrogel matrix.

When the paraquat release test was measured hourly for 8 hours, generally, the release of paraquat increased exponentially. After 24 hours, the released paraquat tends to be slower. Increasing the irradiation dose up to 30 kGy caused the paraquat release to decrease. At 240 hours, the number of paraquat that was released from the matrix was 36.69% (0.07); 33.53% (0.05); 25.17% (0.11); 17.95% (0.04); and 12.00% (0.02) with irradiation doses of 0; 10; 15; 20; and 30 kGy respectively. In this case, with increasing irradiation dose, the release rate of paraquat from the hydrogel decreased. An increase in the dose of irradiation given causes the amount of paraquat released to decrease. This condition indicates that the cross-linking density increases with increasing irradiation dose, which causes the paraquat release rate to decrease. The paraquat will be trapped in the hydrogel matrix longer (Erizal et al., 2018). The 30 kGy doses of PVA-glucomannan-acrylamide matrix had the smallest cumulative release of paraquat compared to other irradiation doses. It shows that the PVA-glucomannan-acrylamide matrix at a dose of 30 kGy can control the rate of paraquat release in the water medium to minimize the excessive use of pesticides, such as reducing toxicity, increase efficacy, reduce the environmental impact of pesticides, and pesticide application. Instead of applying one large dose of pesticide, the same amount released over a while will have a much pesticide effect(Abd El-Rehim et al., 2005).

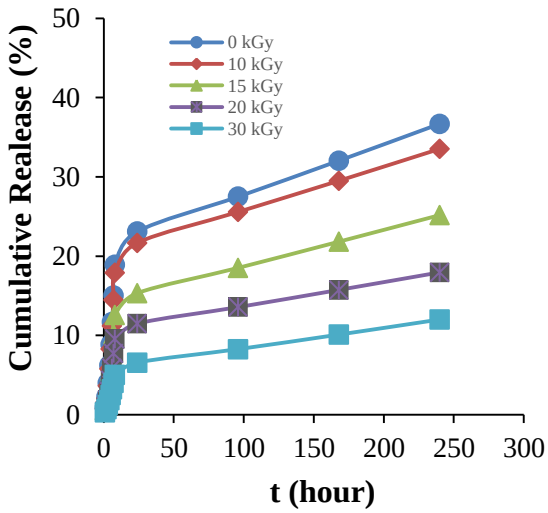

Figure 3. The relationship between time and cumulative realease of paraquat in hydrogel matrix

Characteristics of the PVA-Glucomannan-Acrylamide Hydrogel

PVA, Glucomannan, Acrylamide, and PVA-GM-AAm hydrogel were identified in

their functional groups using an FTIR spectrometer. In Figure 4, the hydrogel wavenumber PVA-Glucomannan-Acrylamide absorption showed a hydroxyl group (–OH) on absorption wavenumber 3450-2700 cm^{-1}, which is a combination of the –OH vibrations of PVA and glucomannan. There is also a typical absorption peak in the hydrogel at 2853-2962 cm^{-1} (-CH vibrations) originating from PVA and glucomannan rich in the –CH group. At the peak of acrylamide absorption, there is initially shown absorption in the 1800 cm^{-1} (C=O) region with a sharp peak, but this is not present in the hydrogel. In the hydrogel, there is a peak absorption in the area of 1350 cm^{-1} (C–O); this is thought to be due to the occurrence of bonds between acrylamide and glucomannan, as shown in the schematic of the approximate reaction that occurs in the formation of the PVA-Glucomannan-Acrylamide hydrogel in supplementary information 1.

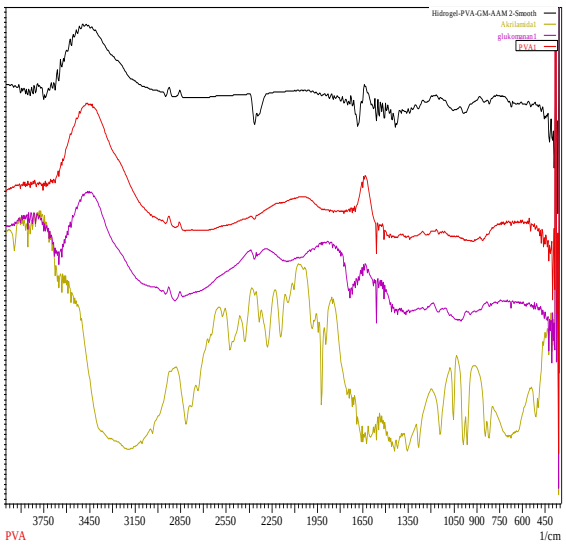

Figure 4. FTIR spectrum of acrylamide (—), glucomannan (—), PVA (—) and PVA-GM-AAm hydrogel (—)

The hydrogel surface morphology was analyzed using a scanning electron microscope (SEM). Micrographs of the cross-section of PVA-Glucomannan Hydrogel, PVA-Glucomannan-Acrylamide Hydrogel without irradiation and with irradiation with the magnification of 100x (left side) and 500x (right side) are shown in Figure 4. In the figure, it can be seen that the surface of the hydrogel with the addition of acrylamide has a porous structure with a pore size that is quite large compared to the hydrogel without the addition of acrylamide. The pore structure shown in the hydrogel cross-section is shaped like a sponge and an interconnected network. It indicates that there is a cross-link in the hydrogel structure. The large pore size in the hydrogel makes it easier for water to diffuse into the hydrogel structure so that the resulting absorption capacity is quite large. PVA-Glucomannan-Acrylamide hydrogel with irradiation shows a denser structure. The irradiation process causes the cross-link density of the constituent elements. The tight structure allows water or the active material in the hydrogel structure to be retained longer, so it is very suitable for use as a controlled release matrix (Rahman et al., 2019).

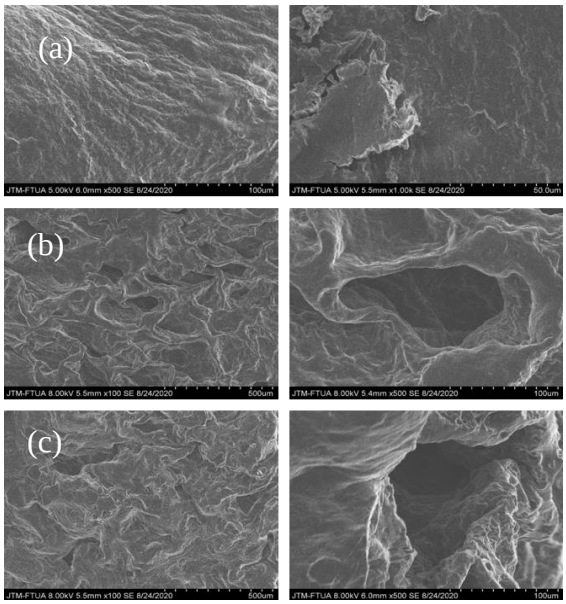

Figure 5. Micrograph of PVA-GM hydrogel with irradiation (a), PVA-GM-AAm Hydrogel without irradiation (b) and with irradiation (c)

4. CONCLUSIONS

Hydrogel based on Poly (vinyl alcohol), glucomannan, and acrylamide were successfully synthesized by forming semi-IPN using the gamma irradiation technique. Paraquat can be immobilized on the PVA-glucomannan-acrylamide matrix, and the release ability of the paraquat from the PVA-glucomannan-acrylamide hydrogel is affected by the gamma irradiation dose. In this research, increasing the dose of gamma irradiation on the PVA-GM-AAm hydrogel caused the cumulative release of paraquat to

decrease. PVA-glucomannan-acrylamide hydrogel with 30 kGy of irradiation dose can be used as a controlled paraquat release matrix.

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