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Effect of Potassium Peroxodisulphate and Microwave Power on Hydrogel Character Based on Banana Peel Waste Using Microwave Grafting Method

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ABSTRACT. *Musa paradisiaca var. raja* peel waste contains cellulose which has the potential to be a raw material for synthesizing hydrogels. This research utilizes acrylamide monomer grafted onto banana peel cellulose backbone using the microwave grafting method to produce hydrogel. The banana peel waste was dried to a constant weight and then crushed into powder. Banana peel powder was reacted with aqueous NaOH in the delignification process and bleached with NaClO to recover cellulose of the banana peel. The aqueous mixture of banana peel cellulose-acrylamide-potassium peroxodisulfate was reacted with variations in KPS concentration and microwave power. The reaction was terminated with a hydroquinone solution addition and washed with acetone. The result of precipitated solid was dried to a powder and then called cellulose-g-PAAM. A homogeneous solution of 2% carrageenan-cellulose-g-PAAM underwent a physical crosslinking process using KCl and $CaCl_2$ solutions after passing through palm oil to form a bead gel. The purpose of this research is to determine the effect of potassium peroxodisulfate (KPS) initiator concentration and microwave power on the swelling capacity in water. The properties of obtained dried bead gels were characterized their functional groups using FTIR and swelling capacity test in water. From this research, it can be concluded that banana peel cellulose was successfully grafted onto acrylamide monomer as evidenced by the FTIR test results. The lower KPS concentration resulted the higher of swelling capacity. The microwave power has no effect on the swelling capacity of bead gels.

1. INTRODUCTION

Hydrogel is an absorbent gel or called superabsorbent that has the ability to absorb and retain large amounts of the aqueous solution. Hydrogels have a three-dimensional cross-linked matrix structure and contain hydrophilic functional groups [1]. In the agricultural sector, hydrogel as a growing medium can reduce irrigation frequency by up to 50%. Hydrogels have also been widely used for biotechnology, sanitary products [2], and fertilizer-controlled release agrochemicals [3].

Hydrogels can be synthesized from natural polymers, and one of them is cellulose. Natural polymers are biodegradable, biocompatible, non-toxic, and have a high capacity to expand [4]. Banana peel waste (*Musa paradisiaca var. raja*) contains 14.58% cellulose so it can be used for synthesizing hydrogels [5]. This research used banana peel cellulose and carrageenan to synthesize hydrogels. This research utilized natural polymers of linear sulfate galactans extracted from red seaweed (*Rhodophyta*), such as *Kappaphycus alvarezii* (known industrially as *Eucheuma cottonii*). Kappa carrageenan has the ability to form a reversible gel and tends to be hydrophilic. Kappa carrageenan has the structure (1,3)-D-galactopyranose and (1,4)-3,6-anhydrous-α-Dgalactopyranose with sulfate groups in certain amounts and positions [6].

The structure of banana peel cellulose needs to be modified in order to function as a hydrogel. In this research, the modification of the structure of cellulose was done by grafting acrylamide (AAM) monomer to the banana peel cellulose backbone with microwave irradiation. This method was chosen because it was an environmentally friendly method with a cleaner approach and better control. In the grafting process, potassium peroxodisulfate (KPS) was used as an initiator. The power regulation was done to speed up the reaction. The powder resulted from the microwave grafting of banana peel cellulose and acrylamide monomer was called cellulose-g-PAAM.

Cellulose-g-PAAM-carrageenan 2% solution was crosslinked to produce a bead gel. The properties of obtained dried bead gels were characterized including their functional groups using FTIR and their swelling capacity in water. This research studied the effect of the concentration of KPS and microwave power on the swelling capacity

bead gel in water and to determine the morphological structure and functional groups of the bead gel.

2. MATERIALS AND METHODS

The materials used were banana peel (*Musa paradisiaca var. raja*) obtained from the waste of local traders in Surakarta, acrylamide brand Merck, aquadest (CV. Agung Jaya), 96% ethanol (CV. Cipta Kimia), hydroquinone ($C_6H_6O_2$) brand Merck, aluminum foil, acetone (C_3H_6O), indicator paper, sodium hydroxide (NaOH) brand Merck, sodium hypochlorite (NaClO) (CV. Cipta Kimia), potassium chloride (KCl) brand Merck, calcium chloride ($CaCl_2$) brand Merck, palm oil (Bimoli brand), carrageenan, and water.

2.1 Made The Banana Peel Cellulose

In this research, the method to produce banana peel cellulose referred to Fitriyani and Abdullah [7] with little modification. Banana peels were cleaned of impurities, washed, and drained at a temperature 70°C until brittle. After the banana peels were dried, they were mashed and sieved with a 200 mesh sieve and stored. The 100 g of dried banana peel powder was put into a beaker glass, and then 1 L of 4% NaOH was added. It was heated at 90°C. Then the filtered product was soaked in 1 L of 3.5% NaClO solution for 24 hours at room temperature. It was filtered and the residue washed off. The sample was isolated with 200 mL of 17.5% NaOH solution heated at 80°C for 1 hour. Then it was filtered and the residue washed off. Bleaching was carried out with 200 mL of 3.5% NaClO solution. It was filtered and the residue was washed and then dried at 50°C until it gained constant weight. After dried, the sample was put in a desiccator to keep it dry. The flow process to produce banana peel cellulose is shown in Fig. 1.

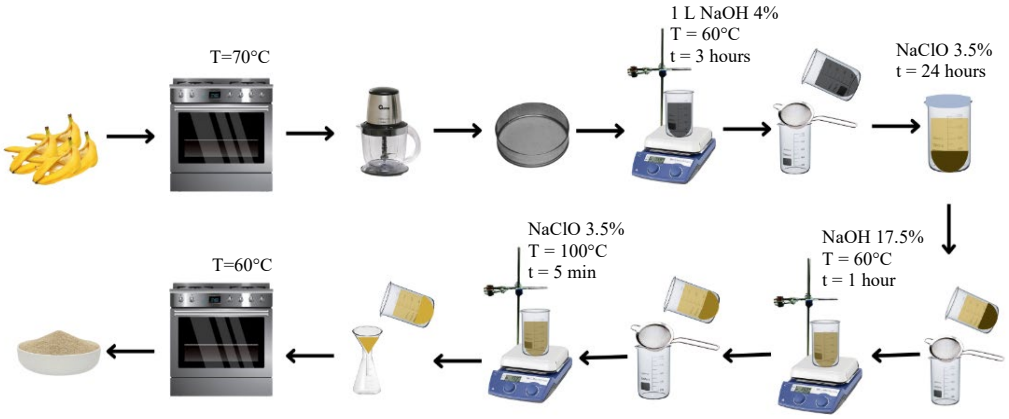

Figure 1. Process production of banana peel cellulose

2.2 Cellulose-g-PAAM Synthesis

In this research, the method to prepare the cellulose-g-PAAM referred to Purwanto et al. [8] with little modification. Two grams of banana peel cellulose were put into a 1000 mL beaker glass. Ten grams of acrylamide (AAM) and 100 mL of distilled water were put into a beaker glass. The mixture was stirred for 30 minutes at 300 rpm. The process for producing grafted polymer was repeated by varying the concentration of the KPS solution (1% wt AAM, 1.25% wt AAM, and 1.5% wt AAM) and certain the microwave output power (540 watts, 630 watts, and 720 watts). Table 1 shows the composition mixture of the initial grafting process. The reaction time was about 500 seconds with the cooling cycle treatment maintained at 60-70°C. The mixture was allowed to stand for 24 hours. The reaction was terminated by adding 20 mL of a 5% w/v saturated hydroquinone solution. After that, the solution was stirred and allowed to stand for 30 minutes. The obtained solid phase was added with excess acetone and then filtered. The solids were dried, crushed, filtered, put in a desiccator, and then labeled. The powder

produced from the microwave grafting process was called cellulose-g-PAAM. The flow process of synthesis the cellulose-g-PAAM is shown in Fig. 2.

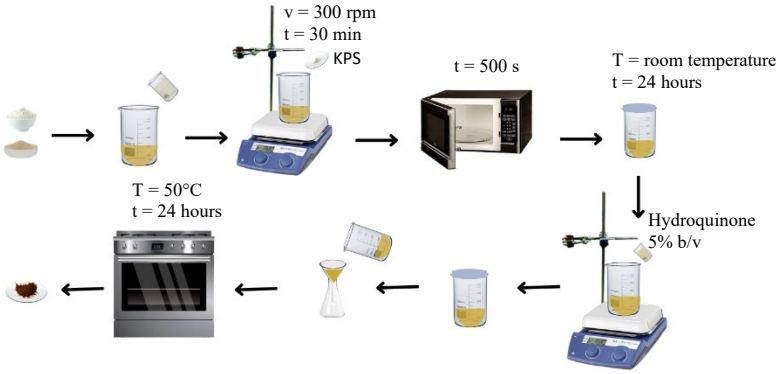

Figure 2. Process production of Cellulose-g-PAAM

2.3 FTIR Test

The FTIR test was conducted to know the changes in the chemical compound's structure due to chemical reactions during the grafting process. These changes were identified by the new peak of the compound groups using FTIR. The FTIR analysis of banana peel cellulose and Cellulose-g-PAAM was recorded by IR-Spirit Shimadzu FTIR Spectrometer with a spectral range between 500 and 4000 cm^{-1}.

2.4 The Bead Gel Production

Bead gel was made by mixed carrageenan and cellulose-g-PAAM with a total weight of 2 grams of bead gel using a 1:1 ratio variation. The mixture was added to the distilled water heated to a temperature of 85°C and stirred with a magnetic stirrer until the solution was homogeneous so that a solution concentration of 2% was obtained. The solution was injected into a mixture of 0.2M $CaCl_2$ solution and 0.2M KCl solution which was added with palm oil as high as 1 cm in an ice bath. A 0.2 M $CaCl_2$ solution was prepared by dissolving 2.2 grams of CaCl2 in 100 mL of distilled water, while a KCl solution was prepared by dissolving 1.49 grams of KCl in 100 mL of distilled water. The granules formed were allowed to stand for 15 minutes while being stirred after which they were filtered. Then, the bead gel granules were immersed in 200 mL 96% ethanol solution for 4 hours then drained and dried at room temperature to constant weight. The flow process of the bead gel production is shown in Fig. 3.

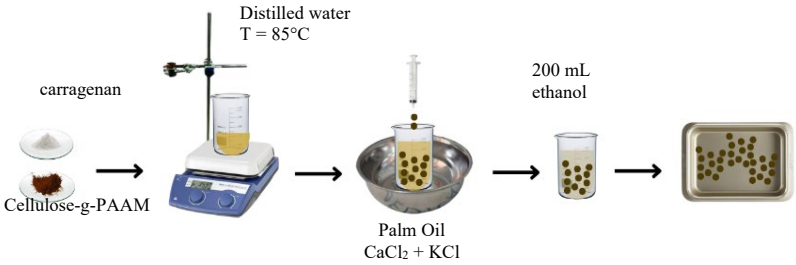

Figure 3. Process of Bead Gel Production

2.5 Swelling Test

The swelling capacity (SC) indicates the bead gel's ability to expand due to the medium entering the bead gel structure. It is necessary to know how much the bead gel's ability to expand. Before the immersion process, the dried bead gels were weighed first and expressed as dry weight (Md). Then, 3 grains of similar bead gels were immersed in water for 3.5 hours so that the water will diffuse into the bead gel. Bead gel was weighed every 30 minutes to constant weight and expressed as wet weight (Mw). The calculation of the swelling capacity (SC) can be described as following Eq. (1).

$$SC = \frac{(Mw - Md)}{Md} \quad (1)$$

3. RESULTS AND DISCUSSION

3.1 FTIR Result

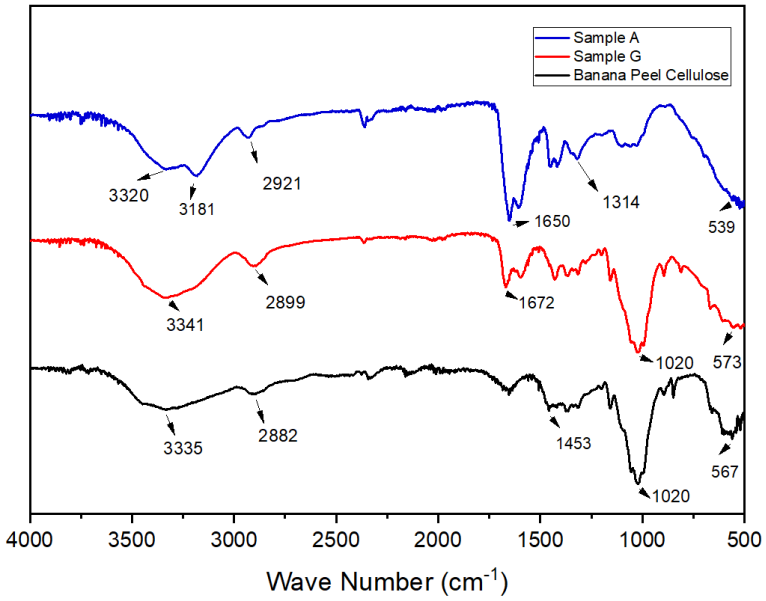

Figure 4. FTIR result

The results of analysis using FTIR qualitatively have several absorption peaks as shown in Fig. 4. The FTIR spectra of cellulose-g-PAAM were compared with banana peel cellulose. Cellulose-g-PAAM sample A and G showed the wave numbers 3341 cm^{-1} and 3320 cm^{-1} which expresses the peak of N-H group. This indicates that the process of grafting acrylamide onto the banana peel cellulose backbone was successful. This refers to the study of Zahra et al. who grafted acrylamide to straw cellulose and wave numbers 3454 cm^{-1} [9].

In sample A and sample G, the wave numbers 1650 cm^{-1} and 1672 cm^{-1} appeared which expresses C=O groups of acrylamide. Therefore, it can be concluded that acrylamide is grafted onto banana peel cellulose. Similar results were found in research Zahra et al. at wave numbers 1655 cm^{-1} expressed of C=O groups. There was a new peak at wave number 1314 cm^{-1} in sample A, which means that a C-N group was formed. So that. it can be concluded that acrylamide was grafted onto the cellulose body of banana peels. The same results were found in research Sunardi et al who grafted acrylamide to *Eleocharis dulcis* at wave numbers 1442.75 cm^{-1} [10].

Figure 4 shows the sample A and G graph as the result of acrylamide grafting on the banana peel cellulose backbone have different wave numbers with pure banana peel cellulose. Cellulose-g-PAAM showed a new peak from the result of microwave grafting. From FTIR result, it was proven that acrylamide could be grafted onto the banana peel cellulose backbone.

3.2 Effect of Initiator Potassium Peroxodisulfate on the Swelling Capacity

The use of potassium persulfate initiator provides a higher grafting efficiency compared to ammonium persulfate [11]. Variations of the mixture for microwave grafting with the addition of potassium peroxodisulfate initiator are presented in Table 1. The microwave grafting process produced cellulose-g-PAAM. The results of microwave grafting were washed with acetone to separate by product through a precipitation process. The yield of Cellulose-g-PAAM with variations in the addition of 1.5% AAM KPS tends higher than the addition of 1% AAM KPS and 1.25% AAM KPS.

Table 1. Variation of microwave grafting addition of potassium peroxodisulphate with 630 watt power

Code	KPS Variation (%AAM)	Cellulose Mass (g)	Acrylamide Mass (g)	*Cellulose*-g-PAAM (g)	*Form*
A	1	2	10	2.04	Powder
B	1.25	2	10	1.87	Powder
C	1.5	2	10	4.74	Solid

The addition of 1.5% AM KPS resulted in higher yields as shown in Fig. 5. This is because the more addition of KPS will increase the number of activated functional groups.

Figure 5. The gel formed with the addition of 1.5% AAM .

Table 2 shows the composition of material for preparing bead gel. The swelling capacity is calculated by Equation (1), Fig 6. shows the swelling capacity of the bead gel with variations in the addition of KPS to water media as a function of time. The x-axis is the time required for swelling in minutes, and the y-axis is the swelling capacity in percent (%).

Bead gel with the addition of 1% AAM KPS shows the higher swelling than the bead gel with the addition of 1.25 % KPS and 1.5% KPS as shown in Table 3. Figure 6 shows that the bead gel with variations in the addition of 1.25% KPS destroyed within 3 hours, while the bead gel with variations in the addition of 1% AAM and 1.5% AAM KPS destroyed in about 3.5 hours.

Table 2. Composition bead gel with KPS amount variation

Code	Mixed Variation
D	Cellulose-g-PAAM (KPS 1% AAM) - carrageenan
E	Cellulose-g-PAAM (KPS 1.25% AAM) - carrageenan
F	Cellulose-g-PAAM (KPS 1.5% AAM) - carrageenan

Figure 6 shows that bead gel with KPS 1% AAM expresses the highest swelling capacity compared to bead gel with KPS 1.25% AAM and 1.5% AAM. Bead gel with KPS 1% AAM shows a swelling capacity value of 2124.64%, bead gel with KPS 1.25% shows swelling capacity value of 1787.69%, and bead gel with KPS 1.5% AAM shows swelling capacity value of 1344.26%. This happened because the more amount of KPS causes excess free radicals of cellulose banana peels. More the free radicals tend to cause faster termination. This may drive the polyacrylamide chains grafted on banana peel cellulose become shorter. As the result, the swelling capacity was low.

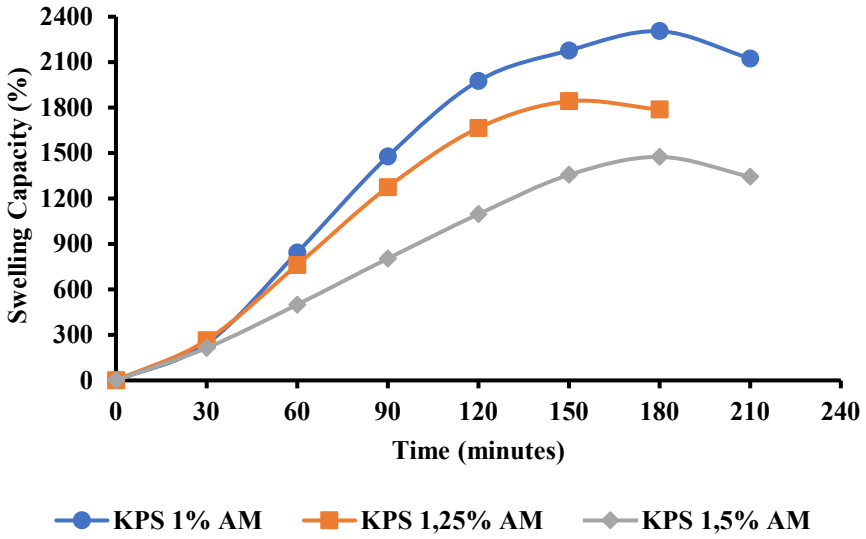

Figure 6. Swelling capacity in water as function of time with variations of KPS amount

Table 3. Comparison of bead gel swollen in water for 3.5 hours

Time (Min)	KPS: Acrylamide (AAM) Ratio		
	1 : 100	1.25 : 100	1.5 : 100
0			
30			
60			
90			
120			
150			
180			
210			

3.3 Effect of Microwave Power on the Swelling Capacity

The power variation of the microwave grafting mixture is presented in Table 4. For the power variation, the yield of Cellulose-g-PAAM with 630 Watt power tends to be higher than the 540 watt and 720 watt power. The range of power variables used in this research did not show a significant difference during swelling test.

Table 4. Variation of microwave power with 1% acrylamide KPS

Code	Power (Watt)	Cellulose Mass (g)	Acrylamide Mass (g)	*Cellulose*-G-PAAM (g)	Form
G	540	2	10	1.56	Powder
A	630	2	10	1.91	Powder
H	720	2	10	1.73	Powder

Table 5 shows the sample of bead gel obtained for further swelling capacity characterization. Figure 7 and Table 6 present the result of swelling behavior of obtained bead gel. The swelling capacity is calculated by Eq. (1), Fig. 7 shows the swelling capacity of the bead gel with variations in power in the water medium in a time function. The x-axis is the time required for swelling in minutes, and the y-axis is the swelling capacity in percent (%). Table 6 shows that there is no significant difference for each power variable. The swelling results show that bead gels with vary of power variations were able to swell for up to 3.5 hour. After that, the bead gels destroyed.

Table 5. Power variation

Code	Power Variation
I	*Cellulose-g-PAAM* (540 Watt) - carrageenan
D	*Cellulose-g-PAAM* (630 Watt) - carrageenan
J	*Cellulose-g-PAAM* (720 Watt) - carrageenan

Figure 7 shows that the bead gel with a power of 630 Watt has the largest swelling capacity compared to a bead gel with a power of 540 Watt and 720 Watt. Bead gel with a power of 630 Watt has a swelling capacity value of 2289.14%, a bead gel with a power of 540 Watt has a swelling capacity value of 2254.6%, and a bead gel with a power of 720 Watt has a swelling capacity value of 2107.92%. The microwave grafting process requires appropriate power, so that the cooling cycle temperature is always maintained in the temperature range of 60°C-70°C. The use of 720 Watts of power has a cooling cycle temperature above 70°C, while the use of 540 Watts of power and 630 Watts of cooling cycle temperatures is maintained between 60°C-70°C. Based on the swelling test, there is no significant difference in the ability to absorb water on the bead gel at a given power variation. Therefore, low power tends to be used for efficient grafting.

Table 6. Comparison of bead gel form before and after swelling in water for 3.5 hours with power variations

Time (minute)	Power Variation		
	540 Watt	630 Watt	720 Watt
0			
30			
60			
90			
120			
150			
180			
210			

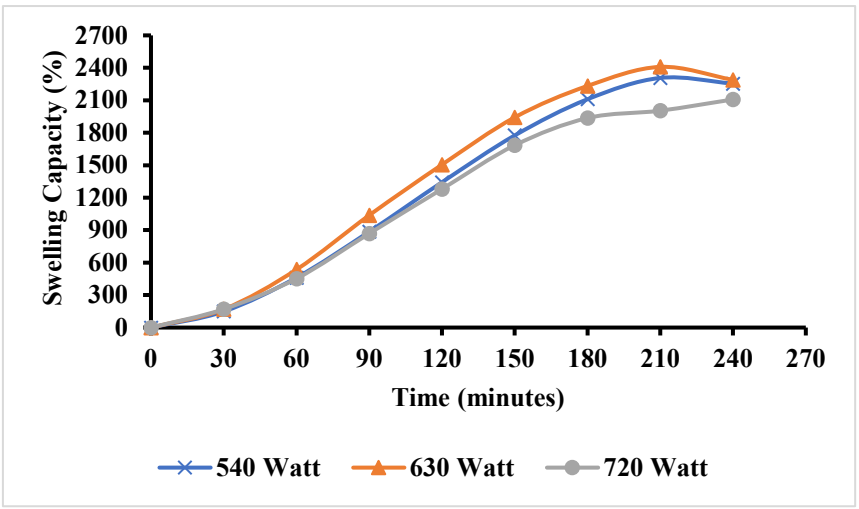

Figure 7. Swelling capacity in water with power variations

4. CONCLUSION

The grafting process acrylamide to the banana peel cellulose backbone was successfully carried out based on the FTIR result. The lower KPS concentration produces bead gels with high swelling capacity. The microwave power has no effect on the swelling capacity of bead gels. The form of swollen bead gels were still the same form with the dried bead gels.

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