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Microwave-Assisted Synthesis of Corn Husk-Based Hydrogels Grafted with Acrylamide

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microwave;
swelling capacity.

ABSTRACT. Corn husk waste contains cellulose, which has the potential as a raw material for hydrogel preparation. Hydrogels can be applied as water purification, diapers, and superabsorbents. This study aimed to synthesize hydrogel from corn husk cellulose grafted with acrylamide monomer using a microwave-assisted grafting method. Potassium peroxodisulfate (PPD) was used as an initiator, and the effects of acrylamide and PPD on hydrogel swelling capacity were investigated. The process involved drying and crushing corn husks into powder, then mixing the powder with acrylamide and PPD for microwave grafting to form a polymer, which was then ground into powder. The grafted polymer was combined with carrageenan to create bead gels soaked in distilled water and urea to measure swelling capacity. Results showed that swelling capacity increased with more acrylamide and decreased with more PPD. The highest swelling capacity reached 1016.16% in water and 961.6% in urea. FTIR analysis confirmed the successful grafting of acrylamide onto corn husk cellulose by detecting changes in the infrared spectrum. Based on FTIR and swelling capacity data, it was concluded that the grafting process was completed using the microwave method with PPD as the initiator.

INTRODUCTION

Hydrogels are cross-linked polymer systems that have a specific capability for soaking enormous amounts of water (Paul Guin *et al.*, 2018). They can undergo volume changes due to temperature, solvent quality, pH, electric field, etc. (Neethu *et al.*, 2018). Currently, hydrogels have been applied as fertilizer-controlled release agrochemicals (Sarmah and Karak, 2020), flocculants in water purification (Mittal *et al.*, 2015), diapers (Bashari *et al.*, 2018) and super-absorbents in field of oil enhanced recovery (Sun *et al.*, 2021). Therefore, hydrogels are important for specific sectors and crucial to modify to reach the terrific ability.

Hydrogels are dominated by synthetic polymers, which are the main material in Indonesia. This polymer is imported and harmful environmentally because it is difficult to decompose naturally. Alternatively, hydrogels are developed based on natural polymers such as polysaccharides. One kind of polysaccharide found in natural products is cellulose. Corn husk has a significant cellulose content, around 44% (Setyaningsih *et al.*, 2020). Corn husk has been widely researched and utilized, including being used for the manufacture of eco-friendly Urea-Formaldehyde Composites, in which corn husk is used for its cellulose fibers to increase the compression strength and water absorption of the composite (Pokhrel *et al.*, 2020). Kampeerapappun (2015) and Choque-Quispe *et al.* (2022) have also researched corn husks by utilizing cellulose nanocrystals as an additive applied in food coatings. The study shows that the addition of corn husk cellulose nanocrystals can improve good heat resistance with low solubility and ease of biodegradation. Aside from corn husks, natural polymers can also be derived from Indonesian seaweed, namely carrageenan. Carrageenan has hydrophilic characteristics because it contains hydroxyl. Carrageenan is essential as a gel maker and reinforcer (Atmaka *et al.*, 2021).

This study aims to synthesize the bead gel (hydrogel) from corn husk–acrylamide with potassium peroxodisulfate (PPD) initiator using microwave grafting method and analyzing the effect of acrylamide and PPD amount addition on the swelling ability of bead gels in various medium. The use of the microwave grafting method has begun to be developed because it is more environmentally friendly and efficient in research conducted by

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Mishra *et al*. (2012), who used microwave grafting on carboxymethyl cellulose with polyacrylic acid (CMC-g-PAA). This study showed that the microwave grafting method supported by FTIR analysis was successfully used. Grafting hydrophilic monomer acrylamide into cellulose as the backbone using microwave energy was used to modify the molecular structure of corn husk cellulose. Grafting was carried out with potassium peroxodisulfate (PPD) as an initiator to accelerate reaction in both grafting and acrylamide polymerization during the process. The modified cellulose was combined with carrageenan to produce bead gels. This method has not been widely applied in general and specifically to corn husk material, which has not been studied before. This method is more efficient, environmentally friendly, has a cleaner approach, and better control. The dry bead gels obtained were characterized by their functional groups applying FTIR. Swelling capacity was tested in distilled water and urea solution media.

RESEARCH METHODS

Corn husk waste was obtained from corn farmers and traders in Surakarta, Indonesia. The carrageenan flour (KRI 02) was purchased from the local market. Other chemicals were used as received without further purification, such as potassium peroxodisulfate ($K_2S_2O_8$) (E. Merck), palm oil (Tropical), ethanol 96% (C_2H_5OH) (Nusa Kimia), acrylamide (C_3H_5NO) (E. Merck), distilled water (H_2O) (CV. Agung Jaya), acetone ($(CH_3)_2CO$) (E. Merck), potassium chloride (KCl) (E. Merck), calcium chloride ($CaCl_2$) (Pudak), and urea (Pudak). The instruments used were a microwave 650 Watts (Krisbow) and an FTIR spectrophotometer (Shimadzu, IRspirit).

Preparation of Corn Husk Powder

Corn husk waste was washed and dried using an oven at 90 ℃ to a constant weight. Furthermore, dry corn husks were mashed using a grinder and then sieved using an 80–mesh sieve shaker to obtain fine corn husk powder.

Preparation of Grafted Polymer

A grafting reaction was carried out using a domestic microwave oven (Krisbow 20 L). One gram of corn husk powder, acrylamide (0.1 – 0.3 g), and PPD (5 – 8 g) were dissolved in 50 mL of distilled water in a 100 mL beaker glass and stirred for 15 minutes at 300 rpm. Table 1 shows the variation of composition mixture for preparing grafted polymer. The solution was transferred to a 1000 mL beaker and irradiated in a domestic microwave with irradiation at 650 watts and cycle of 30 seconds (<70 ℃), then cooled by immersion in cold water until it reached room temperature. Irradiation cycle–cooling was done until the gel formed. When a gel had formed, it was pulled out for 24 hours. The gel formed was soaked with excess acetone until a lumps state was obtained. The lump was dried at 50 ℃ until constant weight, then weighed and crushed.

Table 1. Variation of composition for preparing grafted polymer.

Sample	Mass of Corn Husk (g)	Mass of PPD (g)	Mass of Acrylamide (g)
Sample A	1	0.1	5
Sample B	1	0.1	8
Sample C	1	0.3	5
Sample D	1	0.3	8

Preparation of Bead Gel

Two grams of the grafted polymer–carrageenan powder mixture was put into 100 mL of distilled water and stirred with a magnetic stirrer until the solution was homogeneous. The solution was heated up to 50 ℃. The solution was injected using a syringe into palm oil at 1 cm height and dropped into a mixture of 0.2 M $CaCl_2$ and 0.2 M KCl solutions. Physical crosslinking was performed at this stage, and bead gels were produced. The beaker glass with an ice bath was used for this bead gel preparation. The $CaCl_2$ 0.2 M solution was prepared by dissolving 2.2 g of $CaCl_2$, while a KCl solution was prepared by dissolving 1.49 g of KCl in 100 mL of distilled water. The bead gels formed were allowed to stand for 15 minutes while stirring, after which they were filtered. Afterward, the bead gel was soaked in 200 mL ethanol 96% solution for 4 hours before being drained and dried to a constant weight. Each experiment was performed at least once and averaged.

Swelling Test of Bead Gel

The bead hydrogel obtained in this research was to develop the diaper application field. For this reason, the swelling capacity of the bead gel was tested in distilled water and urea medium. The urea percentages were

prepared similarly to the urea composition in human urine. The swelling capacity test was carried out in urea solution at a concentration of 0.25% (w/w) (Putro *et al*., 2019). The dry bead gels formed were expressed as dry weight (Md) immersed in the medium and weighed every 30 minutes to a constant weight. The weight after immersion was defined as the wet weight (Mw). The swelling capacity was calculated using Equation (1). Each experiment was done with at least three samples, and the mean value was used to show the percentage swelling capacity.

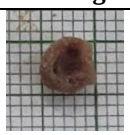
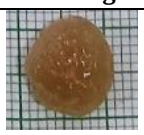
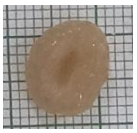
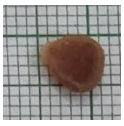
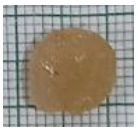
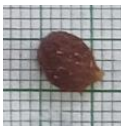
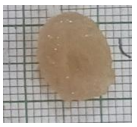
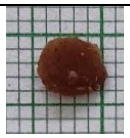
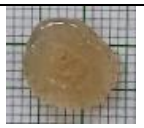
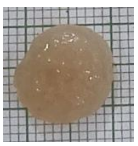
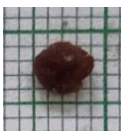
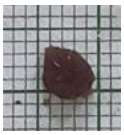
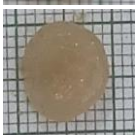
$$\text{Swelling capacity (\%)} = \frac{(\text{Mw - Md})}{(\text{Md})} \times 100\% \quad (1)$$

RESULTS AND DISCUSSION

The Effect of Potassium Peroxodisulfate on the Swelling Capacity of Bead Gels in Various Medium

Potassium peroxodisulfate (PPD) is an initiator to producing free radicals that create active sites during the grafting process between the cellulose backbone and acrylamide monomer, resulting in a higher percentage of grafting (Omer *et al*., 2023). Figure 1 and Figure 2 show the results of swelling capacity in distilled water medium and urea solution medium. The x-axis is the time required for swelling capacity, and the y–axis is the percentage of swelling capacity. Table 2 shows the image of the bead gel before and after the swelling test.

Table 2. Comparison of bead gel before and after swelling in distilled water and urea solution medium.

Variation		Appearance of bead gel		Swelling Capacity (%)	Time (min)	Medium
PPD (g)	Acrylamide (g)	Before Swelling	After Swelling			
0.1	5			977.1	240	Distilled Water
				903.6	240	Urea Solution
0.1	8			1016.6	240	Distilled Water
				961.6	240	Urea Solution
0.3	5			876.8	240	Distilled Water
				762.8	240	Urea Solution
0.3	8			880.3	240	Distilled Water
				778.1	240	Urea Solution

The Swelling capacity of sample B was compared to sample D in the distilled water medium, as shown in Figure 1. The grafted polymer with 0.1 g PPD resulted in a higher swelling capacity, namely 1016.6%, than the grafted polymer with 0.3 g PPD that showed 880.3% swelling capacity, the sample B was compared to sample D. Similar results were also obtained when comparing samples A and C. The grafted polymer with 0.1 g PPD resulted in a higher swelling capacity, namely 977.1%, than the grafted polymer with 0.3 g PPD which showed 876.8% swelling capacity.

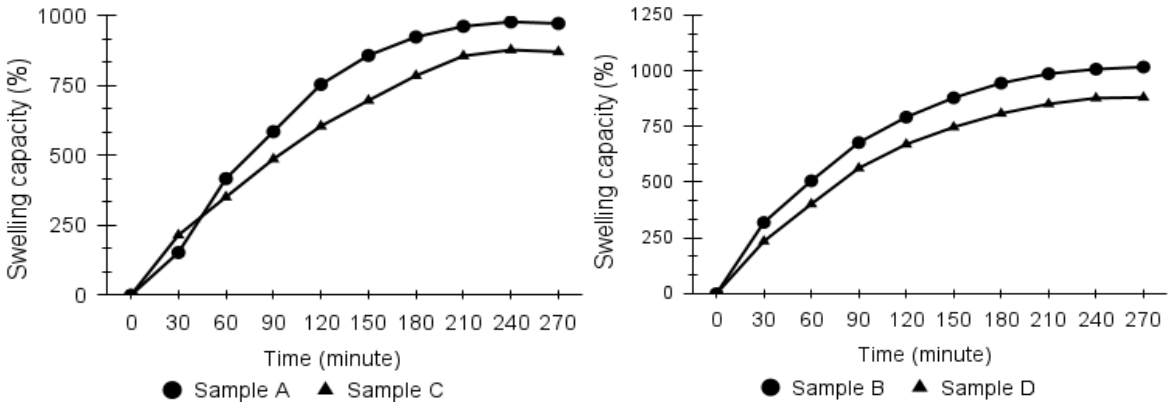


Figure 1. Results of swelling capacity in distilled water medium: (a) Sample A and C and (b) Sample B and D.

The swelling capacity of sample B was compared to sample D in the urea solution medium, as shown in Figure 2. The bead gel with 0.1 g PPD resulted in a higher swelling capacity (961.6%) than that with 0.3 g PPD (778.1%). Similar results were also obtained when comparing samples A and C. The results show a constant data trend because the more PPD added, the smaller the swelling capacity. Similar results were obtained in the research by Kenawy *et al*. (2020), showing that the grafted copolymerization of acrylamide and sugarcane bagasse was carried out using potassium peroxodisulphate as initiator and methylene-bis-acrylamide as crosslinker. Increased amounts of PPD produced more free radicals, accelerating chain termination reactions and reducing swelling capacity. In addition, decreasing the swelling capacity is caused by increment in polymerization products. The increment of PPD causes excess free radicals from corn husk cellulose to join, possibly causing faster termination. There are too many active groups that terminate each other, so it inhibits grafting because it is terminated. Polyacrylamide chains grafted onto corn husk cellulose become shorter and reduce swelling capacity.

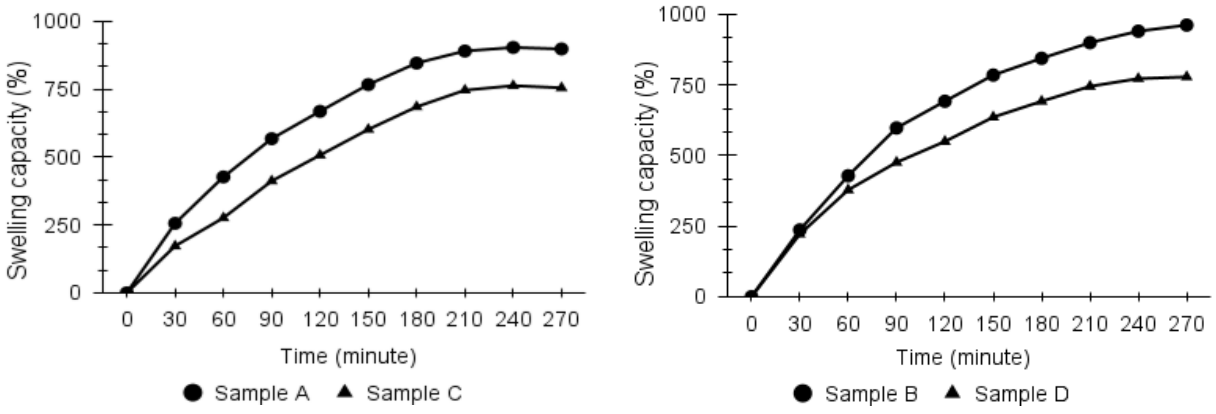


Figure 2. The swelling capacity in 0.25% urea solution: (a) sample A and C and (b) sample B and D.

The Effect of Acrylamide on the Swelling Capacity of Gel Bead in Various Medium

Based on Figure 3 and Figure 4, the higher the amount of acrylamide mixed with the corn husk, the more hydrophilic groups possibly present in the polymer hydrogel. This will enhance the swelling capacity. In Figure 3, in a distilled water medium, the bead gel's swelling capacity value was obtained sequentially with 5 g of acrylamide (samples A and C), and the highest swelling capacity was 977.1%. The 8 g acrylamide (samples B and D) showed

the most significant swelling capacity at 1016.6%. In Figure 4, samples A and C had the highest swelling capacity at 961.6% in the urea solution medium 0.25% (w/w). Samples B and D reached a higher swelling capacity at 778.1%. This data shows that the more acrylamide added, the higher the swelling capacity value. This is due to the increased hydrophilic properties of CH–g–PAAM. In other research, the synthesis of sugarcane bagasse–g–polyacrylamide also obtained similar results, namely an increase in swelling capacity with an increase in the acrylamide monomer, which was caused by increased hydrophilic properties (Kenawy et al., 2020).

Figure 3 and Figure 4 also show the stability property. The swelling capacity of the sample with 5 g of acrylamide continued to increase up to 240 minutes of immersion and decreased at 270 minutes. At 270 minutes, the swelling capacity with 8 g of acrylamide continued to increase. This shows the results of natural polymer grafting with acrylamide monomer, resulting in a stronger bead gel structure.

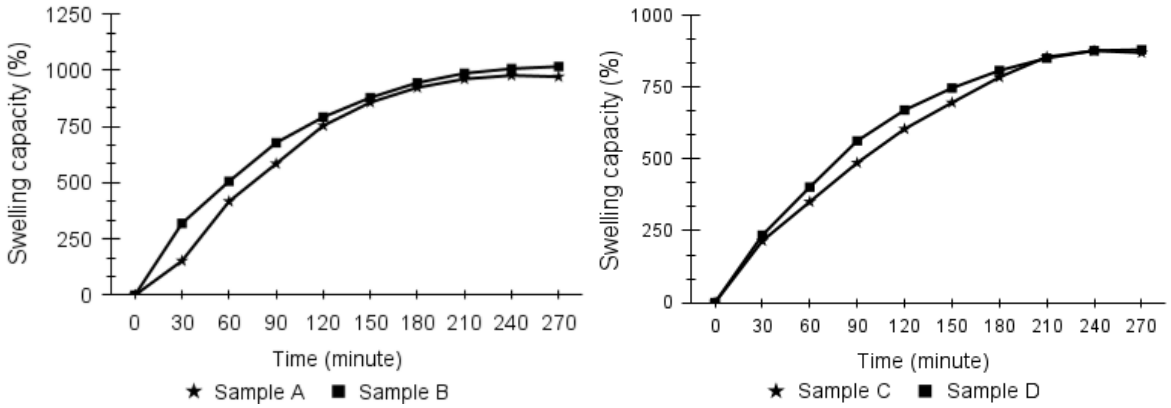


Figure 3. Results of swelling capacity in distilled water medium: (a) Sample A and B and (b) Sample C and D.

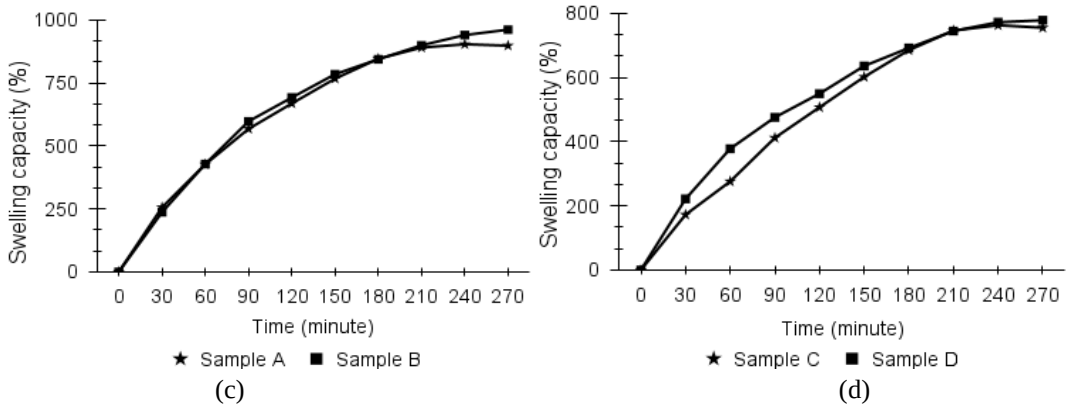


Figure 4. The swelling capacity in 0.25% urea solution: (c) sample A and B and (d) sample C and D.

Based on Table 2, the image data proves that the hydrogel made from corn husk flour has good swelling capacity in medium distilled water and urea with more than 4 hours of swelling resistance. Abd Alla *et al*. (2012) examined the swelling ability of tara gum/acrylic acid-based superabsorbent hydrogel in urea media and found that the swelling resistance reached 6 hours. With these capabilities, the hydrogel can be applied in everyday life, one of which is for diapers. The swelling ability of urea medium needs to be further developed with a better method, resulting in a higher swelling capacity with a longer shelf life, which can then be applied as a material for making diapers from environmentally friendly natural polymers.

FTIR Spectra of Samples

An FTIR spectrophotometer (Shimadzu, IRspirit) was used to identify changes in the molecule structure of the material before and after the grafting reaction. The material was analyzed directly without forming a KBr pellet. The FTIR spectra of corn husk showed prominent absorption peaks, including 3330.39 cm^{-1} (O–H strain vibration), 2919.62 cm^{-1} (C–H strain vibration), 1635.95 cm^{-1} (C=O strain vibration), and 1034.06 cm^{-1} (strain vibration C–O–C) (Figure 5 and Table 3). FTIR spectra on acrylamide show prominent absorption peaks, including

3336.09 cm^{-1} and 3163.51 cm^{-1} (strain vibrations O–H and N–H), 1667.33 cm^{-1} (strain vibration C=O), 1611.71 cm^{-1} (C=C strain vibration), 1424.86 cm^{-1} (C–N strain vibration), and 959.89 cm^{-1} (C–H strain vibration). In the FTIR spectrum of the CH–g–PAAM sample, it is observable that there was an absorption peak at wavenumbers 3340.37 cm^{-1} corresponding to the N–H and also O–H groups. The appearance of a new absorption peak at wavenumbers 2928.17 cm^{-1} indicates an extension of the C–H strain vibration. At wavenumber 1651.64 cm^{-1}, it shows the pulse of the C=O group, and at wavenumber 1447.68 cm^{-1}, which is the C–N group.

In the previous report studied grafted acrylamide onto carboxymethyl starch, there are absorptions at wavenumbers 3281 cm^{-1} corresponding to the N–H and O–H groups; at 2913 cm^{-1} corresponding to the C–H group and at 1637 cm^{-1} corresponding to the C=O group (El-Sheikh, 2016). Synthesis of polyacrylamide grafted xanthan absorption also appeared at wavenumbers 3420 cm^{-1}, indicating the presence of O–H groups; at 2880 $cm^{-1,}$ indicating the presence of C–H groups; at 1675 $cm^{-1,}$ indicating the presence of C=O groups; and at 1410 cm^{-1} which suggests the presence of C–N groups (Chami *et al*., 2021).

Table 3. Comparison FTIR spectra between CH–g–PAAM hydrogel and previous research.

Spectrum	Peak (cm^{-1})		
	CH–g–PAAM (this research)	**CMS–g–polyacrylamide** El-Sheikh (2016)	**Polyacrylamide grafted xanthan** Chami *et al*. (2021)
O–H	3340.37	3281	3420
C–H	2928.17	2913	2880
C=O	1651.64	1637	1675
C–N	1447.68	–	1410

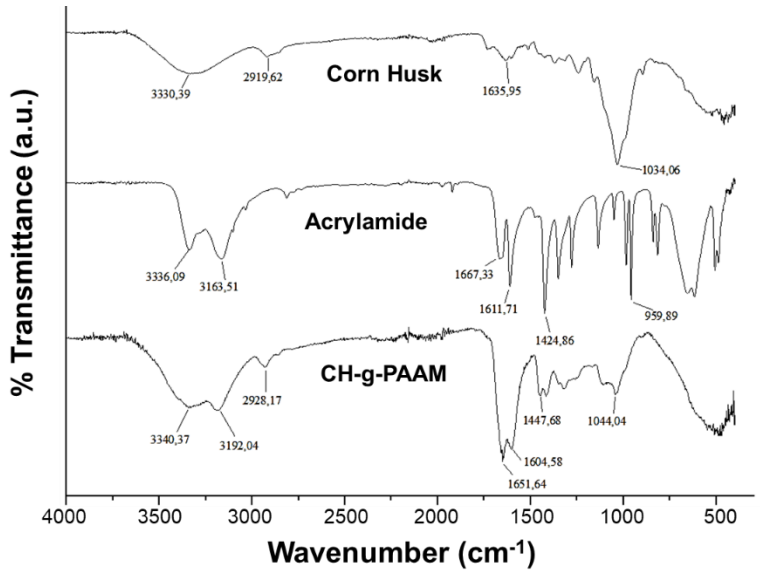


Figure 5. FTIR spectra of corn husk, acrylamide, and CH-g-PAAM.

The FTIR result (Figure 5) shows additional peak groups in the CH–g–PAAM spectra, namely the O–H, C–H, C=O, and C–N groups. It can be concluded that the appearance of these additional peak clusters proves that the grafting of acrylamide on the corn husk cellulose backbone using the microwave grafting method and the addition of potassium peroxodisulfate was successful.

CONCLUSION

Successful preparation of hydrogel from corn husk–acrylamide, utilizing a microwave-assisted grafting technique with the addition of potassium peroxodisulfate (PPD) initiator, has been achieved. A correlation emerged between PPD usage and swelling capacity, with lower PPD amount resulting in more significant bead gel swelling. Furthermore, increased acrylamide addition correlated positively with enhanced swelling capacity. The accomplishment of acrylamide grafting onto the corn husk cellulose backbone was confirmed through FTIR

testing, demonstrating the effectiveness of the microwave grafting method in this innovative hydrogel synthesis. The obtained hydrogel may be potentially developed for diaper application.

CONFLICT OF INTEREST

There is no conflict of interest in this article.

AUTHOR CONTRIBUTION

GPA, MNSA, and SD: conceived and planned the experiment. GPA and MNSA: performed experiments, analyzed data, and wrote visualization and manuscript. SD and MK: developed idea and funding acquisition, resources, managed project administration, and supervised GPA and MNSA. All authors reviewed, discussed the results, and commented on the manuscript.

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