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Modification of Rice Straw Cellulose with Acrylamide using Ammonium Persulfate Initiator by Microwave Assisted

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ABSTRACT

In this study, rice straw powder was grafted with acrylamide (AA) using amonium persulfate (APS) as an initiator to obtaine rice straw cellulose-graft-polyacrylamide (RS-g-PAA). The resulting RS-g-PAA was then mixed with carrageenan to form bead gel. This research aimed to determine the influence of the amount of acrylamide and ammonium persulfate as an initiator on the swelling capacity of bead gels. The mixture of 1 g of rice straw cellulose, AA (5, 10, 15 g), APS (0.05, 0.10, 0.15 g), and 50 mL aquadest was irradiated using microwave at 540 W for 30 s with cooling cycles. The aqueous mixture of RS-g-PAA and carrageenan was injected into palm oil layer and then crosslinked using KCl and $CaCl_2$. The results of FTIR show that the successful grafting by the appearance of amide groups in the RS-g-PAA structure. The bead gel with 15 g AA showed the highest swelling capacity of 1806.12% in aquadest and 1611.58% in urine solution. Bead gels from APS of 0.10 g produced the highest swelling capacity of 1218.15% in aquadest and 975.20% in urine solution. Therefore, the bead gels based on RS-g-PAA and carrageenan demonstrate strong potential as an environmentally friendly superabsorbent polymers.

Keywords: acrylamide, carrageenan, hydrogel, microwave grafting, rice straw.

1. INTRODUCTION

Indonesia, as an agricultural country, still prioritizes agriculture as its primary commodity, for example, rice, which produces rice straw as a byproduct. Rice straw is a lignocellulosic material available in large quantities and has not been optimally utilized in Indonesia. In terms of composition, rice straw from Indonesia contains 37.5% cellulose, 35.4% hemicellulose, 14.5% lignin, and 11.3% ash [1]. Cellulose is a great starting material, but due to dissolution complexity, the application is limited. Another approach to broaden its applications is through chemical modification into various derivatives [2]. Besides the cellulose, the lignin was chemically modified by grafting method with polyacrylamide [3,4]. Recent research has proven that biopolymer has the potential to be used as a raw material for hydrogels, as demonstrated in studies conducted to create superabsorbent hydrogels based on rice straw for agricultural applications [5] and pectin-gelatin-dragon fruit peel for burn wound dressings [6].

Based on previous research, cellulose adsorption capacity and hydrophilicity can be improved by grafting acrylamide monomers onto its main chain cellulose as the back bone [7]. Acrylamide (AA) is a monomer that has an amide group which has high water absorption properties and easily reacts with electrophiles and free radicals [8]. Higher acrylamide content increases swelling capacity but decreases the bead gels strength [9]. In addition, ammonium persulfate (APS) acts as a free radical initiator for grafting acrylamide onto rice straw cellulose, where its concentration affects the swelling capacity and network structure of the hydrogel [10]. A study reported that the addition of an initiator at an optimal dose can drastically increase the swelling capacity, however, too high an

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initiator dose can reduce the pore size and increases the crosslink density, which reduces the swelling capacity [11].

In this research, all of kinds of polysaccharides in rice straw powder were then called as cellulose. We have grafted acrylamide (AA) onto the backbone of rice straw powder, the resulting in formation of polyacrylamide grafted rice straw cellulose (RS-g-PAA). The grafting method is carried out using microwaves because it is known to be more efficient, faster, and environmentally friendly than conventional methods. To the best our knowledge, RS-g-PAA preparation using microwave energy never been reperted previously.

Grafting results in the form of RS-g-PAA powder, then was mixed with kappa-carrageenan, a polysaccharide from the red algae *Rhodophyceae* [12]. Carrageenan in hydrogel formation is as a thickener, texture modifier, and gel former [13]. The advantage of carrageenan-based hydrogel is that it is good at absorbing water [14]. The combination of grafted cellulose and carrageenan is expected to form a bead gel with a stable structure and high absorption capacity.

This study aims to investigate the effect of acrylamide (as monomer) and ammonium persulfate (as initiator) on the swelling behavior in aquadest and synthetic urine.

In the swelling test, aquadest was used as the primary medium and urine as a comparison for the quantity of hydrogel absorption. The hydrogel was confirmed to absorb more aquadest compared to synthetic urine because urine contains sodium, which reduces the hydrogel's absorption capacity [15].

2. RESEARCH METHODS

2.1. Material

The materials used were rice straw from Pati Indonesia, acrylamide (E.Merck), carrageenan, ammonium peroxydisulfate (E.Merck), potassium chloride (KCl) (KGaA), aceton, calcium chloride dihydrate ($CaCl_2.H_2O$) (E.Merck), aquadest, urea, Na_3PO_4, and palm oil.

2.2. Preparation of Cellulose

The preparation of rice straw cellulose (RS) powder was shown in Figure 1. The rice straw was rinsed and dried under the sun for 12 h, then cut into 2-3 cm and dried in the oven at 85°C for 16 h. The dried RS was grinded and sieved until it passed 100 mesh to obtain a fine powder of rice straw.

2.3. Synthesize of Rice Straw Cellulose Graft Polyacryl-Amide (RS-g-PAA)

Figure 2 shows the schematic of the synthesis of RS-g-PAA. The preparation of rice straw cellulose graft polyacrylamide (RS-g-PAA) was carried out by mixing 1 g of rice straw powder and various weight of AA (5; 10; 15 g) dissolved in 50 mL of aquadest. This mixture was placed in a 1000 mL beaker and covered with aluminum foil. The mixture was stirred using a magnetic stirrer at 200 rpm for 30 min to obtain a well-dispersed suspension. After that, the suspension was left for 24 h at room temperature. After 24 h, ammonium persulfate (APS) was added as an initiator with various masses (0.05; 0.10; 0.15 g). The mixture was stirred for 15 min at 200 rpm. The samples in this study were identified by the sample code listed in Table 1.

It was irradiated for 30 s using a microwave (Krisbow 23L-10108236) at 540 W, cooled in an ice bath to 30°C. This cycle was repeated until the mixture thickened [16]. For 10 g of acrylamide the mixture required 5 cycles, for 15 g it required 3 cycles, and for 5 g it required 15 cycles. The liquid phase was separated, while the solid phase was treated with excess acetone (100 mL) for 1 h to stop the reaction [17]. The mixture was then filtered, and the solid was dried at 70°C to constant weight. Finally, the dried solid was

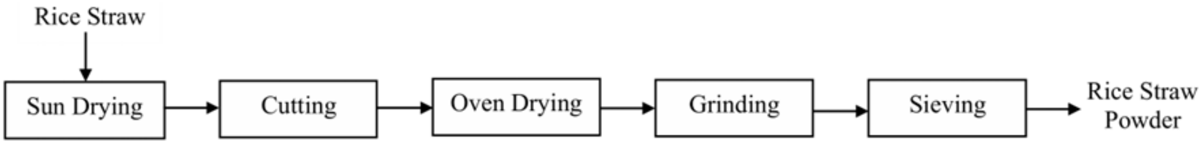


Figure 1. Schematic of rice straw powder reparation.

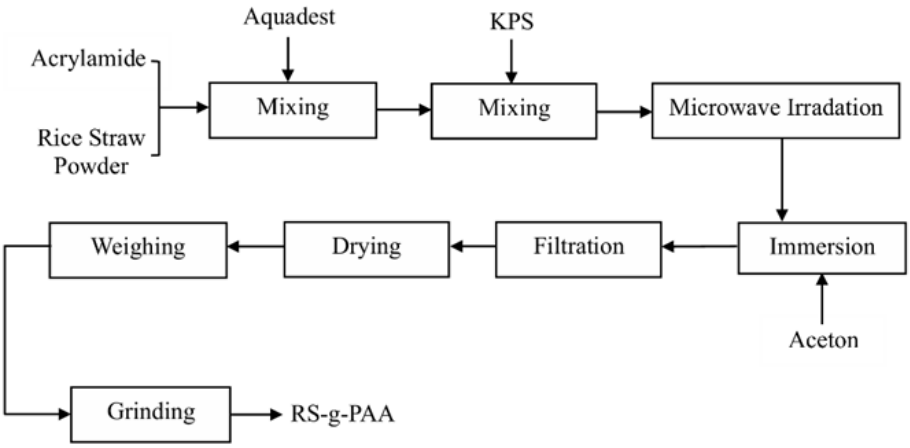


Figure 2. Schematic of synthesis of RS-g-PAA.

Table 1. Variation of grafting sample composition.

Code	Rice Straw Powder (g)	AA (g)	APS (g)	Irradiation Time (min)
A	1	10	0.05	2.5
B	1	10	0.10	2.5
C	1	10	0.15	2.5
D	1	5	0.10	2.5
E	1	10	0.10	2.5
F	1	15	0.10	2.5

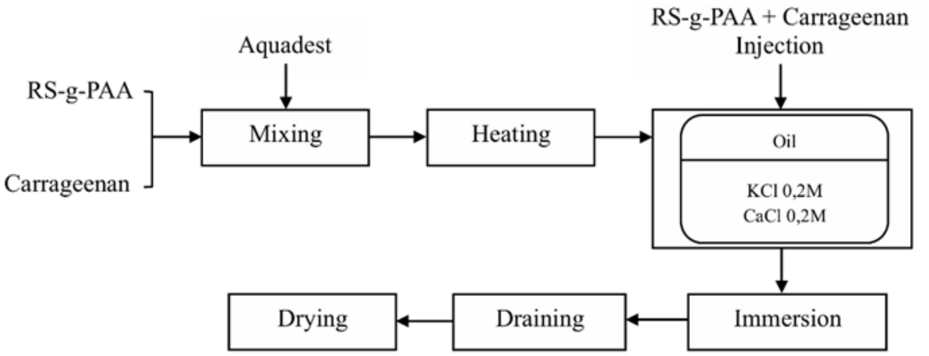


Figure 3. Schematic of bead gel preparation.

ground (Fomac FGD-Z200) into RS-g-PAA powder.

The grafting ratio (%) can be calculated using equation (1) [18].

$$\text{Grafting ratio}=\frac{\text{wt. of RS-g-PAA - wt. of rice straw powder}}{\text{wt. of rice straw powder}}\times 100\% \quad (1)$$

2.4. Fourier Transform Infra-Red (FTIR)

FTIR test was conducted to identify structural changes in chemical compounds resulting from the grafting process. These changes were observed through the characteristic peaks of functional groups. The spectra of rice straw cellulose, acrylamide, and RS-g-PAA were recorded using a FTIR Spectrometer (Agilent Cary 630) [17]. Samples were analyzed at wavelengths between 4000 and 500 cm^{-1}.

2.5. Bead Gel Preparation

Bead gel was made by combining RS-g-PAA and carrageenan in a 1:1 weight ratio [19]. 1 g of RS-g-PAA and 1 g of carrageenan was dissolved in 100 mL of aquadest, then stirred with hot plate magnetic stirrer until homogeneous, and heated to a temperature of 70°C. The next solution was injected using a syringe into a 1000 mL beaker containing a solution consisting of 0.2 M $CaCl_2$ and 0.2 M KCl, topped with 1 cm of palm oil placed on an ice bath. In making bead gel, the $CaCl.2H_2O$ 0.2 M is made by dissolving 2.94 g of $CaCl.2H_2O$ in 100 mL of aquadest, while 0.2 M KCl solution is made by dissolving 1.5 g of KCl in 100 mL of aquadest.

The formed bead gels were then left to stand for 15 min, stirring gently by hand occasionally. The bead gels were then filtered and soaked for 4 h in 200 mL of technical acetone solution. After 4 h of soaking, the bead gels were drained and dried at 70°C until constant weight. Each experiment was repeated three times, and the average value was used to display the data. The schematic procedure for bead gels preparation were shown in Figure 3.

2.6. Swelling Capacity Test of Bead Gel

The swelling test was conducted to evaluate the capacity of bead gels to absorb liquid. Three dried bead gel sample were weighed to constant weight. Each sample was then immersed in 10 mL of test media (aquadest and synthetic urine), for 180 min. Synthetic urine was prepared by dissolving 2.43 g of urea, 0.6 g of KCl, 1 g of NaCl, and 0.64 g of Na_3PO_4 into 100 mL of aquadest [20]. Grafting success can be evaluated based on the swelling capacity value [21].

The sample of bead gels were weighed before soaking and recorded as the dried sample. They were then soaked for 180 min in aquadest and synthetic urine to allow diffusion into the bead gels. During soaking, the sample were weighed at 30 min intervals and recorded as the swollen sample. The calculation of the swelling capacity of the bead gels used equation (2) [22].

$$\text{Swelling capacity} = \frac{\text{Weight of swollen sample - Weight of dried sample}}{\text{Weight of dried sample}}\times 100\% \quad (2)$$

The morphological appearance of the bead gels was examined using a Dino-Lite digital microscope (stand RK10, digital basic AM211).

3. RESULTS AND DISCUSSION

3.1. Functional Group Analysis of Materials

This study utilized cellulose from rice straw, an abundant agricultural residue mainly composed of cellulose, hemicellulose, and lignin. Cellulose is a naturally occurring polymer containing long chains of glucose molecules. Each glucose unit contains hydroxyl groups (–OH) that contribute to the hydrophilicity of cellulose [23]. Hydrophilicity refers to a material's ability to attract or interact with water. Acrylamide was grafted onto cellulose to increase hydrophilicity. Acrylamide contains a vinyl group ($-CH=CH_2$) and an amide group ($-CONH_2$). The amide group forms stronger hydrogen bonds than carboxyl groups, so it is polar and very soluble in water [24,25].

FTIR analysis was conducted to confirm the grafting of acrylamide onto rice straw cellulose, as shown in Figure 4.

The grafting reaction used ammonium persulfate (APS) as an initiator under microwave irradiation. The FTIR spectrum showed changes in functional groups after grafting. From Figure 4, in spectrum of AA, the peak of 3154 cm^{-1} and 1652 cm^{-1} were attributed to the N-H and C=O stretching vibration, respectively. The O–H stretching band of pure cellulose appeared at 3339.51 cm^{-1}.

The grafted polymer (RS-g-PAA) showed two new peaks that were absent in pure cellulose. The first peak appeared at 1636.76 cm^{-1} and corresponded to C=O stretching. The second peak appeared at 1592.33 cm^{-1} and was assigned to N–H bending or C=N stretching. The appearance of these peaks confirmed that acrylamide was covalently grafted onto the cellulose backbone and not only physically mixed.

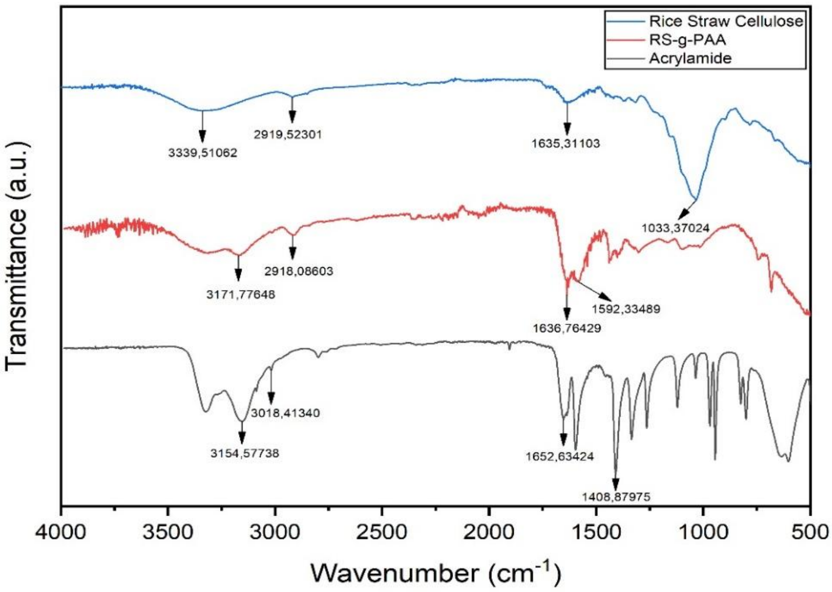


Figure 4. FTIR Spectrum of (a) rice straw cellulose, (b) acrymalide, and (c) RS-g-PAA.

Table 2. Comparison of FTIR wave number of RD-g-PAA (sample F) with previous studies.

Functional Group	Wave Number (cm^{-1})			
	This Research (RS-g-PAA)	Acrylamide grafted cassava waste pulp [26]	Acrylamide grafted cellulose [27]	Polyacrylamide grafted waste paper cellulose [16]
O–H	3171.78	3200	3354	3426.32
C–H	2918.09	2930	2903	-
N–H	1592.33	1600	-	1504.48
C=O	1636.76	1650	1706	1654.70

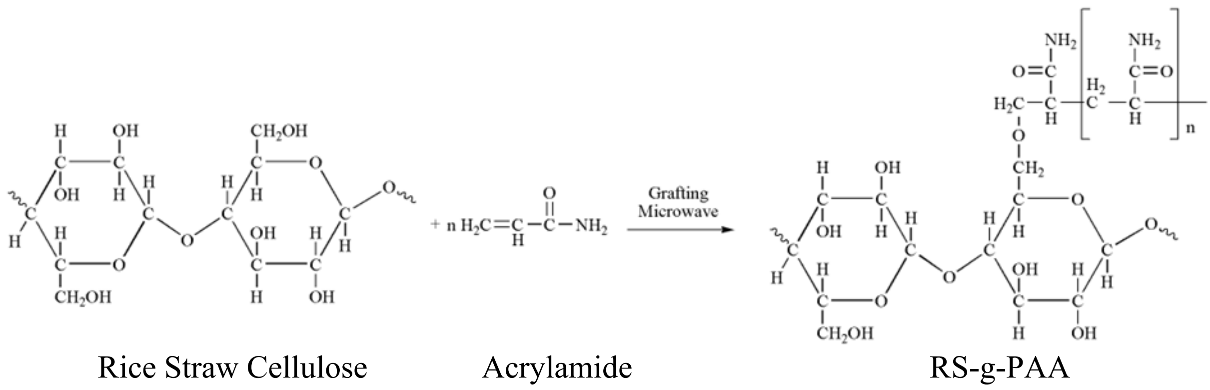


Figure 5. Grafting reaction scheme.

The presence of O–H, C–H, N–H, and C=O groups in RS-g-PAA was consistent with the results of previous studies [16,26,27] who reported FTIR spectra of acrylamide grafted onto cellulose, as shown in Table 2.

Therefore, the spectral shifts and the new absorption bands proved that microwave-assisted grafting successfully produced RS-g-PAA, as shown in Figure 5. Cellulose has poor elasticity, which can be overcome by monomer grafting [18]. RS-g-PAA was obtained by grafting acrylamide onto the rice straw cellulose backbone, forming a grafted structure that improves hydrophilicity, strength, and absorption that shown in Table 4 and Table 6.

3.2. The Effect AA Amount of on Swelling Capacity

Acrylamide (AA) as a monomer is grafted onto the cellulose chain using an ammonium persulfate (APS) initiator and microwave irradiation, forming a grafted polymer called RS-g-PAA. The grafting process produces chemical bonds between the hydroxyl groups of cellulose and the amide groups of acrylamide and allows the formation of some polyacrylamide (PAA) as a result of AA polymerization (Figure 5). This is in line with previous research, that grafting acrylamide onto natural polymers produces hydrogels with high swelling capacity and crosslink density [28]. The mass of AA used was varied to evaluate its effect on the swelling capacity of the bead gel. The results showed that increasing the amount of acrylamide (AA) from 5 g to 15 g resulted in an increase in the grafting ratio and mass of the RS-g-PAA product (Table 3).

The resulting RS-g-PAA was mixed with carrageenan to produce bead gels. The bead gels were then tested for swelling to determine their swelling capacity every 30 min for 180 min. The swelling test results are shown in Figure 6 for aquadest media and Figure 7 for synthetic urine media.

From Figure 6, at 180 min immersion of the sample in aquadest media with a composition of 15 g of AA (sample F) produced the highest swelling capacity, namely 1806.12%. Then, followed by sample E with a composition of 10 g of AA having a swelling capacity of 1204.62% and sample D with a composition of 5 g of AA producing the lowest swelling capacity, namely 697.93%.

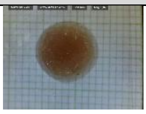
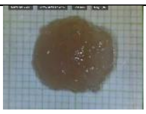
The swelling results in synthetic urine media are shown in Figure 7. Sample F produced the highest swelling capacity, at 1611.58%, compared to sample E which showed a swelling capacity of 949.20% and sample D with a swelling capacity of 528.56%. The swelling capacity in synthetic urine was lowe than ini aquadest because the ions on urine such as Na, Cl, K, reduce the osmotic pressure gradient and limit water uptake by restricting polymer chain expansion. In contrat, ion free aquadest allows the hydrogel to absorb water more freely.

From the Figure 6 and Figure 7, it can be seen that the swelling capacity increased during soaking from 30 to 180 min. The data shows a tendency that in both swelling media, bead gels with sample F has a higher swelling capacity compared to samples E and D.

Table 3. Microwave grafting data on acrylamide variation.

Code	RS-g-PAA (g)	Grafting Ratio (%)	Phase after aceton treatment
D	5.80	487.79	Solid
E	11.32	1032.04	Solid
F	16.22	1521.72	Solid

Table 4. Comparison of acrylamide variations before and after swelling on various media after 180 min.

Code	Appearance of Bead Gel		Swelling Capacity (%)	Media
	Before	After		
D			697.93	Aquadest
			528.56	Synthetic Urine
E			1204.62	Aquadest
			949.20	Synthetic Urine
F			1806.12	Aquadest
			1611.58	Synthetic Urine

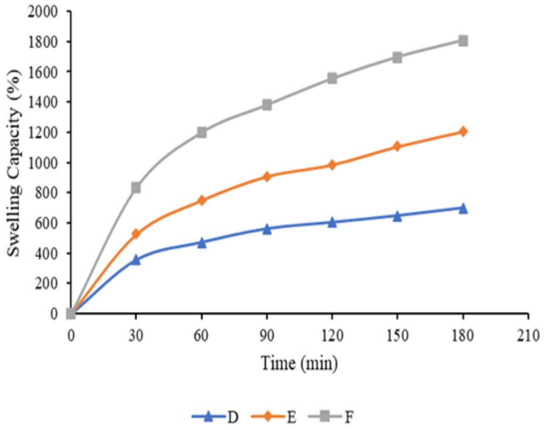


Figure 6. Swelling capacity for acrylamide variation in aquadest.

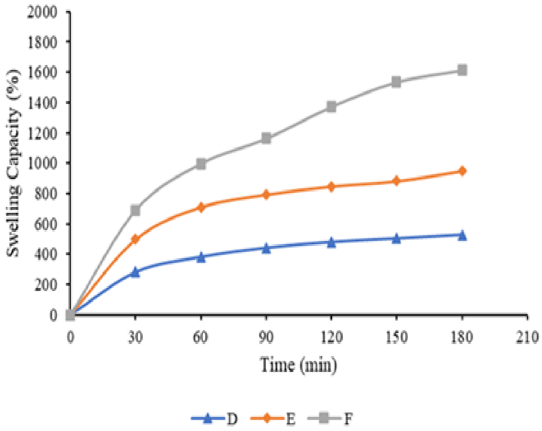


Figure 7. Swelling capacity for acrylamide variation in synthetic urine.

This result is in line with Laksono et al. [29], that the swelling of hydrogel will increase with the addition of acrylamide, this result is due to the hydrophilic nature of the acrylamide monomer will cause an increase in the swelling value when the acrylamide concentration is greater, so that the hydrophilic group binds more water along the hydrogen polymer chain.

In this study, the swelling test was conducted for 180 min following the previous studies that used this duration as a standard measurement time [19]. Figure 6 and 7 show that the swelling capasity curve were still increasing at 180 min, indicating that the equilibrium had not yet been reached.

Based on the results, hydrogels with a higher acrylamide ratio had a greater swelling capacity, which indicated a positive relationship between grafting ratio and swelling capacity at the optimum condition.

Table 4 displays the physical appearance of the bead gel before and after being immersed in aquadest and synthetic urine media for 180 min.

The image of the bead gels in Table 4, one box represents 1 mm. After the soaking process, the bead gel become more transparent and larger in size. Bead gels produced from different acrylamide composition ratio have different swelling capacities. This occurs as the increase in acrylamide ratio enhances the number of hydrophilic amide groups (–$CONH_2$) in the polymer structure, there by increasing the bead gels' ability to absorb water. Furthermore, increasing the concentration of polyacrylamide chains grafted onto the cellulose backbone can expand the porosity and interchain space, strengthening the absorption mechanism. These results are consistent with the findings who reported that increasing the acrylamide ratio increases the swelling ratio, driven by the hydrophilic structure and the formation of an open pore network [30].

3.3. The Effect of APS on Swelling Capacity

Ammonium persulfate (APS) acts as a radical initiator in the grafting process between cellulose and acrylamide [31]. APS generates free radicals that enable acrylamide to graft onto the cellulose backbone, producing RS-g-PAA. In this study, APS was used at 0.05 g, 0.1 g, and 0.15 g. The effect of APS mass variation on the grafting ratio is shown in Table 5. The swelling capacity results in aquadest and synthetic urine media are shown in Figure 8 and 9.

From Figure 8, immersion of the sample in aquadest media with a composition of 0.1 g of APS (sample B) produced the largest swelling capacity (1218.15%). Then, followed by an APS composition of 0.15 g (sample C) having a swelling capacity of 927.98%, and sample A with a composition of 0.05 g of APS producing the lowest swelling capacity (831.90%).

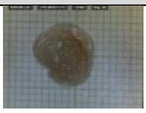
From Figure 9, the sample B produced the highest swelling capacity, at 975.20%, compared to sample C which showed a swelling capacity of 785.26%, and then sample A with a swelling capacity of 328.88%.

Figures 8 and 9 show that sample A had the lowest swelling ratio because limited radical were generated, resulting in poor grafting [32]. The highest swelling was obtained at sample B. In contrast, sample C showed a lower swelling capacity than sample B. Based on the experimental data, the increase in APS concentration from 0.05 g to 0.15 g raised the grafting percentage, but the swelling capacity decreased in 0.15 g APS because the higher crosslinking density made the hydrogel network more compact and restricted its ability to absorb solution [29]. When APS concentrations were above the optimum, homopolymerization or premature termination occurred, producing low molecular weight polymers and reduced swelling [30]. This result was also in agreement with Sadeghi et al. [33], who demonstrated that an excess of radicals from APS accelerated the termination step and promoted self-crosslinking, leading to a den ser gel network and, consequently, a lower water absorption capacity.

Table 5. Microwave grafting results on ammonium persulfate variation.

Code	RS-g-PAA (g)	Grafting Ratio (%)	Phase after aceton treatment
A	10.71	971.25	Solid
B	11.32	1032.04	Solid
C	11.37	1036.68	Solid

Table 6. Comparison of APS variations before and after swelling after 180 min.

Code	Appearance of Bead Gel		Swelling Capacity (%)	Media
	Before	After		
A			831.90	Aquadest
			328.88	Synthetic Urine
B			1218.15	Aquadest
			975.20	Synthetic Urine
C			927.98	Aquadest
			785.26	Synthetic Urine

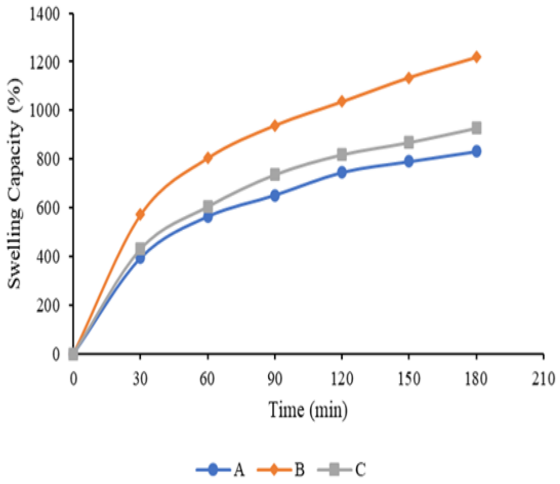


Figure 8. Swelling capacity for ammonium persulfate variation in aquadest.

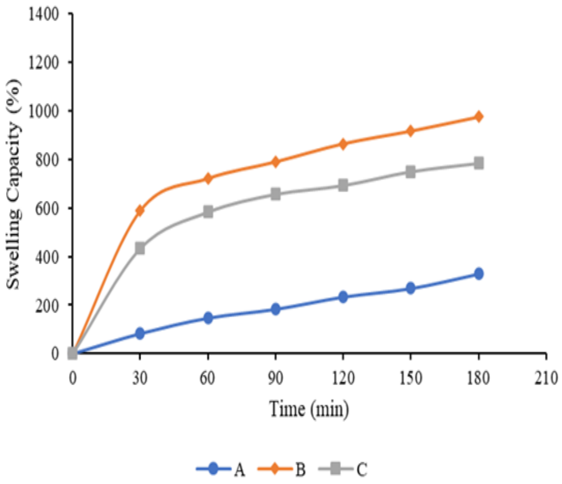


Figure 9. Swelling capacity for ammonium persulfate variation in synthetic urine.

Table 6 shows the physical appearance of the bead gels before and after being soaked in aquadest and synthetic urine media for 180 min.

Based on the bead gel observations in Table 6, increasing the concentration of ammonium persulfate (APS) affected the bead gel size after immersion. Bead gel prepared with the optimum APS concentration were larger and more transparent, indicating greater swelling capacity. This observation was based on qualitative visual comparison of the bead gel photographs, as no quantitative optical measurements were performed. The increased transparency simply reflects greater water uptake, which is consistent both the swelling data shown in Figure 8 and 9. After immersion in aquoeous media, the dried bead gel could swell back into its original bead shape. The swollen bead gel form shows that they have stable structure.

At sufficient levels, APS generates radicals that initiate acrylamide grafting onto rice straw cellulose, there by enhancing polyacrylamide chain formation and porosity to improve water absorption. However, excessive APS produces surplus radicals that trigger side reactions such as homopolymerization and bimolecular termination. This leads to a denser polymer network with short chains and low molecular weight, which inhibits water absorption and reduces swelling capacity [34].

3.4. Media Effect On Swelling Capacity

Swelling results showed that bead gels soaked in synthetic urine had lower absorption capacity than those in aquadest. As reported in previous study, superabsorbent polymers (SAP) absorb more water in aquadest due to the absence of ions, whereas the presence of ions such as Na^+ and Cl^- in urine reduces the osmotic pressure gradient and limits water uptake [35].

Synthetic urine used in this study contained aqueous urea, KCl, NaCl, and Na_2PO_4, providing a significantly higher ionic strenght than ionized water [20]. This difference was clearly shown in Figure 6 and 7 (acrylamide amount variation) as well as Figure 8 and 9 (APS amount variation), where the swelling curves in synthetic urine consistently lower at all immersion times. The lower swelling is also caused by ionic interactions with amide and hydroxyl groups, while Cl^- disrupts hydrogen bonding. This ions also create charge shielding and may form chains, leading to a denser hydrogel network that restricts water penetratiion.

4. CONCLUSION

This study successfully synthesized RS-g-PAA bead gels via microwave assisted grafting. Variation of acrylamide (AA) and ammonium persulfate (APS) significantly affected the swelling capacity of bead gels. FTIR test confirmed successful grafting, indicated by the amide group ($–CONH_2$) in the RS-g-PAA spectrum. The bead gel with 15 g AA showed the highest swelling capacity of 1806.12% in aquadest and 1611.58% in urine solution. Bead gels from APS of 0.10 g produced the highest swelling capacity of 1218.15% in aquadest and 975.20% in urine solution. The bead gels swelled more in aquadest than in synthetic urine, because the absence of ions that hinder water absorption in RS-g-PAA. These results show that rice straw hydrogel is a promising eco-friendly absorbent for diapers. Optimizing the ratio of monomer and initiator is crucial for hydrogel performance.

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