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Preparation and Flocculation Performance of Polyacrylamide Grafted Banana Stem Cellulose: Effect of Acrylamide and Ammonium Peroxydisulfate

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ABSTRACT. Banana stem was identified as an attractive source of cellulose and a promising raw material for developing friendly bioflocculant hydrogels in water treatment. Polyacrylamide grafted banana stem cellulose (BS-g-PAA) was synthesized for flocculation application. The objective of this study was to investigate the effect of ammonium peroxydisulfate (APS) and acrylamide (AA) amount on the grafting efficiency and flocculation performance of obtained BS-g-PAA. In this study, 1 g of banana stem flour was mixed with distilled water, followed by the addition of AA at different weights (10, 15, and 20 g) and APS at various weights (0.1, 0.15, and 0.2 g). The mixture was grafted using microwave irradiation at 540 W for 60 s per cycle, repeated for three cycles (total 180 s). The sample was then dried using an oven until constant weight and ground to obtain BS-g-PAA powder. The results of FTIR showed that the successful grafting of acrylamide onto the cellulose backbone. The grafting percentage increased with the increasing of AA amount, from 1,008% at 10 g AA to 2,098% at 20 g AA. Ammonium peroxydisulfate from 0.1 g to 0.2 g increased grafting percentage from 980% to 1,008%. BS-g-PAA hydrogel prepared by 1 g banana stem, 0.2 g APS, and 10 g AA produced the most effective turbidity reduction by reducing the turbidity value from 53.3 NTU of kaolin suspension to a final value of 15.29 NTU after 80 s of flocculation test. These results demonstrate that hydrogels derived from banana stems have promising potential as sustainable bioflocculants for wastewater treatment.

1. INTRODUCTION

Wastewater treatment is one of the major challenges in the industrial world. It is seen from the increasing amount of liquid waste produced with hazardous chemical content. One method used to address this issue is flocculation. Flocculation is a separation process of fine particles from water by adding a flocculant or coagulant. However, the use of synthetic flocculants such as polyacrylamide often leads to adverse effects, such as the accumulation of harmful substances in the environment and risks to human health [1].

The use of conventional metallic-based coagulants and flocculants to remove suspended particles in drinking water and wastewater is now raising new concerns regarding environmental and health impacts. As an alternative, bioflocculants based on biological agents do not produce chemical residues, generate sludge that is easily making them safe for the environment [2].

In this study, banana stems are selected as the raw material for bioflocculant hydrogels. Indonesia is known as a major banana producer in the world, producing around 7,280,658 tons per year, and this figure is expected to increase every year [3]. Based on data from the Central Statistics Agency and the Directorate General of Horticulture in 2019, Indonesia produced around 7 million tons of bananas from a harvest area of 107,683 ha with a yield of 67.46 tons/ha. In Indonesia, there are 13 types of bananas, and Indonesians typically usthe fruit, leaves, heart, tubers, and roots for processing according to their needs [4]. Banana stems which are generally considered agricultural waste, contain large amounts of 74.37% cellulose, 8% lignin, and 17.7% hemicellulose [5] that may be synthesized into hydrogels with high water absorption capacity. In addition to being abundant and renewable, cellulose from banana stems is environmentally friendly [6], making it a potential source for the production of bioflocculants[7].

Hydrogels are attractive biomaterials due to their large water absorption capacity. Their properties are influenced by the degree, type of cross-linking, tacticity, and crystallinity of their constituent polymers [8].

Hydrogels are defined as three dimensional networks of polymer chains that are insoluble in water but capable of absorbing and retaining large amounts of water [9]. This ability stems from the presence of hydrophilic groups on the polymer backbone, while its resistance to dissolution is determined by the crossnks between chains [10]. In hydrogel synthesis, monomers or polymers, initiators, and crosslinking agents play important roles, as initiators generate free radicals thatrigger the growth of macromolecular chains [11].

The grafting polymerization process is an effective technique for converting cellulose into hydrogels with superior properties. Monomers such as acrylamide (AA) are used to form polymer networks that enhance the hydrogel's ability to absorb water. Acrylamide grafting via microwave-assisted methods has been extensively studied as a technique for modifying natural polymers, such as cellulose, to improve their bioflocculant performance. This process usually involved adding an initiator, such as ammonium peroxydisulfate (APS), to generate radicals under certain conditions [12].

Microwave-assisted grafting methods are increasingly being used because they are faster, more efficient, and more environmentally friendly than conventional methods. Microwave waves accelerate the formation of free radicals without the use of harmful organic solvents, there producing more homogeneous hydrogels with better structural control [13].

Although polyacrylamide-grafted cellulose had been extensively reported as an effective bioflocculant, most previous studies had focused on cellulose derived from conventional sources such as waste paper [14], corn husk [15], psyllium [16], or banana peel [17], and had often emphasized either grafting efficiency or swelling behavior alone. Limited attention had been given to banana stem cellulose, despite its high cellulose content and abundant availability as agricultural waste in Indonesia.

Previous studies on banana stem cellulose had primarily explored its conversion into hydrogels and evaluated swelling or water-tension properties, without investigating acrylamide grafting or flocculation performance [7]. In addition, although cellulose-based hydrogels had been proposed for water purification applications, systematic studies integrating microwave-assisted grafting, initiator–monomer optimization, and turbidity removal efficiency had remained scarce [6].

In this study, the cellulose structure of banana stems was modified by grafting acrylamide monomers onto the cellulose backbone using microwave irradiation with ammonium peroxydisulfate as an initiator. The influence of APS and AA amount on grafting efficiency and bioflocculant performance for kaolin turbidity removal was systematically investigated.

2. MATERIALS AND METHODS

The materials used were aquadest (H_2O), banana stem *(Musa paradisaca L.)*, acrylamide (E.Merck), ammonium peroxydisulfate (Supelco), technical ethanol 95% (Supelco), acetone, and kaolin clay (lissewaxing.id).

2.1 Preparation of Banana Stem

Banana stems were collected from Karanganyar, Jawa Tengah, Indonesia. They were then cleaned and cut into small pieces. The banana stems were then dried in an oven at 80°C for 24 h until dry. The dried banana stems were mashed with a grinder and then sieved using a sieve shaker (AKAMAI) with a 60 mesh. The powder passing through a 60-mesh screen was collected as banana stem flour (BSF).

2.2 Polyacrylamide grafted banana stem cellulose preparation

The production of polyacrylamide grafted banana stem cellulose (BS-g-PAA) was performed by adding 1 g of banana stem flour (expressed as W_{BSF}) into a 1000 mL beaker (Iwaki). Acrylamide (AA) with varied weights (10, 15, and 20 g) was mixed with 50 mL of distilled water. The AA solution was added along with cellulose, and the mixture was stirred at 200 rpm for 30 min. The mixture was then covered with aluminum foil and left to stand for 24 h. After 24 h, the solution was treated with the APS initiator at varying weights (0.1, 0.15, and 0.2 g). The weight was determined using an analytical balance (OHAUS AX124). The mixture was stirred at a speed of 200 rpm for 15 min. The grafting reaction was conducted by irradiating the mixture in a microwave with a turntable (Microwave 700 W Krisbow 20 L). This study was conducted with several variations of APS and AA, with sample codes listed in Table 1.

Table 1. Variations in the composition of (BS-g-PAA) preparation

Code	Component		
	BSF (g)	APS (g)	AA (g)
A	1	0.20	10
B	1	0.20	15
C	1	0.20	20
D	1	0.10	10
E	1	0.15	10

In this study, microwave irradiation was applied at a power of 540 W for 60 s per cycle using a stopwatch (Trusco). The cooling cycle temperature was measured with a thermometer (Gea), which ranged from 30℃, cooled using an ice bath until reaching room temperature [18]. This step was considered one cycle, and the cycle was repeated until the total irradiation time in the microwave matched the required time of 180 s. The sample was left at room temperature for 1 h, and then the liquid phase on top was collected, while the solid phase was mixed with acetone to stop the reaction by adding an excess amount until it was submerged for 30 min, then filtered [19]. The solid sample was dried in an oven at 100℃ for 22 h until a constant weight was achieved. The solid from the oven was ground using a grinder, yielding the polyacrylamide grafted banana stem cellulose (BS-g-PAA). The percentage grafting of this microwave irradiation synthesized BS-g-PAA (W_{BSGP}) was evaluated as Eq. (1) [20].

$$\text{percentage grafting} = \frac{W_{BSGP} - W_{BSF}}{W_{BSGF}} \times 100\% \tag{1}$$

2.3 Characterization of polyacrylamide grafted banana stem cellulose

The functional group of banana stem flour, polyacrylamide grafted banana stem cellulose and acrylamide was investigated using the Fourier Transform Infrared Spectroscopy (FTIR). This test was conducted to identify changes in structure (compound cluster peaks) after the grafting process. The FTIR test was conducted at the Bioprocess Laboratory, Sebelas Maret University, using an FTIR spectrophotometer (Agilent Cary 630) with a wavelength range of 4000-500 cm-1.

2.4 Bioflocculant polyacrylamide grafted banana stem cellulose test using Turbiditymeter

Kaolin solution was prepared by dissolving 10 g of kaolin in 1000 mL of distilled water. BS-g-PAA solution was prepared by dissolving 0.01 g of BS-g-PAA (variations of APS and AA with codes A, B, C, D, E in Table 1) into 20 mL of distilled water. The kaolin solution was separated into 500 mL. The grafted polymer solution was taken to 4 mL and added to a 500 mL kaolin solution containing 10 g/L kaolin in a 1000 mL beaker (total of 6 samples). The sample solutions were stirred using a stirrer in two stages, namely 120 rpm for 2 min, followed by 50 rpm for 10 min. After that, the sample solutions were taken at 0, 20, 40, 60, and 80 s using a centrifuge tube, and the experiment was repeated 3 times to obtain the average Nephelometric Turbidity Unit (NTU) using a turbidimeter (Eutech TN-100). The average NTU value was reported.

3. RESULTS AND DISCUSSION

3.1 FTIR Results

In this study, cellulose is obtained from banana stem and combined with grafted polymers using acrylamide into the backbone of banana stem cellulose. Cellulose as a biopolymer, consists of long chains of glucose units each of which contains three hydroxyl groups (-OH). These -OH groups enhance its hydrophilic properties, its ability to interact with water [21]. To further improve this hydrophilicity, cellulose is grafted with acrylamide, a monomer containing vinyl bonds (C=C) and amide groups ($-CONH_2$). The amide groups form stronger hydrogen bonds than carboxyl groups [22], which makes acrylamide highly soluble in water and polar in nature [23].

A graft copolymer is a type of polymer that consists of a main chain (backbone) derived from natural polymers such as cellulose or starch, with one or more side chains from another polymer covalently grafted onto the backbone. In this study, the grafting of acrylamide is carried out using the microwave grafting method, which produces the graft copolymer shown in Fig. 1.

FTIR analysis is employed to evaluate the success of acrylamide grafting onto the cellulose backbone of banana stem using the microwave grafting method with APS as the initiator. Fig. 2 and Table 2 present the FTIR spectra of banana stem flour, polyacrylamide grafted banana stem cellulose (BS-g-PAA), and acrylamide.

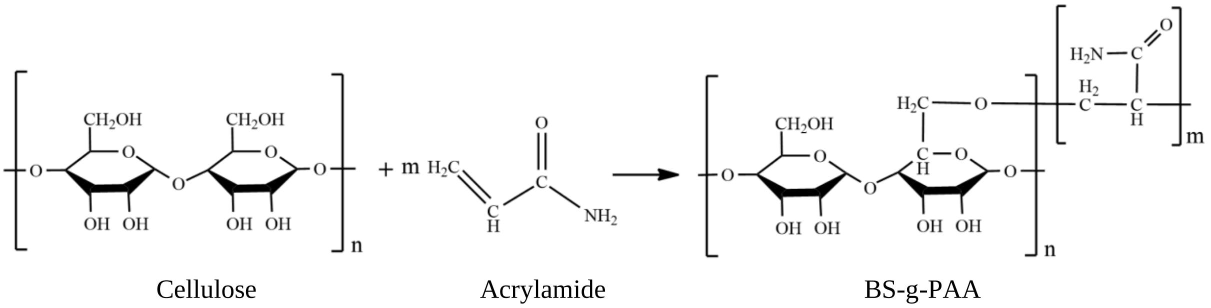

Figure 1. Grafting reaction

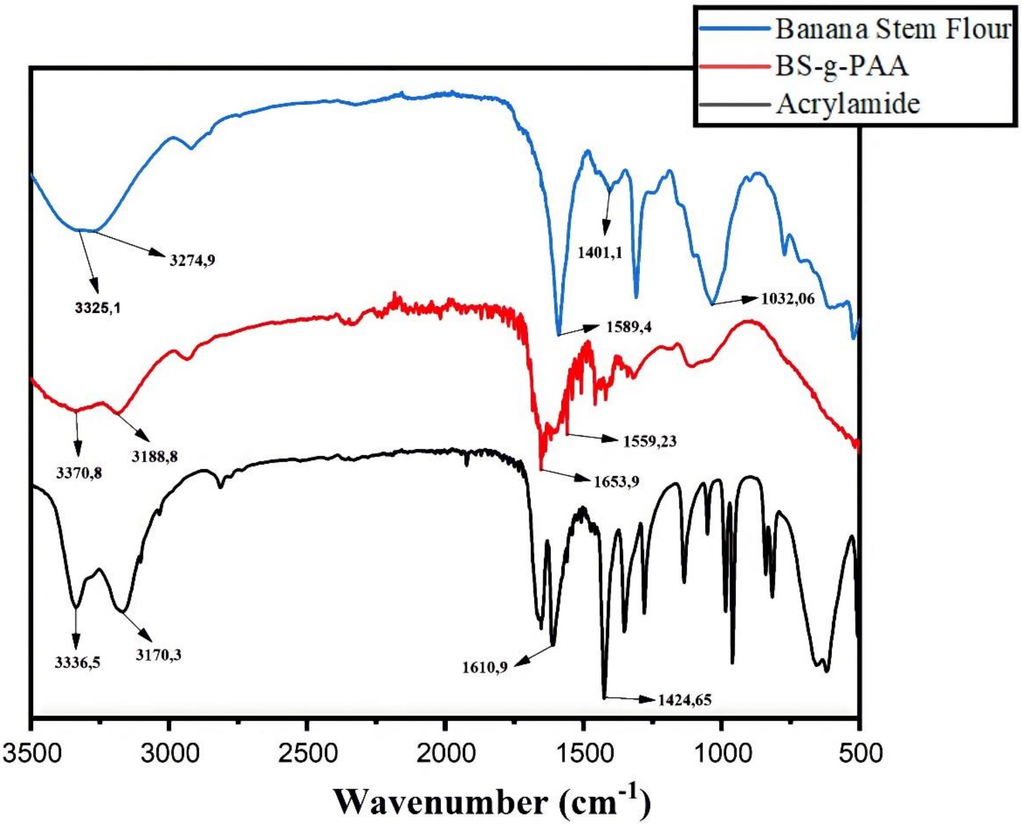

Figure 2. FTIR results

Based on the FTIR spectra, cellulose from banana stem shows O–H stretching at 3325.1 cm^{-1} and a small peak at 3274.9 cm^{-1} corresponding to C–H stretching. The absorption band appearing at around 1032.06 cm^{-1} is assigned to the β-1,4-glycosidic linkage between glucose units, which is a well-known fingerprint band of cellulose, thereby confirming that cellulose is the main structural component of the banana stem flour. The presence of these characteristic cellulose bands provides sufficient qualitative evidence to support the cellulose-rich nature of the banana stem material. The FTIR spectrum of the grafted polymer (BS-g-PAA) exhibits new peaks at 3370.8 cm^{-1}, 3188.8 cm^{-1}, and 1559.2 $cm^{-1,}$ indicating O–H, N–H, and C–H groups, respectively, along with a carbonyl C=O stretching at 1655.9 cm^{-1}. These findings are consistent with spectra of PAM-g-cellulose, who report O–H, C=O, and N–H stretching at 3426.3 cm^{-1}, 1654.7 cm^{-1}, and 1504.5 cm^{-1} in grafted cellulose from waste newspaper [14]. Similarly, [16] observe peak shifts in O–H, C–H, and C–O stretching vibrations after microwave grafting of psyllium cellulose(psy-g-PAM), with additional peaks at 1678.0 cm^{-1} and 1596.99 cm^{-1} corresponding to C=O and N–H stretching, confirming successful grafting. A comparison of functional group assignments from this study with those of [14] and [16] is summarized in Table 2.

Table 2. Comparison of wave numbers from FTIR results

Functional Group	Wave Number (cm^{-1})		
	BS-g-PAA	PAM-g-cellulose [14]	Psy-g-PAM [16]
O-H	3370.8	3426.32	3631.96
C-H	3188.8	3030	2992.16
N-H	1559.2	1504.5	1596.99
C=O	1655.9	1654.7	1678.0

3.2 The Effect of Ammonium Peroxydisulfate (APS) and Acrylamide (AA) Ratio on Percentage Grafting (BS-g-PAA)

The microwave grafting process is carried out using microwave radiation to accelerate the formation of free radicals, which enables the bonding between acrylamide monomers and the cellulose backbone [15]. Fig. 3 and 4 demonstrate the influence of acrylamide and ammonium peroxydisulfate amount on the grafting efficiency of banana stem cellulose. It is shown that the use of microwaves in acrylamide grafting on corn cob cellulose increases the hydrogel swelling capacity, although at excessively high weight of initiators such as potassium peroxodisulfate (KPS), the efficiency is reduced due to the possible formation of homopolymers [15]. A similar study, the hydrogel based on banana peel waste showed that its swelling capacity is higher at lower initiator weight and controlled microwave power [17] As shown in Fig. 3, increasing the AA amount from 10 g to 20 g enhances the grafting percentage from 1,008% (Sample A) to a maximum of 2,098% (Sample C). This trend highlights that greater monomer availability increases the probability of covalent bonding between acrylamide and cellulose backbones. The grafting percentage of sample C was higher than 2,000%. This may be caused by the trace of moisture content in the dried grafted product. Although the purification process included washing unreacted material using aceton, and then drying the sample until a constant weight was attained.

Fig. 4 shows a gradual increase in grafting percentage with higher APS loading, from 980% at 0.1 g APS (Sample D) to 1,008% at 0.2 g APS (Sample A). APS functions as a free radical initiator; thus, higher weight generates more active sites that facilitate grafting reactions with monomers. These findings are consistent with previous studies reporting that the monomer-to-initiator ratio is a key factor in hydrogel synthesis. For instance, Liang et al. [24] observed that acrylamide and acrylic acid grafting onto carboxymethyl cellulose shows maximum efficiency at an optal balance between the monomer and the peroxydisulfate initiator. Similarly, Vijan et al. [25] emphasized that the initiator-to-monomer ratio critically controls grafting efficiency in redox systems.

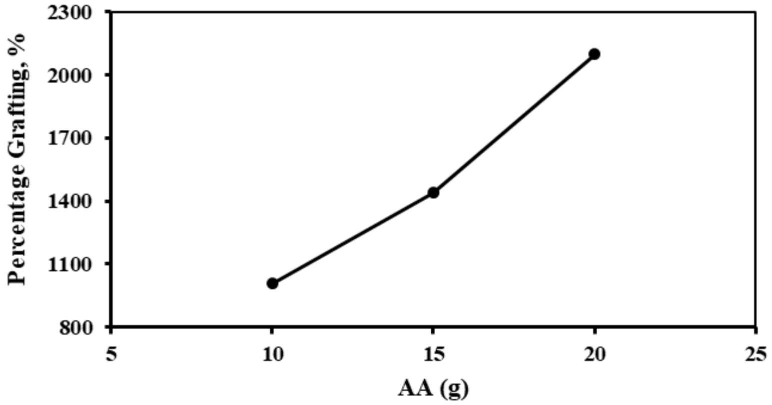

Figure 3. Effect of AA weigth on percentage grafting (APS = 0.2 g, 540 W, 180 s)

However, the weight variations of acrylamide (AA) and ammonium peroxydisulafate (APS) must be maintained within an optimal range. The effect of varying the weight ratio of AA to APS has been widely reported in graft copolymerization studies, where an optimal balance between monomer and initiator is required to achieve maximum grafting performance [25]. The result in Fig. 3 indicates that higher monomer availability increases the probability of effective radical–monomer collisions, leading to more extensive chain propagation on activated cellulose sites. Similar behavior has been reported in microwave-assisted acrylamide grafting onto gellan gum, where an increase in monomer concentration significantly improved grafting yield by enhancing radical utilization efficiency and accelerating diffusion under microwave irradiation [24]. The role of APS as an effective free radical initiator has been widely reported in cellulose-based grafting systems, where an increase in initiator amount increases radical density and promotes graft formation [24]. The efficiency trend observed in Fig. 3 and Fig. 4 suggests that the grafting saturation point has not yet been reached at the tested weight, indicating further potential for optimization. Therefore, these results confirm that monomer and initiator weight play complementary roles in determining grafting efficiency and that the right balance is essential to maximize hydrogel bioflocculant performance.

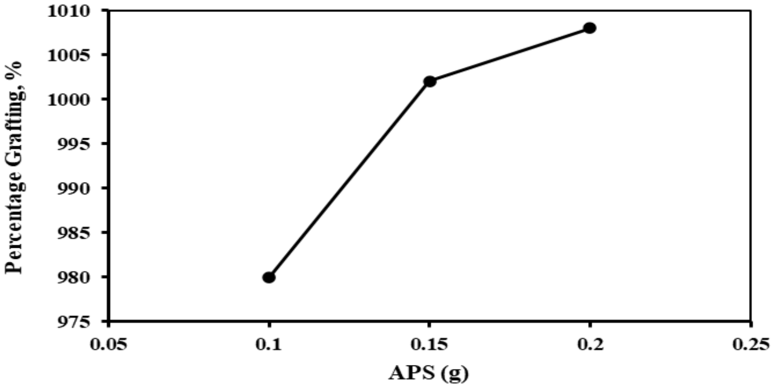

Figure 4. Effect of APS weight on percentage grafting (AA = 20 g, 540 W, 180 s)

3.3 The Effect of APS and Acrylamide on the Effectiveness of Bioflocculants

The effectiveness of bioflocculants produced by grafting acrylamide onto cellulose via microwave treatment is influenced by the addition of an ammonium peroxydisulfate initiator. Free radicals that trigger the formation of covalent bonds between acrylamide monomers and the cellulose backbone are produced by APS. The APS weight must be carefully controlled, as too low a weight is associated with insufficient radical formation, leading to inefficient grafting. Conversely, the formation of unbound acrylamide homopolymers can be promoted by an excessive APS dosage, which can potentially damage the cellulose backbone and reduce flocculation efficiency. It is also shown in the hydrogel of banana peel graft acrylamide [17] that the swelling capacity of hydrogels, as an indicator of water-absorption activity, increases as the APS weight decreases. So, that an excessive initiator amount can actually inhibit water absorption efficiency. Similar findings were reported by previous researchers [15], who observed that a certain weight of APS produced a balanced radical interaction between acrylamide and corn husk cellulose. Therefore, determining the appropriate APS amount is essential for synthesizing effective, environmentally friendly bioflocculants.

Table 3. Turbidity before and after flocculation test at 80 s of BS-g-PAA at different APS and AA amounts

Code	Turbidity kaolin suspension (NTU)	Final turbidity (NTU)	Difference in reduction (NTU)
A	53.3	15.29	38.21
B	53.3	17.90	35.4
C	53.3	18.20	35.1
D	53.3	29.3	24
E	53.3	28.15	25.15

Table 3 summarizes the turbidity of kaolin suspensions before and after flocculation using BS-g-PAA at different APS and AA amount measured at 80 s. Before flocculation, the turbidity of kaolin suspension was identified as the initial turbidity. The initial turbidity of all samples is constant at 53.3 NTU, ensuring that the observed differences in final turbidity and turbidity reduction are attributable solely to the performance of the synthesized flocculants. The results clearly demonstrate that both APS and AA concentrations significantly influence the flocculation efficiency of BS-g-PAA.

Sample A exhibits the lowest final turbidity (15.29 NTU) and the highest turbidity reduction (38.21 NTU), indicating the most effective flocculation performance among all samples. This result is consistent with Fig. 5, where BS-g-PAA synthesized using 0.2 g APS and 10 g AA shows the steepest decline in turbidity over time and reaches the lowest value at 80 s. In contrast, Samples B and C show slightly higher final turbidity values (17.90 and 18.20 NTU, respectively) and lower turbidity reductions (35.4 and 35.1 NTU). This trend agrees with Fig. 5, where BS-g-PAA synthesized with lower APS amount (0.1 g and 0.15 g at a constant AA of 10 g) exhibits a slower and less pronounced decrease in turbidity. Insufficient APS results in fewer initiating radicals, which limits the extent of grafting and produces shorter polymer chains, thereby reducing the effectiveness of the bridging mechanism during flocculation [26]. Samples D and E present significantly higher final turbidity values (29.3 and 28.15 NTU) and lower turbidity reductions (24 and 25.15 NTU, respectively), indicating weaker flocculation performance. These results correlate well with Fig. 6, which illustrates the effect of varying AA amount at a constant APS of 0.2 g.

Fig. 5 shows the effect of ammonium peroxydisulfate (APS) concentration on the flocculation performance of BS-g-PAA in kaolin suspensions at a fixed acrylamide (AA) amount of 10 g. The turbidity of pure kaolin decreases only slightly from 71.367 NTU to 53.3 NTU after 80 s, indicating that natural sedimentation alone is insufficient for effective particle removal without a flocculant [26, 27]. Similarly, raw banana stem flour reduces turbidity from 58.033 NTU to 32.927 NTU, confirming its limited flocculation ability due to the absence of long polymer chains required for efficient particle bridging. In contrast, BS-g-PAA samples synthesized with APS exhibit significantly improved performance, where turbidity decreases from 34.8 NTU to 29.3 NTU at 0.1 g APS and from 34.033 NTU to 28.15 NTU at 0.15 g APS, indicating moderate flocculation efficiency [20, 27]. The best result is achieved at 0.2 g APS, with turbidity reduced from 18.707 NTU to 15.297 NTU, demonstrating the highest turbidity removal among all samples [28, 29]. This enhancement is attributed to a higher degree of grafting, which increases the number of polyacrylamide chains on the cellulose backbone and strengthens particle bridging and adsorption mechanisms [27, 28, 29].

At lower APS concentrations, insufficient radical formation limits grafting efficiency, resulting in shorter polymer chains and fewer active sites for effective particle aggregation [20, 27]. Furthermore, microwave-assisted grafting significantly accelerates polymerization by providing rapid, uniform heating, thereby enhancing radical generation and promoting monomer diffusion into the cellulose structure compared to conventional methods [13, 16]. Previous studies report that grafting without microwave assistance requires longer reaction times and often yields lower grafting yields, resulting ineaker flocculation performance [13, 26]. Overall, the results confirm that both APS concentration and microwave-assisted synthesis play crucial roles in improving the flocculation efficiency of BS-g-PAA for water treatment applications [28, 29].

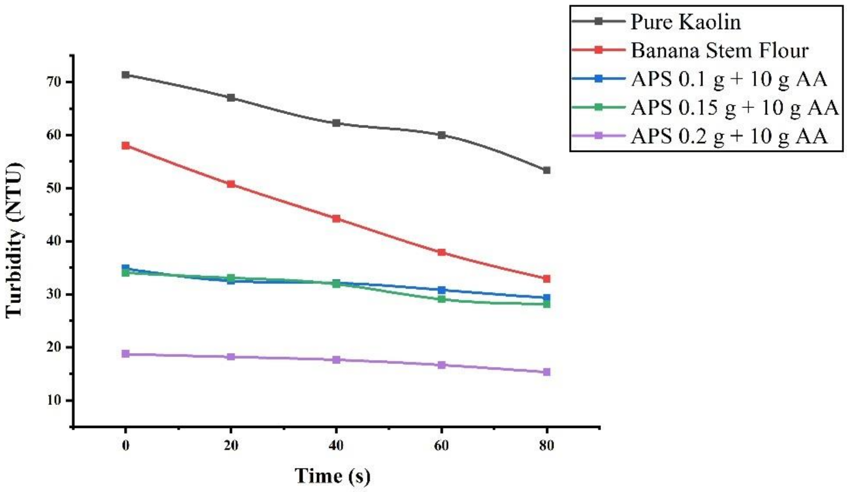

Figure 5. The fffect of APS on the flocculation performance of BS-g-PAA (AA = 10 g, 540 W, 180 s)

Fig. 6 further analyzes the effect of acrylamide concentration on flocculant performance. The turbidity profile shows that 10 g AA produced the most efficient flocculation, with the largest reduction in NTU observed after initial flocculation. The slight difference in performance was observed at 15 g and 20 g AA. The results of this study indicate that although higher monomer concentrations initially increase adsorption sites, excessive AA leads to oversaturation of the polymer chains and a decrease in flocculant performance. This decrease in flocculant performance may be due to the presence of lower polymer chains of acrylic acid groups in cellulose that do not contribute to the flocculation bridging mechanism, resulting in a low water-binding capacity and even disrupting particle aggregation [26, 27]. However, the variation in the weights of acrylamide and ammonium peroxydisulfate should be maintained within the optimal range.

These results emphasize that varying the weight ratios of ammonium peroxydisulfate and acrylamide is a crucial factor in determining hydrogel structure and flocculation efficiency. APS generates sufficient free radicals for effective grafting, while AA expands the hydrogel network, thereby enhancing adsorption capacity. A balanced ratio ensures the formation of longer, more reactive polymer chains, which strengthen bridging interactions with suspended particles, thereby improving flocculation efficiency [29]. However, monomer levels exceeding the optimal threshold trigger particle restabilization, reducing turbidity removal efficiency. Similar observations have been reported in systems incorporating multiple monomers, where optimized polymer network wrinkling enhances bridging activity [30].

Overall, both experimental findings and the literature confirm that the success of BS-g-PAA as a bioflocculant depends not only on the presence of active components but also on the proper balance between monomer and initiator amounts, which governs the formation of a structurally and functionally optimal grafted hydrogel network.

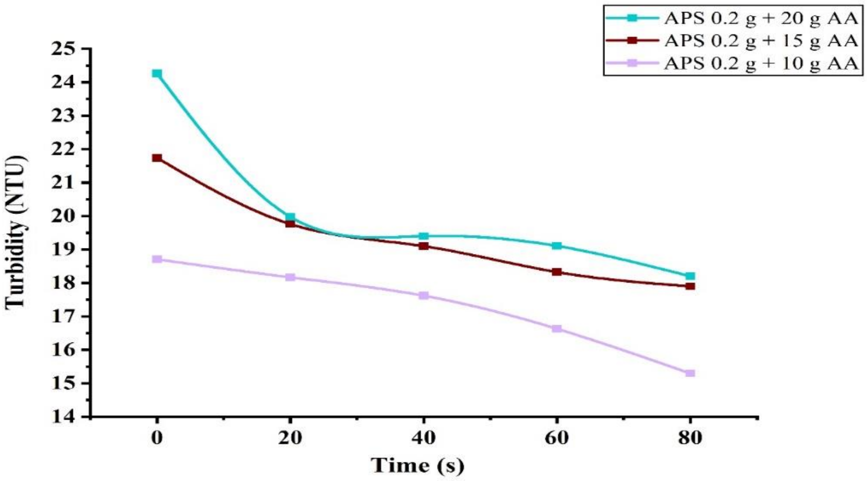

Figure 6. The effect of acrylamide on the flocculation performance of BS-g-PAA (APS = 0.2 g, 540 W, 180 s).

4. CONCLUSION

Based on the research, it can be concluded that banana stems have been proven to be usable as a base material for the production of cellulose-based hydrogels through a grafting process with acrylamide, using various amounts of ammonium peroxydisulfate (APS) initiators at 0.1, 0.15, and 0.20 g. The success of the grafting process was confirmed by FTIR spectroscopy. The addition of ammonium peroxydisulfate (APS) and acrylamide exhibits effectiveness as a bioflocculant. From the research results, the polyacrylamide grafted from banana stems (BS-g-PAA) showed optimal effectiveness at a ratio of 0.2 g ammonium peroxydisulfate (APS) and 10 g acrylamide with microwave irradiation at 540 W for 180 s, resulting in the most efficient flocculation with the highest reduction difference of 38.21 NTU. The production of a hydrogel bioflocculant from banana stems, with the addition of ammonium peroxydisulfate (APS) and acrylamide as an initiator, using the microwave grafting method, has been successfully achieved.

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