Preparation and Characterization of a New Coagulant Based on the Sago Starch Biopolymer and Its Application in Water Turbidity Removal

I. Y. Qudsieh,¹ A. Fakhru' l-Razi,² N. A. Kabbashi,¹ M. E. S. Mirghani,¹ K. G. Fandi,³ M. Z. Alam,¹ S. A. Muyibi,¹ M. M. Nasef⁴

¹Bioenvironmental Research Unit (BERU), Department of Biotechnology Engineering, Faculty of Engineering, International Islamic University Malaysia, P.O. Box 10, Kuala Lumpur 50728, Malaysia ²Department of Chemical and Environmental Engineering, Faculty of Engineering, University of Putra Malaysia, Serdang, Selangor DE 43400, Malaysia ³Department of Piclosy, Faculty of Science, Al Hussein, Pin Talal University, Ma'an, Jordan

³Department of Biology, Faculty of Science, Al-Hussein Bin Talal University, Ma'an, Jordan ⁴Business and Advanced Technology Centre, University of Technology Malaysia, Jalan Semarak, Kuala Lumpur 54100, Malaysia

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ABSTRACT: A new organic coagulant, sago starch (SS)graft-polyacrylamide (PAm), was prepared by the cericion-induced redox polymerization of acrylamide (Am) onto SS at room temperature. The effects of the variation of the concentration of Am and the initiator on the percentages of yield and total conversion were investigated. The chemical composition, viscosity, and side-chain-average molecular weight of the obtained graft copolymers were determined. The newly obtained coagulant was tested for

INTRODUCTION

The modification of raw starch has been recently the subject of extensive investigation. This is because of the applicability of the obtained modified starch to a variety of uses of industrial interest, including as sizing agents for textiles and paper, adhesives for corrugated and laminated paper boards and wall papers, flocculants, binders, fabric printing aids, thickeners, and many other nonfood industrial applications.¹⁻³ This has been motivated by the abundance, renewability, and low cost of starch in addition to its biodegradability, which can be transferred to synthetic polymers when the starch is incorporated with them. The presence of the starchy fraction in such polymers make them easily susceptible to microorganism attack, which leads to decomposition of the polymers to particles small enough to cause minimal damage to the environment.^{4,5}

Sago starch (SS) is the most abundant carbohydrate in Malaysia that is produced commercially and is ranked fifth among important commodities with a production capacity of 30,000 tons per year. Curthe treatment of the turbidity of water. The SS-g-PAm coagulants were found to achieve water turbidity removal up to 96.6%. The results of this study suggest that SS-g-PAm copolymer is a potential coagulant for reducing turbidity during water treatment. © 2008 Wiley Periodicals, Inc. J Appl Polym Sci 109: 3140-3147, 2008

Key words: biopolymers; graft copolymers; polyelectrolytes; water-soluble polymers

rently, the price of this commodity is very low, and its uses are mainly limited to food additives and animal feeds.²⁻⁵ To diversify the demands for different SS products, various modification methods have been exploited, including enzymatic, physical, and chemical modification. Among all of these methods, the chemical modification of starch has been found to be more attractive and can be performed in various forms, including oxidation, crosslinking, and graft copolymerization.^{2–4,6–10}

Graft copolymerization has been used as an important technique for modifying the physical and chemical properties of polymers. The reaction originates from the formation of active sites or radicals on the polymer backbone and the exposure of these sites/radicals to a monomer, mostly proceeding with radical polymerization mechanism. In many а instances, chain-transfer reactions are involved by the hydrogen removal atoms. The application of the graft copolymerization technique in starch modification has led to the improvement of starch properties and the enlargement of its range of utilization.^{3,11–17}

One of the applications that is receiving increasing attention is the use of the modified starch and fellow natural polymers, such as cellulose, alginic acid, and guar gum, as flocculants/retention aids in water treatment processes. Initially, the biodegradability of

Correspondence to: I. Y. Qudsieh (isamyq@yahoo.com).

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such natural polymers was found to be a major drawback for flocculation because it reduces the storage life and efficiency of flocculants because of the decrease in molecular weight. Later, many attempts were made to integrate synthetic and natural polymers to obtain graft copolymers with a reasonable combination of biodegradability and shear resistance.^{18–21}

In this context, the grafting of acrylamide (Am) onto a rigid polysaccharide backbone was found to produce less biodegradable and reasonably shear-resistant graft copolymers that had appealing flocculation properties.^{18,20,22,23} For example, amylopectin-*graft*-polyacrylamide (PAm) and amylose-*graft*-PAm were found to have good flocculation properties despite the superiority of the former caused by its high molecular weight and branched nature.^{16,17} Also, polysaccharide-based graft copolymers have shown good flocculation qualities onto iron ore¹⁷ and kaolin suspension.^{17,22}

In this study, the flocculation characteristics and performance of laboratory prepared SS-*g*-PAm prepared by the graft copolymerization of Am onto SS with a ceric ion initiator was studied for the removal of turbidity of kaolin suspension.¹³

EXPERIMENTAL

Materials

SS, genus *Metroxylon ssp.* (industrial grade), was supplied Wee Kwong Sdn Bhd (Sarawak, Malaysia) and used without further purification. Am was obtained from BDH (United Kingdom) and was used as received. Other chemicals, such as sodium bicarbonate (BDH, United Kingdom), ceric ammonium nitrate (CAN; BDH, Australia), hydrochloric acid (Hamburg Chemicals, Germany), acetone (Merck, Germany), *N*,*N*-dimethylformamide (DMF; Fluka Chemie, Switzerland), acetic acid, and nitric acid (China National Chemicals Import, Export Corp., China), were analytical grade and were used as received. Distilled water was used as a solvent for all of the graft copolymerization reactions. Kaolin (aluminum silicate) was obtained from a local commercial source.

Graft copolymerization

The preparation of SS-*g*-PAm was carried out with a redox initiation process involving ceric ions and according to a procedure reported earlier.^{11–13} We gelatinized SS by heating its slurry (55.6 mmol anhydroglucose units) in a flask for 30 min at 80°C under a N₂ atmosphere. The obtained gelatinized SS was used as a starting substrate in all of the grafting reactions, which were carried out in a round-bottom flask equipped with a reflux condenser, a nitrogen gas inlet, and a water bath with a magnetic stirrer to

control the temperature. We started the initiation process to produce free radicals onto SS chains by adding CAN initiator prepared by dissolving a desired amount (0.55 or 1.09 mmol) in 10 mL of distilled water to the gelatinized SS (cooled to 30°C) 15 min before adding the monomer. The Am was then added to the reactant mixture at a prescribed concentration (84.4 or 113 mmol), and the propagation reaction was allowed to proceed under a N2 atmosphere and continuous stirring for a chosen period of time (3 h). The reaction was terminated by the addition of 3-4 drops of 0.1M nitric acid to the mixture during stirring followed by the addition of a saturated solution of hydroquinone. The obtained graft copolymer was precipitated into 1 L of acetone, collected, and then dried in an oven at 60°C to a constant weight.

Extraction of the homopolymer and determination of the yield percentage (Y%) and total conversion percentage (TC%)

It is an established fact that the redox-initiated process of graft copolymerization onto the polysaccharide backbone generates free radicals on the polysaccharide backbone itself, which leaves little scope of homopolymer formation.¹⁷ Studies on homopolymerization by Owen and Shen²⁴ suggested that no homopolymer is formed upon the use of monomer and CAN concentrations of less than 2.0 and 0.1M, respectively. It has been reported that the treatment of the graft copolymers by a mixture of DMF and acetic acid (1:1 v/v) could remove the PAm homopolymer by Soxhlet extraction.²⁵ Therefore, a mixture of DMF and acetic acid (1:1 v/v) was used as the solvent for extraction of the homopolymer with a Soxhlet extractor for 8 h. The obtained graft copolymer (SS-g-PAm) was dried in an oven at 60°C to a constant weight. To ensure the complete removal of any homopolymer, the extraction process was repeated three times for each sample. Y% of the graft copolymerization reaction and TC% were calculated as follows:

$$TC\% = \frac{W_3 - W_1}{W_2} \times 100$$
 (1)

$$Y\% = \frac{W_3}{W_1 + W_2} \times 100$$
 (2)

where W_1 , W_2 , and W_3 are the weights of SS, Am, and SS-*g*-PAm, respectively.

Viscosity $[\eta]$ measurement and determination of the side-chain-average molecular weight (M_v)

Destruction of the SS component in SS-g-PAm was carried out by acid hydrolysis. We carried this by

heating under reflux 1.0 g of the copolymer in 100 mL of 0.5*M* hydrochloric acid for 6 h at 70°C. The residue was then filtered, washed with acetone, and dried in the oven at 60°C.²⁶ To measure [η] and M_{v} , an Ubbelohde viscometer (Germany) was used. PAm solution was prepared by the dissolution of a desired amount of dry PAm, obtained from the acid hydrolysis conducted in a previous step, in distilled water at room temperature. The Huggins equations were used to estimate the [η] values of the PAm solution as follows:

$$\eta_{sp} = [\eta] + K'[\eta]^2 C \tag{3}$$

$$\eta_{sp} = \eta_{red} C \tag{4}$$

$$\eta_{\rm red} = t_i - t_0 / C_i t_o \tag{5}$$

where η_{sp} is the specific viscosity, η_{red} is the reduced viscosity, *C* is the concentration of the polymer, *K'* is the Huggins constant, and t_i and t_o are the times required for the solvent with or without polymer, respectively, to pass through the Ubbelohde viscometer at concentration C_i . The values of $[\eta]$ were determined from the intercepts of the straight line of $(\eta_{sp}/C) - C$ plot.

 \dot{M}_v of PAm was estimated with the Mark–Houwink equation as reported in the literature:^{11,12,15,27}

$$[\eta] = KMv^a \tag{6}$$

where $K = 6.31 \times 10^{-3}$ g/mL and a = 0.80 for PAm in water. *K* and *a* are empirical (Mark-Houwink) constants.

Preparation of synthetic turbid water

A synthetic kaolin suspension was prepared with 10 g of kaolin in 1000 mL of distilled water. A 50-mg/L sodium bicarbonate solution from a previously prepared stock solution of 1000 mg/L was added to the kaolin suspension and made up to 1000 mL. The kaolin suspension prepared solution was placed under slow stirring (20 rpm) for 30 min to ensure the uniform dispersion of kaolin particles. The kaolin suspension was then allowed to stand for 24 h to complete the hydration of the kaolin particles.^{13,28–30} This solution was then used as a stock solution for the preparation of water samples with 200 NTU turbidity to test the newly prepared coagulant. The pH of all water samples was kept constant at a value of 7.0 (\pm 0.2).

Flocculation jar test

The flocculation test of kaolin turbid water was carried out in a standard flocculation jar apparatus (Chemix Floc Tester model CL5, UK) according to Bratby³¹ with minor modifications. The apparatus was equipped with a six-bladed stirrer connected to a variable speed motor through a gear system. In each 600-mL beaker, 300 mL of 200-NTU kaolin turbid water was placed. The beakers were placed on the flocculator, and the coagulant solution was added to each beaker under low stirring conditions to make a predetermined coagulant dosage (0.1-1.2 mg/L) with respect to the turbid water volume. Immediately after the addition of the coagulant, the suspensions were stirred at a constant speed of 75 rpm for 4 min, which was followed by slow stirring at 25 rpm for 5 min. The flocs were allowed to precipitate for 30 min. Samples of clean supernatant liquid were drawn from a depth of about 1 cm and subjected to turbidity measurements with a digital nephelo turbidity meter (Hanna Instruments, model HI 93703 microprocessor), which allowed us to express the turbidity in nephelometric turbidity units.¹³

RESULTS AND DISCUSSION

Graft copolymerization

The graft polymerization of Am onto SS in this system was carried out by a ceric-ion-induced redox initiation method. Three SS-g-PAm coagulants of different Y%'s and TC%'s and denoted as SS-g-PAm1, SS-g-PAm2, and SS-g-PAm3, were prepared by variation of the amounts of Am and the initiator during the graft polymerization reactions. The selection of this method despite the availability of other ones^{25,31} was prompted by the fact that the redox process initiates free-radical sites exclusively on the polysaccharide backbone (SS in this case) and, thus, provides little scope for homopolymer formation. It was also proven that the addition of CAN to Am under the same grafting conditions without the introduction of SS did not initiate the graft polymerization reaction. Therefore, the introduction of SS was essential to the creation of the free-radical sites on its backbone to initiate the graft polymerization of the Am monomer. Because it is known that polysaccharides with rigid backbones are shear stable and synthetic polymers such as PAm are shear degradable, the obtained SS-g-PAm should have been fairly more stable than that of pure PAm. This was supported by the findings of Deshmukh et al.32 and Deshmukh and Singh,²³ who found that the graft copolymerization of xanthan gum and Am improved the stability of the obtained graft copolymer by the same order as that of the polysaccharide itself, compared to PAm alone. Further proof of the grafting of Am and the absence of homopolymerization in such systems was reported by Owen and Shen.²⁴

Graft copolymer	SS (mmol) ^a	Am (mmol)	CAN (mmol)	TC%	Y%	[η] (mL/g)	[η] fitting results and R^2	$M_v (\times 10^{-5})$
SS-g-PAm1								
$R^2 = 0.9784$	55.6	84.4	0.55	72.9	89.2	251.97	Y = 35.992X + 251.97	5.6
SS-g-PAm2								
$\bar{R}^2 = 0.9396$	55.6	84.4	1.09	51.5	80.6	157.52	Y = 49.539X + 157.52	3.1
SS-g-PAm3								
$R^2 = 0.9225$	55.6	113	0.55	88.6	94.6	314.43	Y = 41.898X + 314.43	7.4

 TABLE I

 Summary of the Preparation Conditions and Properties of Various Samples of SS-g-PAm

^a 55.6 mmol in terms of the anhydroglucose unit.

To prevent the formation of homopolymer in this study, the concentrations of the Am and CAN were kept lower than the critical concentrations that initiate homopolymerization. This was evident from the negligible amount of homopolymer obtained on extraction of the graft copolymers in a DMF–acetic acid (1 : 1 v/v) mixture to remove the PAm homopolymer.

A summary of the preparation conditions and properties of various SS-*g*-PAm graft copolymers, including the amounts of reactants, Y%, TC%, [η] fit results, R^2 , and M_v , is presented in Table I.

As shown, increasing the concentration of the initiator (CAN) from 0.55 to 1.09 mmol at constant SS and Am concentrations, caused a reduction in Y% and TC% by about 9 and 21%, respectively. In the mean time, increasing the Am concentration in the reaction mixture at a constant CAN concentration caused a reduction in both Y% and TC%, which was dramatic in the former (~ 64%). These results suggest that the graft polymerization of SS and Am was favored over homopolymerization at low concentrations of the initiator (CAN). To attain a compact shape in solution, the obtained copolymer had to contain a mixture a number of graft copolymers with various numbers and lengths of PAm chains.³³

Table I shows two possible changes in the average molecular weight. For instance, we had either a small number of long PAm chains (as in SS-g-PAm1) or a large number of short PAm chains (as in SS-g-PAm2). In the former case, the compact shape of the original SS would have been changed because of the presence of long PAm chains. This would have resulted in a larger hydrodynamic volume and would have led to a higher $[\eta]$, as shown in Figure 1. On the other hand, the large number of short PAm chains (as in SS-g-PAm2) was expected to alter the original compact shape to a great extent, thus retaining its lower hydrodynamic volume, which should have been reflected again in its low $[\eta]$ and M_v (Fig. 2). Comparing the three graft copolymers in Table I, we observed the change in $[\eta]$ between SS-*g*-PAm1 and SS-g-PAm2. CAN concentration in SS-g-PAm2 was purposely doubled to reduce the length of PAm chains because a greater number of freeradical sites (created by the higher concentration of CAN) was most likely to compete for the same Am concentration. These results can be understood on the basis of the fact that $[\eta]$ of the polymer was not only dependent on the developing structure of the



Figure 1 $[\eta]$'s of various SS-*g*-PAm samples versus the concentration (g/mL).



Figure 2 $[\eta]$'s of the graft copolymers (prepared coagulants SS-*g*-PAm1, SS-*g*-PAm2, and SS-*g*-PAm3) versus M_v .

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Scheme 1 Proposed reaction mechanism for the grafting of PAm onto SS.

polymer in the solution but also on its molecular weight.^{12,33,34}

 $\eta_{\rm sp}$ and $\eta_{\rm red}$ were experimentally determined from eqs. (4) and (5), respectively. Values of $[\eta]$ were determined from the intercept of the straight lines (as shown in Fig. 1) for the three graft copolymer

samples, SS-*g*-PAm1, SS-*g*-PAm2, and SS-*g*-PAm3, and were found to be 251.97, 157.52, and 314.43 mL/g, respectively, as shown in Table I. Values of M_{ν} , which were then calculated for the copolymers, were found to be 5.6 × 10⁵, 3.1 × 10⁵, and 7.4 × 10⁵, respectively (as depicted in Fig. 2). The increase in

	-	
SS-g-PAm1	SS-g-PAm2	SS-g-PAm3
200	200	200
6.3	6.3	6.3
75	75	75
4	4	4
25	25	25
5	5	5
30	30	30
0.6	0.6	0.6
94.1	92.0	96.6
11.8 ± 0.4	16.0 ± 0.3	6.8 ± 0.8
12.1 ± 0.2	16.2 ± 0.5	7.0 ± 0.6
	$\begin{array}{r} \text{SS-g-PAm1} \\ 200 \\ 6.3 \\ 75 \\ 4 \\ 25 \\ 5 \\ 30 \\ 0.6 \\ 94.1 \\ 11.8 \pm 0.4 \\ 12.1 \pm 0.2 \end{array}$	$\begin{array}{c cccc} SS-g-PAm1 & SS-g-PAm2 \\ \hline 200 & 200 \\ 6.3 & 6.3 \\ 75 & 75 \\ 4 & 4 \\ 25 & 25 \\ 5 & 5 \\ 30 & 30 \\ 0.6 & 0.6 \\ 94.1 & 92.0 \\ 11.8 \pm 0.4 & 16.0 \pm 0.3 \\ 12.1 \pm 0.2 & 16.2 \pm 0.5 \\ \end{array}$

 TABLE II

 Summary of the Coagulation Performance Conditions of SS-g-PAm

 $[\eta]$ was accompanied by an increase in M_{v} , as shown in Figure 2. This was because of the increase in the side chain length of the copolymers.

 $[\eta]$, which is a measure of the hydrodynamic volume of a polymer in solution, is lower for a branched polymer compared to a linear polymer of the same molecular weight. This means that in a series of graft copolymers, the longer the grafted chains are, the higher the hydrodynamic volume will be; hence, $[\eta]$, as well as the average molecular weight, will rise. Similarly, when SS-g-PAm3 was compared with SS-g-PAm1, a fall in $[\eta]$ value was observed (314.43 and 251.97 mL/g), respectively. This was because of the shorter length of the PAm chains, which resulted in a lower Am concentration at a constant CAN concentration. Generally, an increase in the monomer concentration at given grafting time will cause an increase in the rate and the grafting yield. However, both the final grafting yield and the rate of grafting tended to level off and reach a saturation state with longer periods of grafting time and at certain monomer concentrations, beyond which a further increase caused a sharp fall in TC%. Furthermore, in grafting systems where the polymer substrate scarcely swells in the grafting mixture, such a decreasing effect is attributed to the suppression of monomer diffusion by an increase in the $[\eta]$ of the grafting mixture under the effect of homopolymerization and might be attributed to the availability of the monomer backbone grafting sites, and simply grafting is a diffusion-controlled process.³⁵ These results were in agreement with those obtained by Rath and Singh,¹⁸ Tripathy et al.,³⁶ Tripathy and Singh,³⁷ Durand and Hourdet,³⁸ and Tripathy et al.³⁹

Grafting sites and the copolymerization reaction

In the presence of Am vinyl monomer, SS macroradicals were added to the double bond of Am, which resulted in covalent bond formation between the monomer and SS with the creation of a free radical on the monomer; that is, a chain was initiated. Subsequent addition of the monomer molecule to the initiated chain propagated grafting onto SS. Finally, the termination of the graft copolymerization was through a combination of two radicals in the system, which produced grafted SS or a homopolymer. It was assumed that free radicals were formed on the C2 of anhydroglucosamine rings when ceric ion was used to initiate grafting. Ceric ion was attached to SS to produce a SS-ceric complex (Scheme 1). Ce⁴⁺ ion was reduced to Ce³⁺ ion with the release of a proton. Although the bond between C2 and C3 was broken, free radicals of SS were formed. The free radicals formed then reacted with PAm to produce the copolymer.^{11,12}

Performance of the graft copolymers as coagulant for the treatment of water turbidity

The performance of the newly prepared coagulants was tested with freshly prepared artificial turbid water containing a kaolin suspension (200 NTU), as illustrated in Table II. The results of the coagulation performances of SS-g-PAm1, SS-g-PAm2, and SS-g-PAm3 are presented in Figures 3–5. All results were obtained under the following conditions: a pH of 6.3, rapid mixing speed of 75 rpm for 4 min followed by a slow mixing speed of 25 rpm for 5 min, and a settling time of 30 min. Figure 3 shows the relationship between the residual turbidity and the concentration of the SS-g-PAm1 coagulant. As shown, the addition of 1-mg/L coagulant caused a drastic drop in the residual turbidity from 200 NTU to lower than 20 NTU. A further increase in the coagulant concentration caused more but slow reduction in the residual turbidity. The most effective dosage of SS-g-PAm1 that achieved the lowest residual turbidity (11.8 NTU) was found to be 0.6 mg/L beyond which the residual turbidity started rising because of the beginning of the restabilization of the flocculated particles. Lowering the effective dosage to 0.5 mg/L only added a tiny increase in



Figure 3 Coagulation performance of SS-*g*-PAm1 for the treatment of synthetic turbid water (200 NTU).

the residual turbidity (0.3 NTU), which could be neglected when we took into consideration the reduction of 0.1 mg/L in chemical dosage in terms of cost saving, which would be quite substantial when applied on a large scale. These results were in a complete agreement with our previous study.¹³ These results also agreed with those obtained by Tripathy et al.³⁶ for a coagulant composed of Am grafted onto sodium alginate.

Similarly, as shown in Figure 4, the effective dosage of SS-g-PAm2 that achieved the lowest residual turbidity (16.0 NTU) was found to be 0.6 mg/L. Interestingly, the addition of a 0.5-mg/L dosage resulted in a residual turbidity value 0.2 NTU higher than that caused by the 0.6-mg/L dosage. This suggests that 0.5 mg/L can be used as an effective dosage when this coagulant is considered for large-scale application, as a substantial cost savings can be made. This trend was similar to that of SS-g-PAm1, although the latter presented a decrease in the percentage of removal by 94.1% compared to 92% for

Figure 4 Coagulation performance of SS-*g*-PAm2 for the treatment of synthetic turbid water (200 NTU).

SS-*g*-PAm2. Such a difference may have been due to the increase in the average molecular weight of SS-*g*-PAm1, which reflected the longer chains of PAm attached to the SS backbone compared to those in SS-*g*-PAm2.

The effective dosage of SS-g-PAm3 that achieved lowest residual turbidity (6.8 NTU) was found to 0.6 mg/L, as shown in Figure 5. However, a 0.5-mg/L dosage is expected to maintain a substantial cost reduction if applied on a large scale, as discussed for both SS-g-PAm1 and SS-g-PAm2. This is because the difference between the residual turbidity at 0.6and 0.5-mg/L dosages was only 0.2 NTU. The increase in the percentage removal with SS-g-PAm3 to 96.6%, compared to 94.1% for SS-g-PAm1 and 92.0% for SS-g-PAm2, was due to the increase in the average molecular weight of SS-g-PAm3 and the presence of long chains of PAm attached to the SS backbone. These findings were in good agreement with those of Tripathy and Singh,³⁷ who studied the performance of Am-grafted sodium alginate in comparison with some other laboratory synthesized and commercially available flocculants.

The coagulation performance of the SS-*g*-PAm prepared in this study can be understood on the basis of two main predominant mechanisms involving polymeric flocculation, that is, charge neutralization and bridging mechanisms, both of which are functions of average molecular weight, charge density, and the charge of the particles.^{13,40,41–50} The charge neutralization effect played its role in the adsorption of the suspended particles in water on the surface of the SS-*g*-PAm copolymers during the rapid-mixing formation of macroparticles, which subsequently resulted in floc formation.^{21,47} As the initial turbidity of the water increased, the particles in the suspension increased, and therefore, the opportunity for particle aggregation and charge neutralization



Figure 5 Coagulation performance of SS-*g*-PAm3 for the treatment of synthetic turbid water (200 NTU).

increased. This led to an increase in floc formation through a charge neutralization mechanism, and as a result, the turbidity removal was raised. Hence, expectedly, the flocculation process was dominated by a bridging mechanism. The efficiency of SS-*g*-PAm on the kaolin suspension may have another explanation through the higher average molecular weight and the longer PAm chains on the SS backbone.¹³ Similar results were reported by Tripathy³⁶ and Karmakar,¹⁷ who grafted Am onto sodium alginate and amylopectin, respectively. In the flocculation process by grafted polysaccharides, the dangling branches of flexible PAm enhanced easier approachability.

CONCLUSIONS

Organic coagulants composed of SS-g-PAm were successfully prepared by the graft copolymerization of Am onto SS with a ceric ion initiator. Both TC% and Y% were found to be functions of the concentrations of monomer and initiator involved in the reaction. M_v of the coagulants was observed to increase with increasing TC% and Y%. The obtained SS-g-PAm copolymers were found to effectively flocculate the kaolin suspension in water. This suggests that the coagulant obtained in this study has the potential to be used for turbidity removal in water and wastewater treatment processes.

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