

# Flocculation Studies Based on Water-Soluble Polymers of Grafted Carboxymethyl Cellulose and Polyacrylamide

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**ABSTRACT:** Graft copolymers of carboxymethylcellulose and polyacrylamide were synthesized by a ceric-ion-induced solution polymerization technique. By the variation of the monomer and catalyst concentrations, six grades of graft copolymers were synthesized. The synthesized graft copolymers were characterized with intrinsic viscosity measurements, IR spectroscopy, and X-ray diffractometry. The flocculation characteristics of the graft copolymers were

studied in kaolin (5 wt %), iron-ore (10 wt %), and silica (10 wt %) suspensions. The flocculation characteristics of the best performing graft copolymer were then compared with those of various commercially available flocculants. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1000–1007, 2006

**Key words:** graft copolymers; polysaccharides; viscosity; water-soluble polymers

## INTRODUCTION

Many polysaccharides play important roles in science and technology because of their unique properties. Water-soluble polymers based on grafted polysaccharides have drawn much attention in recent decades because of their controlled biodegradability, shear stability, and high efficiency as turbulent drag reducers, viscosifiers, and flocculants.<sup>1–5</sup> Grafted polysaccharide copolymers show better performance in flocculation than ungrafted polysaccharides; this is due to the better approachability of the grafted acrylic chains to the colloidal particles.<sup>6,7</sup> The grafting of synthetic acrylic polymers onto polysaccharide backbones introduces versatile properties into the copolymer because of drastic changes in the original structure. Different types of polysaccharides, such as starch, guar gum, xanthan gum, hydroxypropyl guar gum, and alginic acid, are used as natural flocculants. Synthetic flocculants are divided into three categories: cationic, anionic, and nonionic. Some of the synthetic flocculants are polyacrylamide (PAM), poly(acrylic acid), poly(styrene sulfonic acid), and poly(diallyl dimethyl ammonium chloride). Different types of graft copolymers have been synthesized by the grafting of a synthetic polymer, such as PAM, onto a natural polymer [starch, guar gum, xanthan gum, amylopectin (AP), hydroxypropyl guar gum, and sodium alginate].<sup>6–10</sup> The graft copolymers that have been obtained are fairly shear-stable and have reduced biodegradability.<sup>11</sup> Among all graft copolymers, an AP-based graft copoly-

mer (AP-g-PAM) has shown the best performance.<sup>9</sup> However, the cost of AP is high, and it is not easily dissolved in water. Therefore, in this case, carboxymethylcellulose (CMC) was chosen as the backbone polysaccharide, and PAM was grafted onto it. CMC has higher shear stability than all commercially available flocculants. It is easily acquired and is very cheap.

CMC is a derivative of cellulose formed by its reaction with alkali and chloroacetic acid. The CMC structure is based on the  $\beta$ -(1 $\rightarrow$ 4)-D-glucopyranose polymer of cellulose. CMC dissolves rapidly in cold water and is mainly used for controlling viscosity without gelling. As its viscosity drops during heating, it can be used to improve the volume yield during baking by encouraging gas-bubble formation. The controlled viscosity of CMC allows its use as a thickener, phase and emulsion stabilizer (e.g., with milk casein), and suspending agent. The more hydrophobic, lower substituted CMCs are thixotropic, but the more extended, higher substituted CMCs are pseudoplastic.

This article reports the synthetic details for six different grades of a graft copolymer based on CMC and PAM through the variation of the monomer and catalyst concentrations. The flocculation characteristics of the graft copolymers were determined in 5 wt % kaolin, 10 wt % iron-ore, and 10 wt % silica suspensions. The flocculation characteristics of the best performing graft copolymer are compared with those of some commercially available flocculants.

## EXPERIMENTAL

### Materials

CMC was procured from Supertex, Ltd. (Mumbai, India). Acrylamide was procured from E. Merck

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**TABLE I**  
Sources and Chemical Natures of the Commercial Flocculants

Flocculant	Source	Chemical nature
Magnafloc LT 22	Ciba-Geigy Special Chemical Division (Bradford, England)	Cationic
Magnafloc 156	Ciba-Geigy Special Chemical Division (Bradford, England)	Anionic
Magnafloc 351	Ciba-Geigy Special Chemical Division (Bradford, England)	Nonionic
Rishfloc 440 HV	Rishabh Chemicals Pvt., Ltd. (Mumbai, India)	Anionic
Rishfloc 226 LV	Rishabh Chemicals Pvt., Ltd. (Mumbai, India)	Nonionic

(Darmstadt, Germany). Ceric ammonium nitrate (CAN) was obtained from Loba Chemie (Mumbai, India). Acetone and hydroquinone were procured from S.D. Fine Chemicals (Mumbai, India). Kaolin was procured from B.D. Pharmaceutical (Howrah, India). The iron ore was a gift from Kiriburu iron-ore mines. Silica was obtained from Jyoti Chemicals (Mumbai, India). CMC was used after purification by Soxhlet extraction for 80 h with ethyl alcohol as suggested by Whitcomb and Macoskow,<sup>12</sup> whereas all other chemicals were used as supplied by the company. The sources of the commercial flocculants along with their chemical natures are given in Table I.

### Synthesis and purification of CMC-g-PAM

The graft copolymers of CMC-g-PAM were synthesized by ceric-ion-induced solution polymerization techniques. The temperature of the reaction medium was always maintained at  $28 \pm 1^\circ\text{C}$ . As oxygen inhibited the polymerization of the vinyl monomer, the grafting was carried out in an atmosphere of  $\text{N}_2$ .

First, the desired amount of purified CMC was dissolved in distilled water in a conical flask. It was then placed in a magnetic stirrer and heated at  $60^\circ\text{C}$  for 4 h to obtain a clear solution. The solution was then cooled to room temperature. The flask was placed in a water bath to maintain the temperature always at  $28 \pm 1^\circ\text{C}$ . A slow stream of nitrogen was passed into the solution for about 10 min with constant stirring. The required amount of acrylamide was dissolved in 75 mL of distilled water and mixed with the CMC solution. Then, oxygen-free nitrogen was again purged through the mixed solution for about 25 min. At this stage, 25 cc of the required amount of the CAN solution was added to the solution containing CMC and acrylamide, and nitrogen gas was passed for 10 min more. Then, the passing of the nitrogen gas supply was stopped, and the flask was sealed. The reaction was allowed to continue for 24 h, after which it was terminated by the addition of a saturated solution of hydroquinone. At the end of the reaction, the reaction mixture was poured into a 1-L beaker, in which it was made into a homogeneous slurry with the addition of distilled water. The viscous slurry was then precipitated by the addition of an excess quantity of acetone.

The precipitated polymer was kept in acetone overnight. Scheme 1 shows the purification of the graft copolymer.<sup>13</sup>

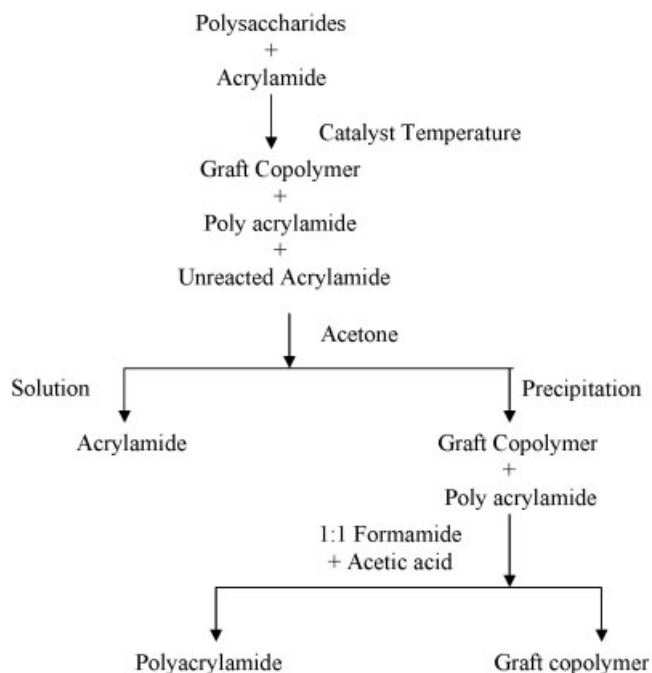
It was then dried in a vacuum oven. Subsequently, it was pulverized and sieved. Through the variation of the acrylamide and CAN concentrations, six grades of CMC-based PAM graft copolymers with variation in the number and length of the PAM chains (CMC-g-PAM 1 to CMC-g-PAM 6) were synthesized. The details of the synthesis parameters are summarized in Table II.

### Characterization

The synthesized graft copolymers were characterized with viscometry, IR spectroscopy, and X-ray diffraction (XRD).

### Intrinsic viscosity ( $[\eta]$ ) measurements

The viscosity measurements of the polymer solutions were carried out with the help of an Ubbelohde vis-



**Scheme 1** Synthesis and purification details of the graft copolymer.

TABLE II  
Synthetic Details of the Graft Copolymers

Sample	Polymer	MSU (mol)	Acrylamide (mol)	CAN (mol × 10 <sup>3</sup> )	Conversion (%) <sup>a</sup>	[η] (dL/gm)	M <sub>n</sub> × 10 <sup>6</sup>	M <sub>w</sub> × 10 <sup>6</sup>
I	CMC-g-PAM 1	0.0082	0.21	0.2006	85.53	8.65	1.58	2.64
II	CMC-g-PAM 2	0.0082	0.21	0.2508	87.26	8.23	1.46	2.48
III	CMC-g-PAM 3	0.0082	0.21	0.3009	91.31	7.46	1.26	2.19
IV	CMC-g-PAM 4	0.0082	0.25	0.2006	83.88	9.11	1.70	2.80
V	CMC-g-PAM 5	0.0082	0.28	0.2006	82.65	10.15	1.71	3.20
VI	CMC-g-PAM 6	0.0082	0.14	0.2006	90.23	7.12	1.18	1.27

MSU = monosaccharide unit.

<sup>a</sup> Conversion (%) = [(Weight of the graft copolymer – Weight of the polysaccharides)/Weight of the acrylamide monomer] × 100.

cometer (Schott, Mainz, Germany) (CS/S (centistokes/second) = 0.003867) at 27 ± 1°C at different dilutions. For the measurement of [η] of CMC and all six graft copolymers (CMC-g-PAM 1 to CMC-g-PAM 6), 0.125 g of the polymer was dissolved in distilled water with slow stirring at 60°C. The resulting solution was cooled to room temperature, and the volume was made up to 100 cc. This was the stock solution. From this stock solution, different concentrations of the polymer samples were prepared, and the viscosity of each polymer solutions was measured from the time of flow. The [η] values of all the graft copolymers and CMC were determined from the point of intersection of two extrapolated (to zero concentration) plots,<sup>14</sup> that is, the inherent viscosity ( $\eta_{inh}$ ) versus the concentration [ $\eta_{inh} = (\ln \eta_{rel})/C$ ] and the reduced viscosity ( $\eta_{red}$ ) versus the concentration ( $\eta_{red} = \eta_{sp}/C$ ), where  $\eta_{rel}$  and  $\eta_{sp}$  are the relative and specific viscosity of the polymer solution, respectively.

#### IR spectroscopy

The IR spectra of CMC, PAM, and CMC-g-PAM 5 were recorded with the help of a PerkinElmer 630 IR (Boston, MA) spectrophotometer in the solid state within the range of 4000–400 cm<sup>-1</sup>.

#### XRD

For the XRD analysis of CMC, PAM, and all the graft copolymers, a PW 1840 diffractometer and a PW 1729 X-ray generator (Philips, Eindhoven, Holland) were used, producing Cu K $\alpha$  radiation. The scattering angle (2 $\theta$ ) was varied from 10 to 45°.

#### Flocculation studies

The flocculation characteristics of CMC, all the graft copolymers, and commercially available flocculants were investigated with the column-settling and jar-test methods.

The column-settling studies were carried out in 5 wt % kaolin, 10 wt % iron-ore, and 10 wt % silica suspen-

sions with the help of a 100-mL, stoppered, graduated cylinder. This test employed a 100-mL, stoppered, graduated cylinder and stopwatch. First, the slurry sample was put in the cylinder, and then the polymer solution was added to it. The cylinder was inverted 10 times for thorough mixing. After the cylinder was set upright, the height of the interface between the water and settling solid bed was measured over time.

The jar-test studies were carried out with the help of a flocculator supplied by M.B. Flocculators (Mumbai, India). It consisted of six pedals on a bench connected to one another by gear mechanism. All the pedals were simultaneously rotated by the same motor, with the provision for speed control. The following procedure was adopted uniformly in all cases. Effluent suspensions (400 cc) were placed in 1-L jars. The jars were placed on the flocculator bench, with the stirrer blades dipped in the suspension. A stock solution of 10<sup>-4</sup> g/cc was prepared. From the stock solution, a desired concentration of the polymer solution was added to each jar under slow stirring conditions. Immediately after the addition of the polymer to all the jars, the suspensions were stirred at a constant high speed of 75 rpm for 2 min; this was followed by slow stirring at 25 rpm for 5 min. When the polymer solutions were added to the effluent suspensions, flocs were developed in the solutions. The flocs were allowed to settle down for 10 min. A clean, supernatant liquid was drawn, and its turbidity was measured with a digital nephelo turbidity meter. The turbidity was expressed in nephelo turbidity units (NTUs). A graph of the turbidity versus the polymer dosage showed the flocculation efficiency of a particular polymer in a particular suspension.

## RESULTS AND DISCUSSION

### Synthesis

Six grades of graft copolymers (CMC-g-PAM 1 to CMC-g-PAM 6) were synthesized by the grafting of acrylamide onto the CMC backbone. By the variation of the acrylamide and CAN concentrations, two types

of graft copolymers were obtained. In the first type (I–III), the amounts of CMC and acrylamide were kept constant; only the CAN concentrations were changed. In the second type (IV–VI), the amounts of CMC and CAN were kept constant, whereas the amount of acrylamide was changed. This was undertaken by a ceric-ion-induced redox polymerization technique. The initiation mechanism of ceric-ion initiation involves the formation of a chelate complex<sup>15</sup> that decomposes to generate free-radical sites on the polysaccharide backbone.<sup>16,17</sup> These active free-radical sites in the presence of acrylic monomers generate graft copolymers. Between two systems containing the same CMC and acrylamide concentrations and different CAN concentrations, that containing a lower CAN concentration initiates fewer free-radical sites on the polysaccharide backbone than the system containing a higher CAN concentration. As the acrylamide concentrations were the same, the graft copolymer produced with a lower CAN concentration contained a smaller number of longer PAM chains. With an increase in the CAN concentration, the active free-radical sites increased, and this resulted in a graft copolymer with a large number of shorter PAM chains. Therefore, in the case of the graft copolymers (CMC-g-PAM 1 to CMC-g-PAM 3), the number of PAM chains increased, but the length of the PAM chains decreased. On the other hand, with an increase in the acrylamide concentration and the CMC and CAN concentrations fixed, graft polymers with longer PAM chains were obtained. Therefore, in the case of graft copolymers CMC-g-PAM 4, CMC-g-PAM 5, and CMC-g-PAM 6, graft copolymer CMC-g-PAM 5 had longer PAM chains than the other two. This discussion shows that graft copolymer CMC-g-PAM 5, having a lower CAN concentration and a greater PAM concentration, contained longer PAM chains than the other graft copolymers.

### $[\eta]$

The  $[\eta]$  value is a measure of the hydrodynamic volume of a polymer in solution. For branched and linear polymers of approximately similar molecular weights, the branched polymer has a lower hydrodynamic volume than the linear polymer and thus has a lower  $[\eta]$  value. Furthermore, in the case of branched polymers, a polymer with longer branching has higher  $[\eta]$  values, and vice versa. This has been observed in practice. In the first series of graft copolymers (CMC-g-PAM 1 to CMC-g-PAM 3), the  $[\eta]$  values gradually decreased. The  $[\eta]$  value of CMC-g-PAM 5 was more than that of the other graft copolymers because of the presence of longer PAM chains. Although the acrylamide concentration of CMC-g-PAM 6 was lower than that of CMC-g-PAM 1, the  $[\eta]$  value of the former was lower than that of the latter one. However, the  $[\eta]$  value of CMC-g-PAM 3 was higher than that of CMC-g-PAM 6, and

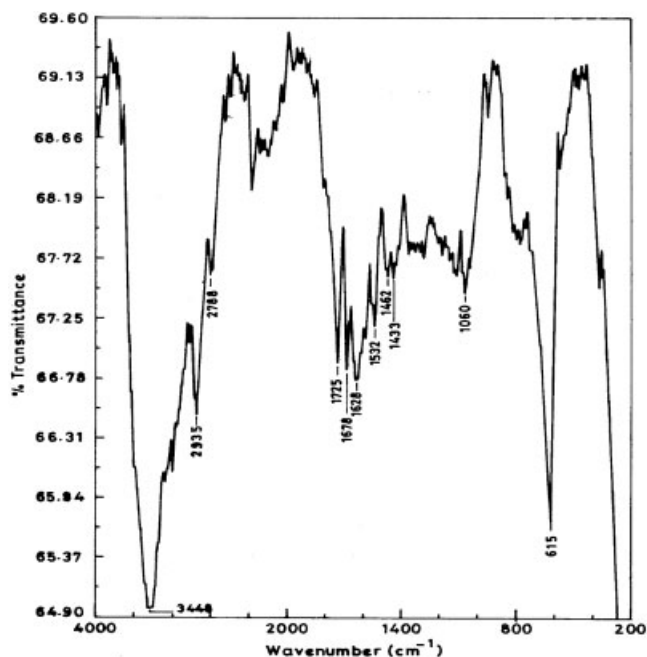


Figure 1 IR spectrum of CMC-g-PAM 5.

this indicated that CMC-g-PAM 3 contained longer PAM chains than CMC-g-PAM 6. The  $[\eta]$  values of all the graft copolymers are reported in Table II.

The molecular weights of the graft copolymers were estimated from the  $[\eta]$  value of the corresponding polymer with the Mark–Houwink equation,  $[\eta] = KM^\alpha$ , where  $M$  is the molecular weight of the polymer,  $K$  and  $\alpha$  are constants for a given polymer/solvent/temperature system. For PAM, the values of  $K$  and  $\alpha$  are given next:<sup>18</sup>

$$[\eta] = 6.8 \times 10^{-4} \times (M_n)^{0.66}$$

$$[\eta] = 6.31 \times 10^{-5} \times (M_w)^{0.80}$$

where  $M_n$  and  $M_w$  are the number-average and weight-average molecular weights, respectively.

### IR measurements

The grafting of the copolymer was confirmed by IR spectroscopy. The IR spectra of CMC and PAM are discussed elsewhere.<sup>19</sup> Figure 1 shows the IR spectrum of CMC-g-PAM 5. The broad absorption band at  $3448 \text{ cm}^{-1}$  is due to the overlap of the —OH stretching band of CMC and the —NH stretching band of PAM. The presence of a band at  $1724 \text{ cm}^{-1}$  is due to free acid groups. A band at  $1678 \text{ cm}^{-1}$  in the IR spectrum of CMC-g-PAM 5 is due to the amide I band of the amide group of PAM. The broad band at  $1628 \text{ cm}^{-1}$  is due to the overlap of  $1603 \text{ cm}^{-1}$  of CMC and the amide II band of PAM. The aforementioned bands of CMC-g-

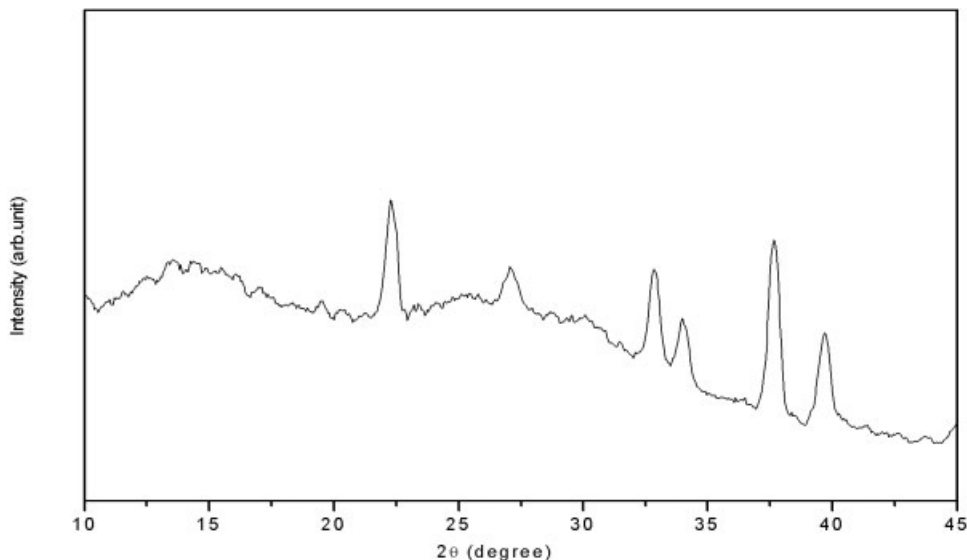


Figure 2 X-ray diffractograms of PAM.

PAM 5 are also present in CMC and PAM, but at slightly different frequencies. As PAM was initially removed from the reaction products, the presence of the aforementioned bands in the graft copolymer is strong evidence of grafting.

### XRD

X-ray diffractograms of CMC, PAM, and all the graft copolymers (CMC-g-PAM 1 to CMC-g-PAM 6) are shown in Figures 2–4. In the case of PAM, the crystalline peaks appear in the  $2\theta$  range of  $21\text{--}42^\circ$  (Fig. 2). The calculated crystallinity percentage<sup>20</sup> was 36%. XRD analysis showed that CMC exhibited very little crystallinity (Fig. 3). However, the grafted products (Fig. 4) did not have any peak indicating crystallinity.

It is concluded that grafting leads to disruption in the original crystalline structure of PAM.

### Flocculation studies

#### Settling test

The flocculation performance of the graft copolymers and some commercial flocculants was determined in kaolin, iron-ore and silica suspensions. The results are shown in Figures 5–7. In each case, the settling time was plotted against the height of the interface. Figure 5 compares the flocculation efficiency of CMC-g-PAM 1 to CMC-g-PAM 6 in kaolin suspensions. The performance of CMC-g-PAM 5 having fewer but longer PAM chains (as evidenced from the  $[\eta]$  values), was

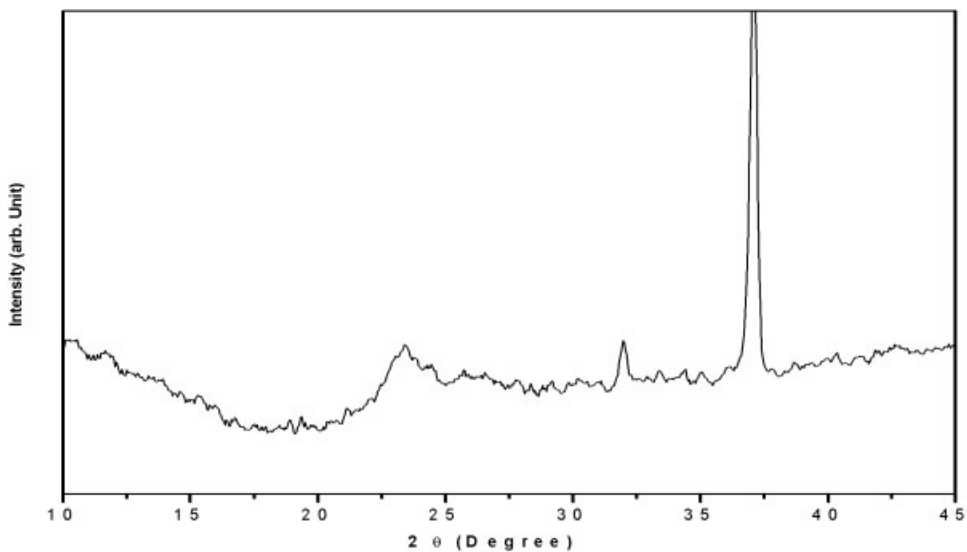


Figure 3 X-ray diffractograms of CMC.

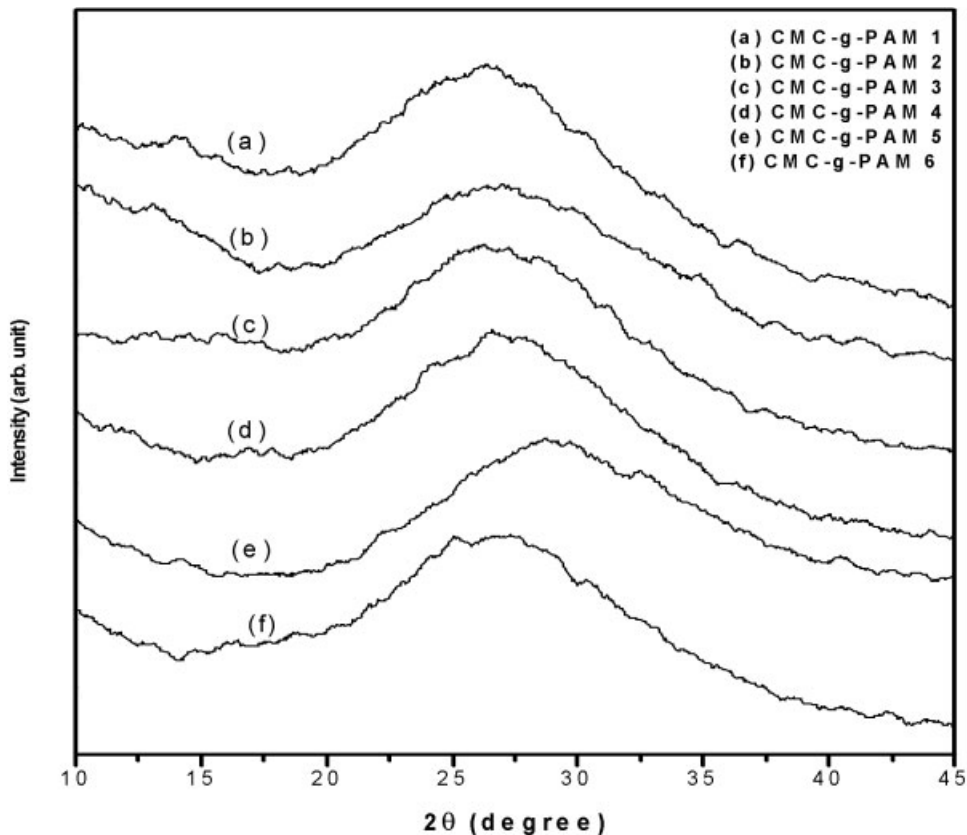


Figure 4 X-ray diffractograms of CMC-g-PAM graft copolymers.

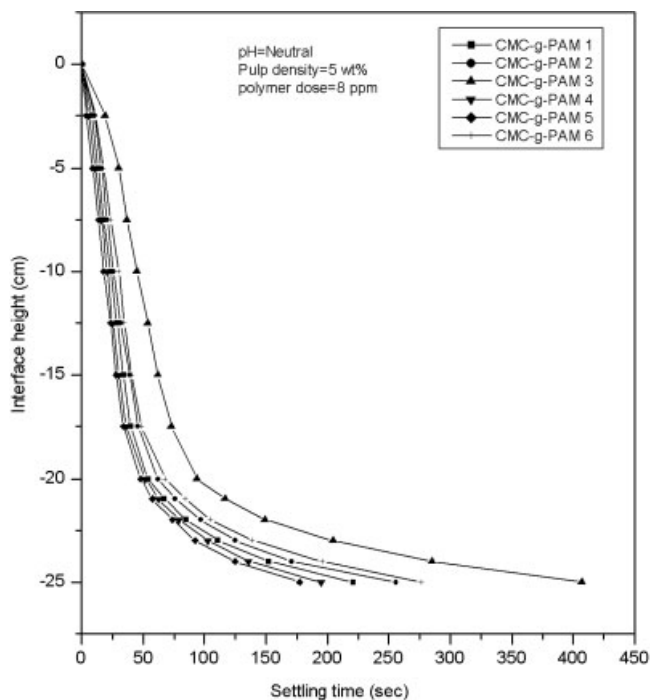


Figure 5 Settling curves for kaolin suspensions with the addition of graft copolymers.

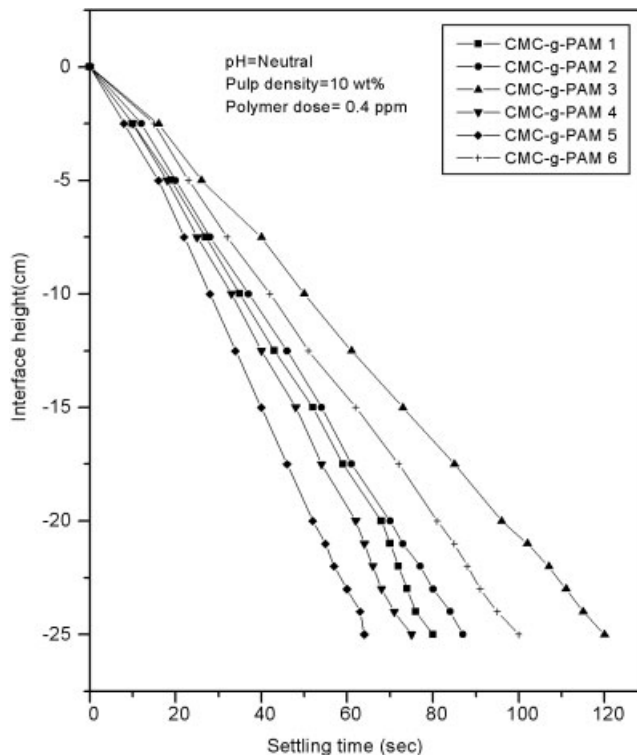
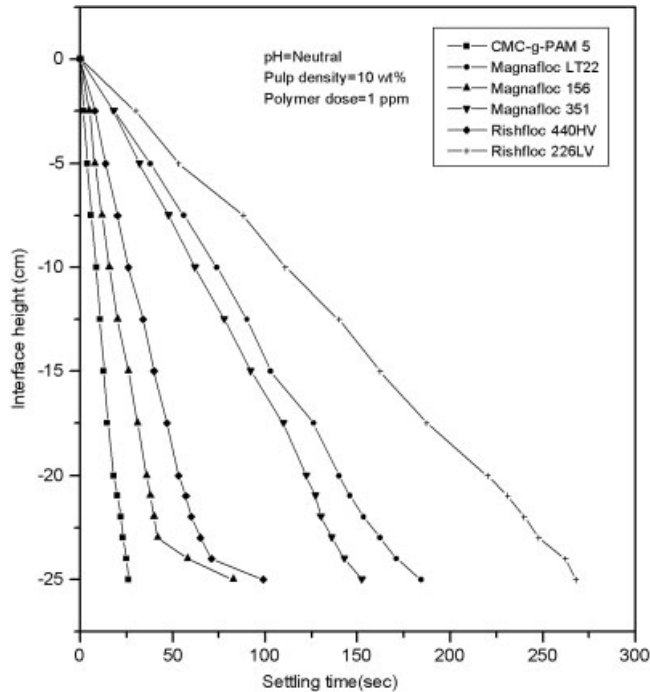


Figure 6 Settling curves for iron-ore suspensions with the addition of graft copolymers.

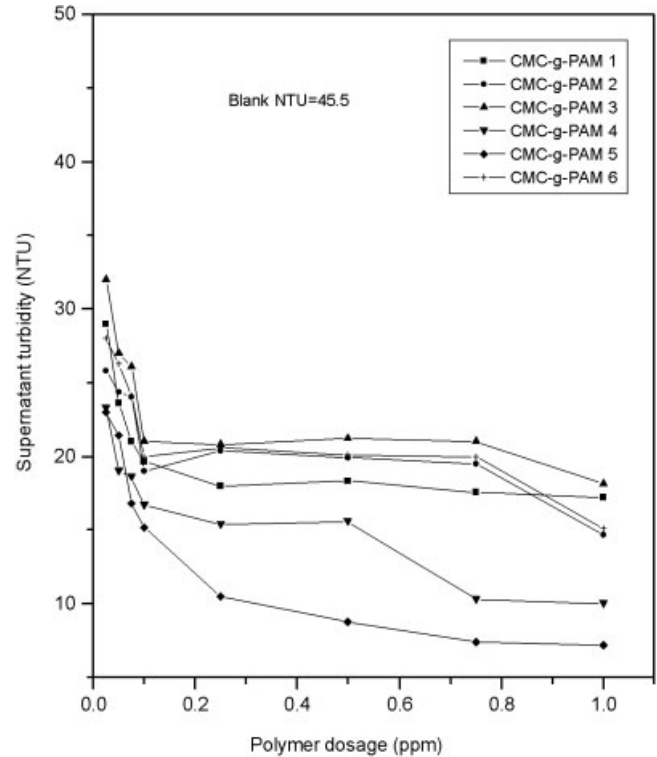


**Figure 7** Settling curves for silica suspension with addition of grafted and commercial flocculants.

better than that of the other graft copolymers. This was due to the increase in the approachability of the dangling longer PAM chains to the contaminants per Singh et al.'s model.<sup>11</sup>

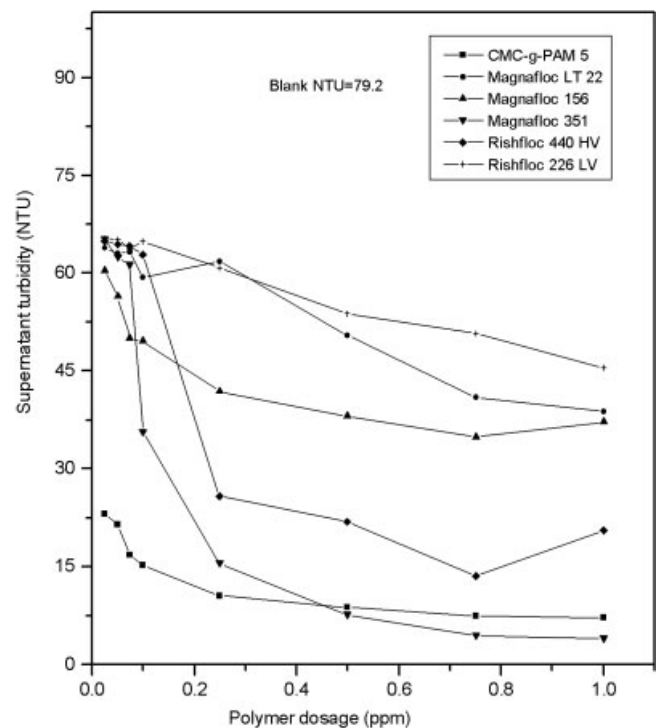
The flocculation characteristics of graft copolymers are caused by polymer bridging.<sup>21,22</sup> The controversy concerning the bridging and charge-neutralization mechanisms of aggregating aqueous suspensions by the adsorption of water-soluble polymers is long-standing. For effective bridging to occur, the length of the grafted chains should be longer to adsorb on a particle surface to another particle surface. Hence, a polymer with longer grafted chains would be more effective than one with shorter chains, as observed in practice. It was also observed that the flocculation efficiency of CMC-g-PAM 5 was much better than that of the other graft copolymers in iron-ore suspensions (Fig. 6). Similar results were also observed for silica suspensions.

Because CMC-g-PAM 5 showed a better performance than the other graft copolymers, it was chosen for comparison with commercial flocculants, namely, Magnafloc LT 22, Magnafloc 156, Magnafloc 351, Rishfloc 440 HV, and Rishfloc 226 LV, in kaolin suspensions. Figure 7 shows the settling curves of CMC-g-PAM 5 and commercial flocculants in silica suspensions. Here, the graft copolymer dominates the commercial flocculants in terms of its flocculation efficiency. In the case of linear polymers, the polymer segments attach to the surface in trains, project into the solution as tails, or form parts of loops, which link

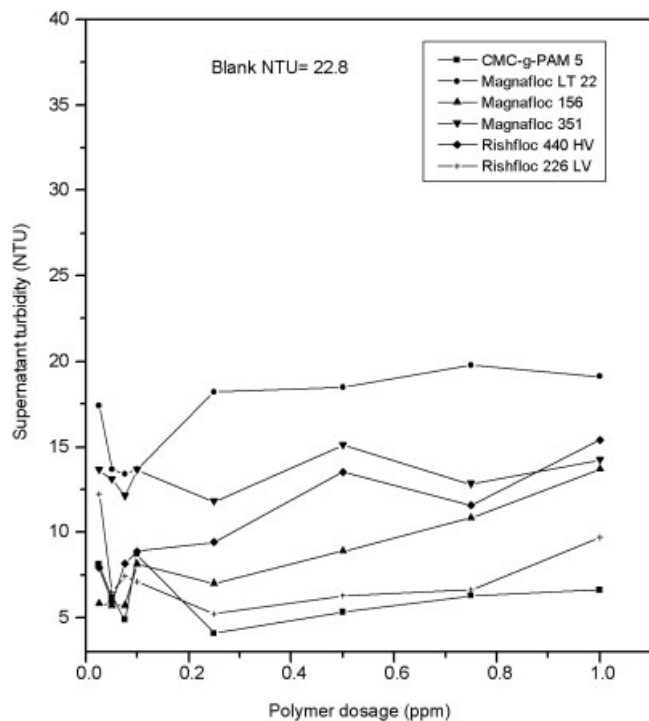


**Figure 8** Jar-test results for silica suspensions (0.25 wt %) with the addition of graft copolymers.

tails together. In this way, they can form bridges between the colloidal particles to form flocs.<sup>23</sup> In the case of graft copolymers, because of the better approach-



**Figure 9** Jar-test results for kaolin suspensions (0.25 wt %) with the addition of grafted and commercial flocculants.



**Figure 10** Jar-test results for iron-ore suspensions (0.25%) with the addition of grafted and commercial flocculants.

ability of the grafted chains; they can easily bind the colloidal particles through bridging. This type of intense bridging is not possible with linear polymers.

#### Jar test

Figure 8 shows the jar-test results of the synthesized graft copolymers in silica suspensions. CMC-g-PAM 5 showed the best performance of the synthesized graft copolymers (i.e., the steepest curve of the turbidity vs the polymer dose was obtained by its addition to the suspension). Because of the presence of the longer PAM chains in this graft copolymer, the approachability to the contaminant increased via a bridging mechanism and Singh's easy approachability model,<sup>11,22</sup> as discussed earlier. Figures 9 and 10 present the jar-test results for graft copolymer CMC-g-PAM 5 and the commercial flocculants in kaolin and iron-ore suspensions, respectively. In Figure 9, the graft copolymer dominates over the commercial flocculants, except for Magnafloc 351. At a very low dose (0.025–0.25 ppm), CMC-g-PAM 5 showed a better performance, but at a higher polymer dose, Magnafloc 351 showed a better performance. When the flocculation efficiency was compared in iron-ore suspensions, the graft copolymer showed a better performance than the commercial flocculants (Fig. 10). However, the commercial flocculants showed slightly different behaviors in the jar-test and settling-test methods. A flocculant having a lower value of turbidity in a jar test showed a higher settling

time in a column test. This can be explained on the basis of the stirring condition, as flocculation performance is greatly influenced by dosing and mixing conditions.<sup>24,25</sup> This shows that not only the nature, structure, and molecular weight of the polymeric flocculants but also the nature of the colloidal particles and their surface properties have a profound effect on the flocculation behavior.

## CONCLUSIONS

It can be concluded that among the grafted products, those having fewer and longer PAM chains showed better flocculation performance than those with shorter PAM chains. Again, synthesized graft copolymer CMC-g-PAM 5 had better flocculation characteristics than commercially available linear flocculants because of the easy approachability of the dangling grafted chains to the contaminants.

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