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Solution properties and flocculation of hydrophobically associating cationic fluorinated polyacrylamide

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Abstract The hydrophobically associating cationic fluorinated polyacrylamide (CFPAM), modified with fluorinated acrylate, 2-(perfluorooctyl)ethyl acrylate (FEA), has been newly synthesized, by free radical micellar copolymerization in aqueous solution. The solution properties of these polymers were investigated in details in this study. In addition, the flocculation of CFPAM was also evaluated. The results showed that there were strong hydrophobic associations in the CFPAM aqueous solution. The addition of NaCl resulted in an increase of solution viscosity, which indicated the good salt-resistant performance of CFPAM. The polymers also exhibited excellent property of temperature and shearing tolerance. Furthermore, the polymer showed high surface activity. At the same time, its flocculation properties were evaluated with kaolin suspensions using a standard jar test. All the results demonstrated the superiority of the cationic fluorinated polyacrylamide as a flocculant.

Keywords Cationic fluorinated polyacrylamide · Hydrophobic association · Fluorinated acrylate · Solution property · Flocculation

Introduction

Flocculation is an efficient and cost-effective method for water and wastewater treatment. The main type of polymeric flocculant is polyacrylamide and its derivatives [1–3], however, hydrophobically associating polyacrylamides (HMPAM) flocculant appeares in recent years and has been of both substantially scientific and industrial interest [4, 5]. With hydrophobic association behavior, hydrophobically associating polymer has strong bridging capacity, which is useful in the flocculation

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process. There are kinds of hydrophobically associating modified polymers and their derivatives that have been used in the treatment of different kinds of wastewater [6].

Hydrophobically associating polyacrylamides (HMPAM) are commonly prepared by free radical micellar copolymerization, in which the solubilization of the hydrophobic monomer within the surfactant micelles and the copolymerization occurring in the water continuous medium can be ensured. They not only consist of chains of predominantly hydrophilic repeat units but also incorporate a small fraction of hydrophobic sequences in the main chain or as side groups. Even with only a small fraction of hydrophobic groups, a dramatic increase in viscosity can be achieved. These results from intra and intermolecular associations of the hydrophobic groups, which forms effectively a physically cross-linked gel structure [7].

Now, a number of classes of hydrophobically associating polymer have been developed. The most used hydrophobic monomers are long alkyl hydrocarbons, such as *N*-alkylacrylamide, *n*-alkyl (meth)acrylate, vinyl alkylate, styrene, and its ramification, which have been attached to an ionic or nonionic hydrophilic backbone [8–13]. Because of the lower cohesive energy density and surface energy, the hydrophobic associations occurring between fluorocarbon groups are stronger than those of the corresponding hydrocarbon co-monomers for the same carbon chain length. Other methods are introducing hydrophobic fluorinated groups to the polymer, like hydrophobic ethoxylated urethane (F-HEUR) polymers [14–17]. However, up to now, there are seldom the studies on the fluorinated HMPAM, and moreover, they mostly focus on the nonionic fluorinated HMPAM [18–20]. As yet, reports about fluorinated ionic HAPAM prepared by free radical micellar copolymerization were scarcely seen, and at present the basic properties of these polymers and their solutions are not known to a satisfactory degree.

In this study, a novel water-soluble cationic fluorinated flocculant (CFPAM) was synthesized from acrylamide (AM), diallyl dimethyl ammonium chloride (DADMAC) and 2-(perfluorooctyl) ethyl acrylate (FEA) by free radical micellar copolymerization in aqueous solution, cetyl trimethylammonium bromide (CTAB) was utilized as the surfactant and potassium persulfate (KPS)/sodium bisulfite (SBS) was utilized as the redox initiator. The solution properties of these polymers were investigated in details, such as intrinsic viscosity, apparent viscosity, salt resistance, temperature resistance, shearing resistance, and the surface activity of CFPAM solution were studied. In addition, the flocculation of CFPAM was also evaluated.

Experimental

Materials

The structure of 2-(perfluorooctyl) ethyl acrylate (FEA) is shown in Fig. 1. FEA was obtained from Liaoning Fuxin Fluorine Chemical Company Limited and used as received. Acrylamide (AM) was recrystallized twice from chloroform. Potassium persulfate (KPS) and sodium bisulfite (SBS) were recrystallized from deionized water, respectively. AM, KPS, SBS were all purchased from Tianjin No. 3 Chemical Reagent Factory. Diallyl dimethyl ammonium chloride (DADMAC, Fig. 2) was

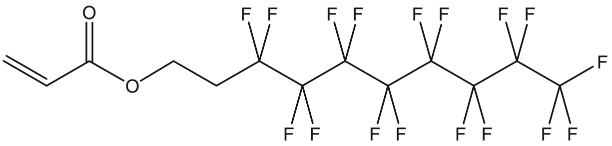


Fig. 1 Structural formula of FEA

bought from Ningbo Inno Pharmchem Company Limited and used as received. Cetyl trimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), acetone, ethanol, sodium hydroxide, hydrochloric acid, sodium chloride were all obtained from Tianjin No. 3 Chemical Reagent Factory and used as received. The water was distilled and deionized.

Preparation of FPAM

A 250 mL three-necked flask equipped with a thermometer, a mechanical stirrer, nitrogen inlet and outlet was charged with AM (20 g), DADMAC (4 g), FEA (variable), CTAB (0.6 g) and water, and then the mixture was stirred under N_2 until a homogeneous phase appeared in the solution. The total monomer concentration in water was a constant at 5 wt% (w/w) and the initiator concentration was 0.2 wt% relative to the monomer feed, in which the molar ratio of KPS to SBS was 2. After the addition of initiator, the polymerization was carried out at 40 °C for 12 h followed by dilution of mixtures with water and precipitation into excess acetone.

The fluorinated polymers (abbreviation as CFPAM-n) with different feed ratios were prepared. The feed ratios are listed in Table 1, they are all relative to the mass of the monomer acrylamide (AM).

The polymers were washed with acetone and extracted through Soxhlet extraction with ethanol for 12 h to remove all traces of water, surfactant and residual monomer before they were dried under reduced pressure at room temperature for 24 h.

Preparation of suspension

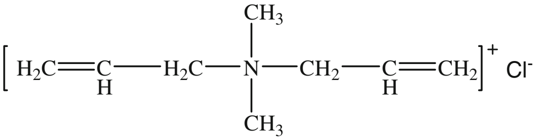
The kaolin suspension was prepared by mixing 10 g kaolin in 5 L water, which was stirred at 500 rpm for 24 h, and then was diluted in 20 times with water. The characteristics of the suspension were as followed: turbidity = 128 NTU, pH 7.5.

Measurements

The polymer solutions were prepared by dissolution of an appropriate amount of polymer in deionized water or aqueous salt solution and stirred for at least 3 days.

Intrinsic viscosities $[\eta]$ were measured by a 0.6 mm Ubbelohde capillary viscometer at 25.0 ± 0.1 °C. The kinetic energy and shear rate were found to be negligible. The density of solution was identified to be approximately the same as that of pure water; and the optimal polymer formulation was determined based on

Fig. 2 Structural formula of DADMAC



$$\left[H_2C{=}\underset{H}{C}{-}H_2C{-}\overset{CH_3}{\underset{CH_3}{N}}{-}CH_2{-}\underset{H}{C}{=}CH_2\right]^+ Cl^-$$

Table 1 Theoretical composition of the polymers

Sample	x(FEA)(wt%)[a]	Mass ratio of CTAB to FEA
CFPAM-0	0	–
CFPAM-1	0.5	6
CFPAM-2	1	3
CFPAM-3	5	0.6
CFPAM-4	10	0.3
CFPAM-5	20	0.15

[a] Means the mass ratio of FEA to the monomer acrylamide (AM)

the minimum intrinsic viscosity. All the subsequent experiments were conducted at the optimal polymer formulation.

Apparent viscosities were measured using NDJ-1 cone-plate viscometer. All viscosities were measured at 25.0 ± 0.1 °C and the shear rate was 5.12 s^{-1}, unless otherwise stated.

The surface tensions of polymer solutions were measured by a surface tensiometer (Chengde jinjian tensiometer JYW-200A) at 25.0 °C.

The flocculation test was carried out using a standard jar apparatus (Hubei Meiyu Instrument Co., Ltd., China). The suspension was put into six 1-L beakers and the flocculants were added in as 1 g/L aqueous solution. Immediately after the polymer was added, the suspension was stirred at a fast speed of 250 rpm for 2 min, followed by 100 rpm for 20 min. After the floc had settled down for 1 h, the turbidity of the supernatant liquid was measured with Digital Turbidity Meter ZD-1 (Tianjin Analytical Instrument Company, China).

Results and discussion

Intrinsic viscosity and apparent viscosity

Effect of FEA content

At low concentrations, the Huggins equation is the most common one used to determine the intrinsic viscosity and the Huggins constant, which could then be used to study the hydrodynamic volume of polymer chains and the interaction between different polymer chains or between polymer chains and solvent [21]. Intrinsic viscosity and Huggins constant were determined by measuring the viscosities of polymer solution of different concentration following Eq. 1, in which $[\eta]$ is intrinsic viscosity (mL/g), K_H is Huggins constant, the η_{sp} is the specific viscosity and η_{sp}/c is the reduced viscosity (η_{red}).

$$\eta_{sp}/c = [\eta] + k_H[\eta]^2 c \tag{1}$$

The $[\eta]$ and K_H were determined by fitting the linear portion of experimental data to the Huggins equation. Table 2 lists the intrinsic viscosity and Huggins constant obtained from Eq. 1. The relationship between intrinsic viscosity, Huggins constant, and polymer concentration can be use to evaluate dilute solution properties of the polymers. From Table 2, we can know that the introduction of hydrophobic groups would affect $[\eta]$ and K_H. With the increasing of FEA content, the intrinsic viscosities of CFPAMs both in water and in NaCl solution decreased and the Huggins constant increased. Moreover, the intrinsic viscosities in water were bigger than those in sodium chloride solution while Huggins constants in water were less than those in sodium chloride solution.

In the dilute aqueous solution, the molecular chain of PAM forms the annular or helical structure, and the chain of polymer extends; but due to the association of hydrophobic groups in polymer molecular chain, the molecular chains of the HMPAM shrink and crimp, and the hydrodynamic volume decreases. Then due to the incorporation of hydrophobic groups, CFPAMs tend to form intramolecular association and then result in the contraction of polymer chains. Furthermore, the intramolecular association is stronger with the increasing of FEA content. So the intrinsic viscosities of CFPAMs are gradually decreased. The Huggins constant reflects the interactions between the polymer molecular and water, and also between polymer molecules. According to the data in Table 2, the Huggins constant increases with the increasing of FEA content, especially the ones for samples 3–5, which reflects the hydrophobic association interactions increased with the increasing of fluorinated segment. On the other hand, the high values of the Huggins coefficient, for sample 3–5, were the indication of poor solubility of the polymers. In this research, when the mass content of FEA reached more than 20%, the copolymer was not completely dissolved in water after a long stirring time.

With the addition of sodium chloride, due to the increase of polarity of the medium and the screening effect of counterion, the intrinsic viscosities in sodium chloride solution were less, but the Huggins constants in sodium chloride solution were bigger than those in water. This was contrary to that of nonionic fluorinated polyacrylamides.

Table 2 Some parameters of samples in water and 1.0 mol/L NaCl aqueous solution

Sample	$[\eta]$ in water (mL/g)	$[\eta]$ in NaCl solution (mL/g)	K_H in water	K_H in NaCl solution
CFPAM-0	928	912	0.35	0.42
CFPAM-1	820	765	0.82	1.16
CFPAM-2	732	703	1.03	1.42
CFPAM-3	692	623	2.41	3.21
CFPAM-4	631	532	3.65	4.03
CFPAM-5	522	502	4.32	4.86

Learned from the results, there were strong hydrophobic associations in CFPAMs.

Effect of polymer concentration

Relationships between polymer concentration and apparent viscosity in water are shown in Fig. 3. From the curves, it could also be seen that the apparent viscosities of solution were increased with the increasing of polymer concentration in the large range, and the polymer solution viscosities were increased dramatically with increasing polymer concentration above a certain concentration, which was about 2.0 g/L; and the more the content of fluorinated segment was increased in polymer structure, the higher the viscosities of solutions could be observed.

For hydrophobic associating polymer, there is a critical concentration above which the polymer solution shows enhanced viscosity. This critical concentration is also known as the overlap concentration, or the critical aggregation concentration (C*). When dissolved in water above the overlap concentration, they tend to self-associate by intermolecular hydrophobic interaction, and then generates a transient "network" via associations of the hydrophobic groups ("liaisons"), and at last results in substantial viscosity increases.

Effect of surfactant additives

It is well-known that surfactant concentration greatly affects viscosity of associating polymer systems, which have been interpreted as micellar bridging and surfactant-mediated polymer dissociation as the surfactant concentration increases. The effects of adding surfactants CTAB and SDS to aqueous solutions of CFPAM-4 (after the CFPAM-4 was already synthesized) on the apparent viscosities are shown in Fig. 4, respectively. It is showed that the viscosity increased firstly to attain a maximum

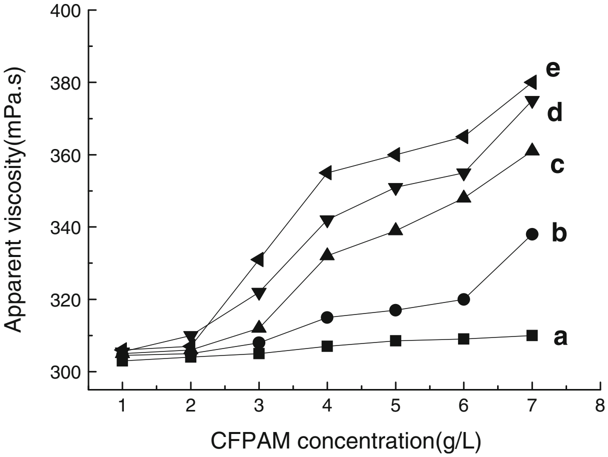


Fig. 3 Effect of polymer concentration on the apparent viscosity of CFPAMs aqueous solution (**a** CFPAM-0, **b** CFPAM-1, **c** CFPAM-2, **d** CFPAM-3, **e** CFPAM-4)

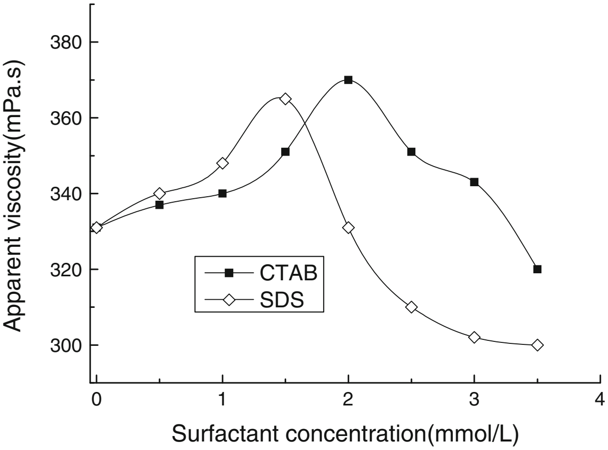


Fig. 4 Effect of surfactant content on apparent viscosity of 2.5 g/L CFPAM-4 aqueous solution

values and later decreased down to the level of the initial value with the increasing of the concentration of surfactants.

With addition of CTAB, cationic surfactant which could adsorb the hydrophobic moieties of polymer chains was dissolved in water and formed micelles, and the formation of micelle would link the hydrophobic groups of different polymer molecular and result in dimensional network, so the viscosities increase. However, further addition of CTAB, more than 2 mmol/L, there were much more micelles to be formed in the solution, which weakened the molecules bridging. The hydrophobic associated interaction between hydrophobic segments in molecular chain reduced, so the crosslinking were destroyed and the viscosities decreased.

Comparing with the cationic surfactant, the SDS molecules immediately combine with the cationic groups of CFPAM, which increased at the same time the hydrophobicity of polymer chains resulting more intermolecular association. At the same time, the screening effect by sodium ion weakened and the polymer chains became extendable. As a result, the viscosity of the solution increased quickly. However, the further addition of SDS would make the polymer unsolvable and thus lead to phase separation.

Salt resistance

With the addition of small-molecule electrolyte, the polarity of the medium increased and the intramolecular association was reinforced; simultaneously, the ionic groups in CFPAMs backbone were screened by the counterion induced from sodium chloride. So the apparent viscosities of CFPAMs solutions were affected by the competition of the screening effect by counterion and intermolecular association because of the increasing of polarity of the medium.

The effects of sodium chloride on the viscosities of CFPAMs solution is shown in Fig. 5. As shown in the Fig. 5, there were maximum values of the apparent viscosity

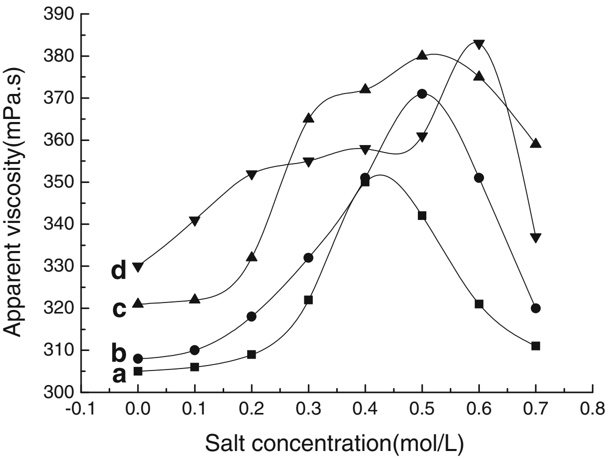


Fig. 5 Effect of ionic strength of salt solution on the apparent viscosity of 2.5 g/L CFPAMs aqueous solution (**a** CFPAM-1, **b** CFPAM-2, **c** CFPAM-3, **d** CFPAM-4)

of CFPAM with addition of sodium chloride, the viscosities of polymer solution increased with the increasing of electrolyte concentration; and with increasing FEA content, the salt concentration corresponding to maximum values of the apparent viscosity increased, so the salt resistance of CFPAMs was improved with the increasing of fluorinated groups.

The tendency of the intermolecular association and the formation of dimensional network were reinforced, which were attributed to the increasing of polarity of solvent induced by electrolytes, and their roles were more important than those of screening effect by counterion. So the apparent viscosity of the polymer increased rapidly. When the salt concentration was up to a certain extent, the phase separation appeared in polymer solution for the further contraction of hydrophobic domains. As a result, the apparent viscosities of the polymer solution decreased. The nature of the association phenomena reported above was further demonstrated by the increase in solution viscosities upon addition of NaCl. At the same time, with increasing of fluorinated groups, the tendency of the intermolecular association increased, and the salt concentration corresponding to maximum values of the apparent viscosity increased, so the salt resistance was improved.

Temperature resistance

The effect of temperature on the apparent viscosity of polymer is an important factor to be taken into account for applications. For comparison, the apparent viscosity versus temperature for CFPAM-0, -2, and -4, respectively, is shown in Fig. 6. In the process of temperature increasing, the solution did not appear phase-separation. For CFPAM-2 and -4, with the increase of temperature, the viscosities of solution decreased slowly (20–80 °C), then dropped down a little and kept

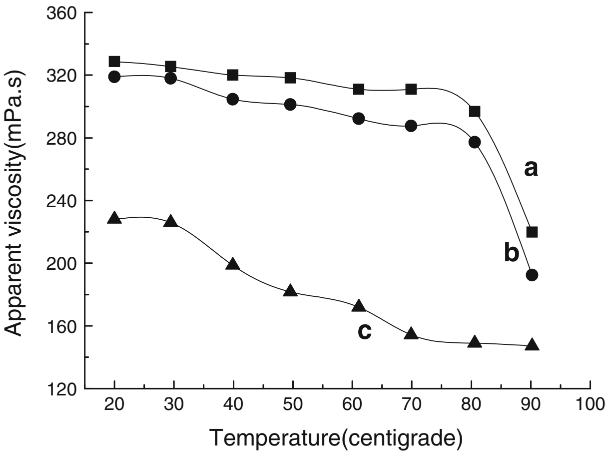


Fig. 6 Effect of temperature on the apparent viscosity of 2.5 g/L CFPAMs aqueous solution (**a** CFPAM-4, **b** CFPAM-2, **c** CFPAM-0)

unchanged, and decreased sharply after 80 °C. But for CFPAM-0, the viscosities of solution showed a sharp decrease after 40 °C.

When the temperature was less than 80 °C, the Brown motion was weak, so the apparent viscosities of aqueous solution added with CFPAM-2 and -4 were decreased slowly due to the existing of interaction of hydrophobic association of polymers. But when the temperature was more than 80 °C, the Brown motion of the molecules, which would destroy the "iceberg structure" around the hydrophobic group, was strongly intensified, so that in this time the hydrophobic association effect was weakened, and at last the viscosity was leaded to be decreased sharply. However, for aqueous solution added with CFPAM-0, there was no interaction of hydrophobic association, so the apparent viscosities were decreased at much lower temperature. All the results indicated that the fluorinated polymer exhibited excellent temperature-resistant performance than non-fluorinated polymer.

Shearing resistance

Upon application of shearing, the effects of shearing on polymer solution behavior were studied by measuring the time-dependent relationship of CFPAM-4 at different shear rates at 25 °C. The effect of shear time on the viscosity of 2.5 g/L solution of fluorinated polymer CFPAM-4 is shown in Fig. 7. As shown in Fig. 7, at low shear rate (5, 10, 20 s^{-1}), the viscosities of polymer increased firstly with time and then dropped down gradually. But at high shear rate (90 s^{-1}), the viscosities decreased quickly.

In the solution, when the shear rate was lower, the polymer molecular experienced the transition of the intramolecular association to intermolecular association, which was favor to the physical crosslinking of different chains, so the viscosity was enhanced. But following with the increasing of shear rate, disassociation was

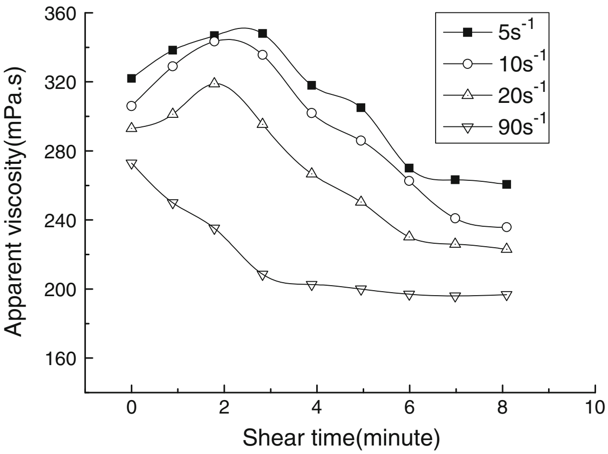


Fig. 7 Effect of sheer time on the apparent viscosity of 2.5 g/L CFPAM-4 aqueous solution

stronger, so polymer viscosities were decreased dramatically following with the increasing of shear time, which also showed highly pseudoplastic behavior; and then, the higher shear rate will break all kinds of association and orientate the molecular chains in the fluid field. So the viscosities were increased at the first stage and then were decreased, which has been described for other hydrocarbon containing associating polymers [22]. All the results showed that the CFPAM exhibited good shearing-resistant performance.

Surface activity

CFPAM has high surface activity due to its strong hydrophobic modification. The fluorinated monomer FEA has multiple pendant $-CF_3$ end groups on the perfluoroalkyl chains, which provide the polymer with extremely low surface energy and the CFPAM exhibits the excellent hydrophobic performance. So the hydrophobic effect of FEA is largely stronger than methacrylic acid fluorine straight chain esters and methacrylic acid alkyl chain esters with the same fluorine atomicity.

CFPAM-4 was dissolved in water, and the relationship between the surface tension and the CFPAM concentration is shown in Fig. 8. The results showed that the surface tension dropped drastically from about 63 to 40 mN/m when the concentration of CFPAM was increased from 0.05 g/L to 0.1 g/L, and then the surface tension was invariable with more addition of CFPAM. The fluorinated groups in the water-soluble molecule make it have high surface activity in solution. CFPAMs can be viewed as “polysurfactants” since they have locally a surfactant-like structure: a long alkyl “tail” is connected to a “polar head”. When the polymer concentration was increased, the orderly distribution of fluorinated groups on the air/water interface, and the side fluorinated groups self-associate and form micellar-like aggregates, so the surface tension decreases quickly.

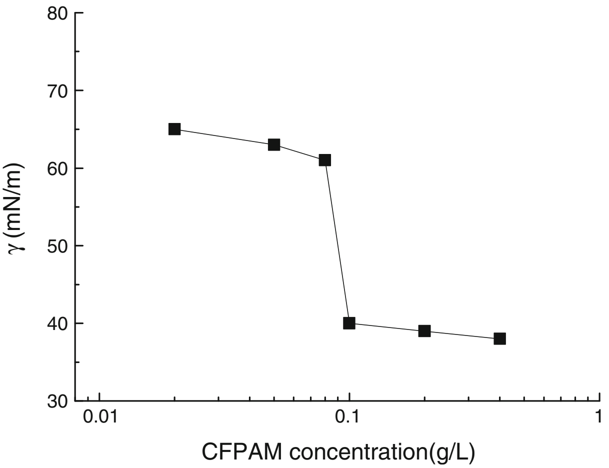


Fig. 8 Surface tension of CFPAM-4 in water as the function of polymer concentration

Flocculation experiment

A comparison of different fluorinated content in CFPAM in the flocculation performance of the kaolin suspension at pH 5 is shown in Fig. 9. With the increasing of the dosage, these were similar trends on the kaolin suspension turbidity removal. The residual turbidity decreased first, and then increased. CFPAM-4 was at a less dosage than the other two samples when the best turbidity removal was reached, and it was the most effective one in turbidity removal in the flocculation. This was attributed to the strong hydrophobic association structure, which enhanced the molecular anchor to the particle surface and promoted the

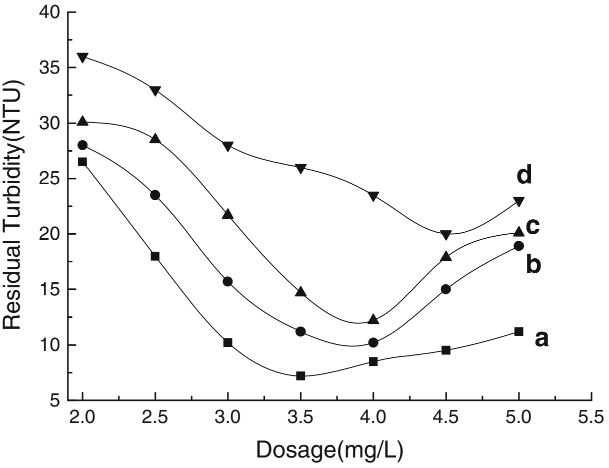


Fig. 9 Turbidity removal of kaolin suspension as a function of the concentration of CFPAM at pH 5 (**a** CFPAM-4, **b** CFPAM-3, **c** CFPAM-2, **d** CFPAM-0)

association among polymer molecules and kaolin particles. This behavior enhanced the anchor of polymer chains onto kaolin particles, and promoted the interaction between particles. As a result, the flocculation performance was enhanced. By comparison, the flocculation efficiency of no-modified CPAM (CFPAM-0) of kaolin suspension turbidity removal is shown in Fig. 9. The results demonstrated the superiority of the fluorinated cationic polyacrylamide over the no-modified cationic polyacrylamide as a flocculant.

Conclusions

The hydrophobically associating cationic fluorinated polyacrylamide (CFPAM) modified with fluorinated acrylate had been newly synthesized of acrylamide (AM), diallyl dimethyl ammonium chloride (DADMAC) and 2-(perfluorooctyl)ethyl acrylate (FEA) by free radical micellar copolymerization in aqueous solution, cetyl trimethylammonium bromide (CTAB) was utilized as the surfactant and potassium persulfate (KPS)/sodium bisulfite (SBS) was utilized as the redox initiator. The solution properties of these polymers were investigated. The solution properties of these polymers were investigated in details, such as intrinsic viscosity, apparent viscosity, salt resistance, temperature resistance, shearing resistance, and the surface activity of CFPAM solution. In addition, the flocculation of CFPAM was also evaluated. The results showed that during the incorporation of FEA, the intrinsic viscosity decreased until the modified polymer was not able to be dissolved in water, the apparent viscosity of CFPAM aqueous solution increased with the increasing of polymer concentration and there were maximum values of the apparent viscosity of CFPAM with addition of surfactant CTAB and SDS, which all showed that there were strong hydrophobic associations in the CFPAM aqueous solution. The addition of NaCl resulted in an increase of solution viscosity, which indicated the good salt-resistant performance. The polymers exhibited excellent temperature and shearing tolerance property. Furthermore, the polymer showed high surface activity. In addition, its flocculation properties were evaluated with kaolin suspensions using a standard jar test. The results demonstrated the superiority of the cationic fluorinated polyacrylamide over the no-modified cationic polyacrylamide as a flocculant.

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