

# Study on Chain Deformation of Polyacrylamides in Solutions and Its Flocculation Performance During the Flocculation Process

Z. L. Gui,<sup>1</sup> J. W. Qian,<sup>1,2</sup> X. K. Li,<sup>1</sup> B. Q. Zheng,<sup>1</sup> Q. F. An<sup>2</sup>

<sup>1</sup>Department of Polymer Science and Engineering, Zhejiang University, Hangzhou 310027, China

<sup>2</sup>Key Laboratory of Macromolecule Synthesis and Functionalization, (Ministry of Education), Zhejiang University, Hangzhou 310027, China

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**ABSTRACT:** Effects of external conditions, solution concentration, solvent quality, added salt, and pH on the chain conformation dimension of two kinds of polyacrylamide (PAAm) flocculants, neutral-PAAm, and cationic-P(AAm-DMC) (DMC, 2-[(methacryloyloxy)ethyl]trimethylammonium chloride), in parent solutions and their flocculation performance for Kaolin suspensions have been investigated by Ubbelohde viscometer and spectrophotometer, respectively. It was found that a negative correlation existed between the flocculation performance of PAAm flocculants and their chain sizes

in parent solutions. This was attributed to the expanded chain deformation of PAAm flocculants during the flocculation process from the flocculant parent solution to the Kaolin suspension and was interpreted in term of the competitive interaction among the polymeric flocculant, solvent, and Kaolin particle in the chain deformation process. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 117: 2915–2922, 2010

**Key words:** polymer solution; chain dimension; chain deformation; flocculant

## INTRODUCTION

Polyacrylamide (PAAm) is one of the widely used organic flocculants for wastewater treatment.<sup>1,2</sup> The flocculation performance of PAAm flocculants primarily depends on the type of flocculants, the ionic-PAAm and nonionic-PAAm,<sup>3,4</sup> the ionic content,<sup>5</sup> the molecular weight,<sup>6,7</sup> the type of wastewater, and their suspensions concentrations,<sup>8</sup> and also depends on some operation processes such as the shear rate.<sup>9</sup> In our previous work,<sup>10,11</sup> the effect of the flocculant dose and the parent solution concentration on the flocculation performance of PAAm flocculants has also been documented.

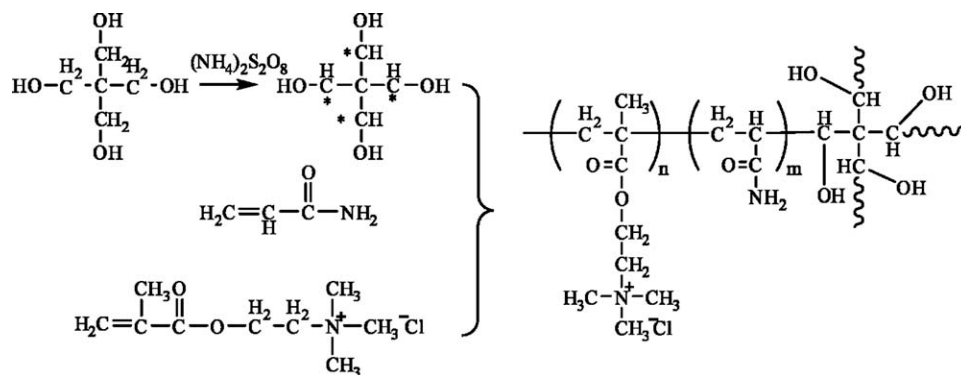
At present, charge neutralization and bridging are the two main kinds of mechanism of flocculation.<sup>12</sup> In addition to these kinds of mechanism, the sorption and interaction in a ternary system polymeric flocculant/solvent/adsorbent, could be an important issue for wastewater treatment by flocculation method. Gramain<sup>13</sup> found that the layer thickness of

polymer adsorbed on cellulose filters was proportional to the intrinsic viscosity of the polymer and further pointed out that the hydrodynamic volume of the polymer was a better parameter than its molecular weight. Sato et al.<sup>14,15</sup> reported that the adsorption amount of polymer decreased as the solvency of the polymer increased as the dimension or hydrodynamic volume of polymer molecules in solution increased with the increase of the solvency. However, the adsorption amount of polymer, dissolved in water, onto TiO<sub>2</sub> from the methanol aqueous solution does not change with the change in the solvency due to the preferential adsorption of water on TiO<sub>2</sub>.<sup>16</sup> In addition, Schwarz et al.<sup>6</sup> have reported that the conformation of adsorbed polyelectrolyte molecules is a key factor determining its efficiency as flocculant or stabilizer in colloidal dispersions.

As discussed above, we believe that the chain dimension or hydrodynamic volume and its conformation or shape of a polymer flocculant in a given system should be of importance for a better flocculation performance. We have reported the influence of incipient chain dimension of ethylene-vinyl acetate (EVA) copolymer flow improver, in parent solutions, on the rheological behavior of waxy solvents, waxy kerosene and crude oil.<sup>17–19</sup> In fact, Brostow et al.<sup>20</sup> even explained the increase of drag reduction efficiency of flowing liquids with polymer additives in

Correspondence to: J. W. Qian (qianjw@zju.edu.cn).

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**Scheme 1** Schematic diagram for synthesizing cationic-P(AAm-DMC).

term of the decrease of chain overlap produced by deformation.

However, to our knowledge, no specific study has been carried out on the effects of incipient chain size and chain shape in flocculant parent solutions on the flocculation performance of a given polymer flocculant. That is, no specific study is made on the effect of the chain deformation extent of the polymer flocculants from the parent solution to Kaolin suspension during the flocculation process on the flocculation performance.

In this study, the chain dimension and shape of two kinds of PAAm flocculants in parent solutions and in Kaolin suspensions were adjusted through changing external experimental conditions, the concentration of PAAm flocculants, solvent quality, added salt, and pH in parent solutions or in Kaolin suspensions. The flocculation performance of PAAms under corresponding experimental conditions was measured. The dependence of flocculation performance on the chain expansion extent of PAAm will be investigated and interpreted in terms of the chain deformation interaction during the flocculation process.

## EXPERIMENTAL

### Materials

The polymer flocculants used were neutral-PAAm and cationic-P(AAm-DMC), which were synthesized as reported in our previous work<sup>21</sup> using penta-

erythritol ( $C(CH_2OH)_4$ ) and ammonium persulfate ( $(NH_4)_2S_2O_8$ ) as initiator systems (shown in scheme 1). Samples of both polymer flocculants with different intrinsic viscosities were obtained by different polymerization conditions, which are given in Table I. Sodium hydroxide (NaOH), hydrochloric acid (HCl), and added salt NaCl and  $CaCl_2$  were all of analytic grade. Kaolin clay (1250 mesh) was purchased from XinYang Company, Taiyuan, China. Deionized water with a resistance of more than 18 M $\Omega$  was used in all experiments.

### Intrinsic viscosity measurement

The neutral-PAAm and the cationic-P(AAm-DMC) were dissolved in aqueous solution and 1.0 mol/L NaCl aqueous solution respectively at 45°C, and then filtered. The intrinsic viscosity ( $[\eta]$ ) measurement of PAAm sample was conducted with an Ubbelohde viscometer at  $30 \pm 0.02^\circ C$ . The flux-times were recorded with an accuracy of  $\pm 0.1$  s, without kinetic energy correction. Extrapolation from data obtained for several concentrations of solution was used to evaluate  $[\eta]$  by the Huggins equation,

$$\eta_{sp}/C = [\eta] + k_H[\eta]^2C \quad (1)$$

Where the  $\eta_{sp}$  is the specific viscosity at a given solution concentration  $C$  and  $k_H$  is the Huggins constant. The values of  $[\eta]$  of samples as well as their polymerization conditions are listed in Table I.

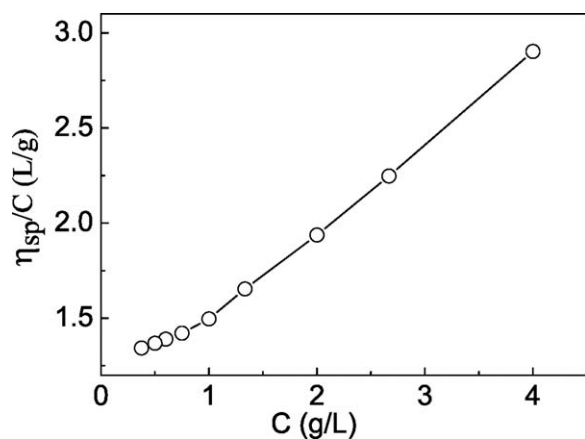
**TABLE I**  
Polymerization Conditions and  $[\eta]$  and CD of Polymer Flocculants Used

Sample	Initiator1/ Initiator2/AAm/DMC	Temperature ( $^\circ C$ )	Time (h)	$[\eta]^a$ (L/g)	CD <sup>b</sup> (wt %)
neutral -PAAm-1	0.02/0.04/100/0	60	8	1.249	–
cationic-P(AAm-DMC)-1 <sup>c</sup>	0.024/0.178/83.3/16.7	40	8	0.294	21.17
cationic-P(AAm-DMC)-2 <sup>c</sup>	0.024/0.178/46.8/9.40	20	8	0.103	20.56

<sup>a</sup> Solvents are water and water with 1.0 mol/L NaCl for PAAm-1 and the cationic-P(AAm-DMC), respectively.

<sup>b</sup> Cationic Degree: DMC weight fraction in cationic-P(AAm-DMC).

<sup>c</sup> Both  $C(CH_2OH)_4$  and  $(NH_4)_2S_2O_8$  compose an initiator system. Thus, Initiator-1/Initiator-2/AAm/DMC= $C(CH_2OH)_4/(NH_4)_2S_2O_8$ /AAm/DMC (wt/wt/wt/wt).



**Figure 1** Reduced viscosity  $\eta_{sp}/c$  of the neutral-PAAm-1 with broad concentration region in water at 30°C.

However, the other  $[\eta]$  of the PAAm at different external experimental conditions such as, added salt and pH of the solution were determined by the single point method with the equation,<sup>22</sup> where the  $\eta_r$  is the relative viscosity at a given solution concentration  $C$ .

$$[\eta] = \eta_{sp}/C\sqrt{\eta_r} \quad (2)$$

### Light transmittance measurement

The flocculant parent solution was prepared as PAAm sample dissolved completely in deionized water with or without added salt at 45°C. Kaolin clay was dispersed in deionized water and with or without added salt at room temperature. The flocculation performance of PAAm samples was conducted in a 100 mL stoppered graduated cylinder.<sup>23</sup> The Kaolin suspension was placed in the cylinder, then the flocculant parent solution with the concentration of 7 g/L was added, and the cylinder was inverted 10 times. After mixing, the cylinder was set upright for a residence time of 5 min. Then, the light transmittance ( $T$  %) of the supernatant was measured with a Spectrophotometer (Spectrophotometer-722, Shanghai Third Analytical Instruments Company, China) at room temperature. The 680 nm wavelength was selected at which the maximum absorbance displays. The error of the light transmittance ( $T$  %) was  $\pm 1\%$ .

## RESULTS AND DISCUSSION

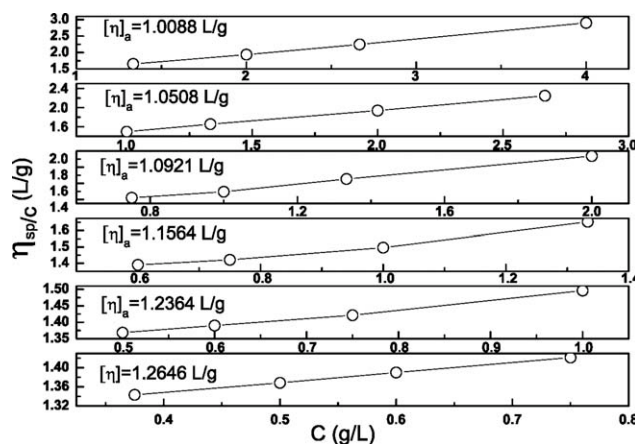
### Effect of solution concentration

Figure 1 shows the reduced viscosity  $\eta_{sp}/c$  of the neutral-PAAm-1 with broad concentration region in water at 30°C. As can be seen in Figure 1, the  $\eta_{sp}/c$  decreases with decreasing solution concentration nonlinearly and the correlation coefficient of the

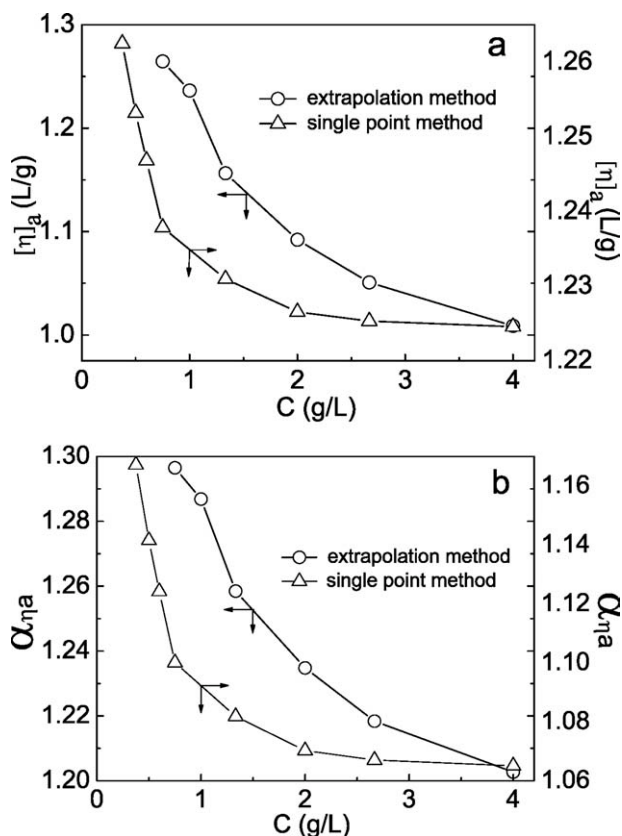
least square fitting equals to 0.9964. Figure 2 gives six reduced viscosity–concentration ( $\eta_{sp}/c$ ) curves of the neutral-PAAm-1 at different concentration region, i.e., with different incipient concentration in water at 30°C. The correlation coefficients of the least square fitting of these curves are near unity. Six  $[\eta]$  were obtained by extrapolating each curve. Obviously, these extrapolated value of  $[\eta]$  is not the real  $[\eta]$  as defined, especially for those obtained from higher concentration region, and are called here as apparent intrinsic viscosity  $[\eta]_a$ .

Figure 3(a) shows the concentration dependence of apparent intrinsic viscosity  $[\eta]_a$  of neutral-PAAm-1 in water at 30°C, by the extrapolation method and single point method according to equation (2). The concentration dependence of the viscosity expansion factor  $\alpha_\eta$  of a polymer coil is given in Figure 3(b), where the  $\alpha_\eta$  is defined as  $([\eta]/[\eta]_0)^{1/3}$ ,<sup>24</sup> the  $[\eta]_0$  is a  $[\eta]$  of neutral-PAAm-1 at theta conditions. In Figure 3(a,b), the left coordinate corresponds to values calculated by extrapolation method and the right coordinate corresponds to values calculated by single point method. The  $[\eta]_0$  here could be calculated by a convenient method<sup>25</sup> and equals to  $[\eta]_0 = 0.538$  L/g for neutral-PAAm-1. It should be noted that the  $\alpha_\eta$  should be called as apparent viscosity expansion factor  $\alpha_{\eta_a}$  because of the  $[\eta]$  was replaced by  $[\eta]_a$  in this work. As can be seen from Figure 3(b), the  $\alpha_{\eta_a}$ , either from the extrapolation method or the single point method, decreases rapidly at the beginning, and then slowly with increasing solution concentration. The decrease of  $\alpha_{\eta_a}$  is in good agreement with that reported by Graessley.<sup>26</sup>

Figure 4 shows a typical variation of the flocculation performance with the parent solution concentration ( $C$ ) of neutral-PAAm-1 at three different flocculant doses ( $C_d$ ) in 2.0 wt % Kaolin suspensions (i.e., with  $2 \times 10^{-2}$  g/mL solid contents). The light



**Figure 2** Reduced viscosity–concentration ( $\eta_{sp}/c$ ) curves of the neutral-PAAm-1 with different incipient concentration or with narrow concentration region in water at 30°C.



**Figure 3** Concentration dependence of apparent intrinsic viscosity  $[\eta]_a$  (a) and apparent viscosity expansion factor  $\alpha_{\eta a}$  (b) of neutral-PAAm-1 in water at 30°C by extrapolation method (circle) and single point method (triangle).

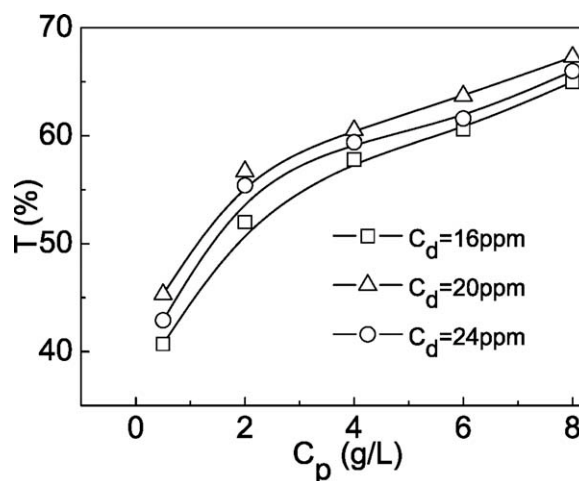
transmittance ( $T\%$ ) of the supernatant of Kaolin suspensions, which are treated by flocculant, represents the flocculation performance. It can be seen from Figure 4, the value at each  $C_d$  increases rapidly first and then slowly with increasing parent solution concentration  $C_p$ . Compared to the result in Figure 3, the changing trend of is reverse to that of  $[\eta]_a$  and  $\alpha_{\eta a}$  with increasing  $C_p$ . As we all know, high polymer concentration in solutions will result in decrease of the hydrodynamic volume of a given polymer,<sup>27</sup> and also less solvency extent or low excluded volume. In this case, the polymer chains are shrunk, compact and with less bounded solvents. Thus, when the flocculant parent solution with high polymer concentration was added to Kaolin suspensions to be diluted to a large extent, the polymer chain will expand significantly. At least two interactions exist. One is the further solvency of the polymer chain and the other is the adsorption between the polymer chain and the tiny Kaolin particles. Obviously, the latter is easy to occur due to less bounded solvents and thinner solvent layer on the polymer chains, and this is good for flocculation. Contrarily, when the flocculant parent solution with low polymer concentration was added to Kaolin suspensions,

the latter adsorption between the polymer chain and the tiny Kaolin particles is hard to occur due to the more bounded solvents and thicker solvent layer already on the polymer chains, except for the desorption of the bounded solvent molecules. This is why the light transmittance ( $T\%$ ) of the supernatant of Kaolin suspensions treated by high concentration parent solution was better than that by low concentration parent solution. The key factor here is a largely expanded chain–deformation from the compact chains in the parent solution with higher concentration to the expanded chains in the Kaolin suspension during the flocculation process. This explanation of positive chain–deformation interaction was also supported by the fact that the optimal parent concentration  $C_{op}$  existed and should be larger than  $C_{cr}$ <sup>11</sup> at which the polymer coils start to overlap each other to become compact chains.<sup>28</sup> In fact, the chain deformation interaction is somewhat similar to the “Polymer-flow Interaction”.<sup>29</sup>

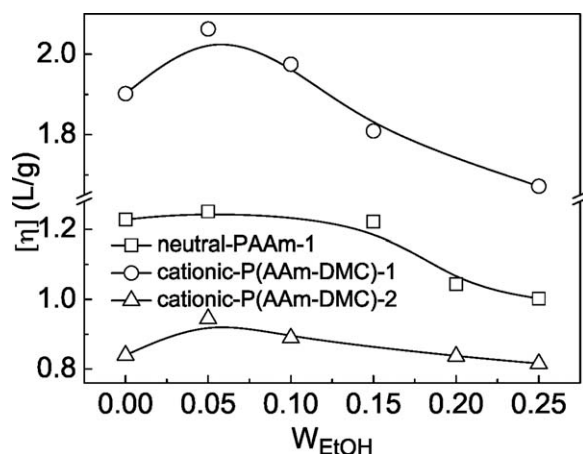
#### Effect of solvent quality

Besides the polymer concentration, solvent quality is another simple, common, and effective method to control the chain size and shape of a polymer in solutions. When the solvent quality in the parent solution is poor, the polymer chain should be compact, with small hydrodynamic volume and less bounded solvents, and then expands significantly or undergoes a large chain deformation during the flocculation process. Thus, better flocculation performance should be expected.

Figure 5 shows the variation of  $[\eta]$  of neutral-PAAm-1, cationic-P(AAm-DMC)-1, and cationic-P(AAm-DMC)-2 with ethanol weight fraction,  $W_{EtOH}$  in ethanol/water solvent mixtures. The  $[\eta]$  here was



**Figure 4** Parent solution concentration ( $C$ ) dependence of light transmittance ( $T\%$ ) of neutral-PAAm-1 in 2.0 wt % Kaolin suspensions with different doses of flocculant ( $C_d$ ).

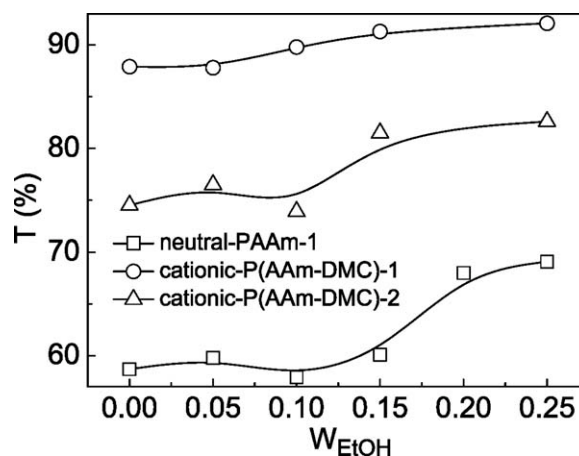


**Figure 5** Effect of ethanol weight fraction on  $[\eta]$  of neutral-PAAm-1, cationic-P(AAm-DMC)-1 and cationic-P(AAm-DMC)-2 in ethanol/water solvent mixtures at 30°C.

obtained by the extrapolation method. Ethanol is the nonsolvent or precipitator for both PAAm flocculants. The  $[\eta]$  of neutral-PAAm-1 and cationic-P(AAm-DMC) decrease with increasing  $W_{\text{EtOH}}$  in the region of  $W_{\text{EtOH}} > 0.05$ . The  $[\eta]$  in  $W_{\text{EtOH}} = 0.05$  is larger than in  $W_{\text{EtOH}} = 0$ , and this is attributed to the effect of cosolvent.<sup>30</sup> Figure 6 shows the effect of  $W_{\text{EtOH}}$  on the supernatant of 2.0 wt % Kaolin suspensions treated by three PAAms flocculants at room temperature. As can be seen in Figure 6, the flocculation performance of the three PAAms increases with decreasing  $[\eta]$  in ethanol/water solvent mixtures due to the increase of  $W_{\text{EtOH}}$ . That is, the lower  $[\eta]$  results in a higher T% value. However, it should be noted that the negative relation between the chain size in the parent solution and its flocculation performance is only for the same flocculant sample and not for different samples. It is because the flocculation performance of a polymer flocculant is also affected by other factors such as, polymer flocculant type (e.g., neutral or charged flocculant) and its molecular weight. For example, the cationic-P(AAm-DMC)-1 flocculant has much high flocculation performance, compared to that of the cationic-P(AAm-DMC)-2 flocculant, due to its high molecular weight.

#### Effect of added salt

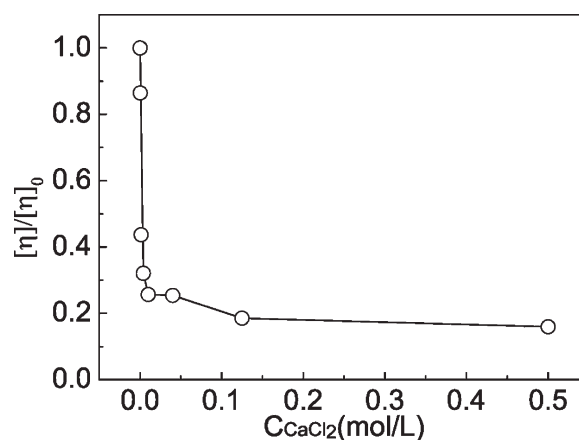
The chain size or chain shape of a polyelectrolyte in water decreases or shrinks sharply in the presence of added salt. The higher the concentration of added salt  $C_{\text{salt}}$  in water, the more shrinkage the chain size will be. This is attributed to the shield effect of charges on the polyelectrolyte chains and the depression of the repulsion between chain segments due to the presence of added salt.<sup>31</sup> Thus, a largely chain deformation would occur if a polyelectrolyte



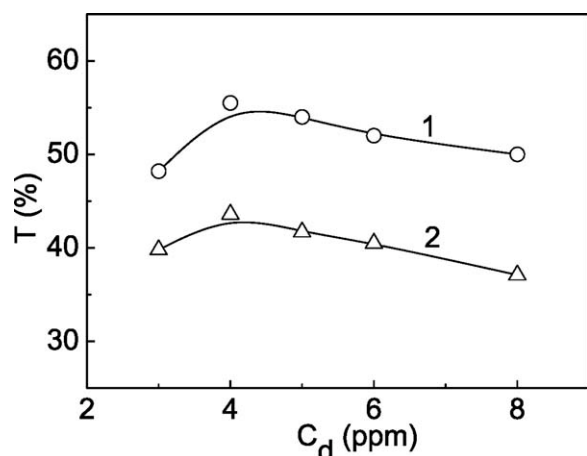
**Figure 6** Effect of ethanol weight fraction in PAAms-ethanol/water parent solutions on the flocculation performance of neutral-PAAm-1, cationic-P(AAm-DMC)-1, and cationic-P(AAm-DMC)-2 with  $C_d = 20$  ppm in 2.0 wt % Kaolin suspensions.

flocculant parent solution with added salt was used in flocculation process, and a better flocculation performance would be expected. Figure 7 shows the effect of added salt ( $\text{CaCl}_2$ ) on the  $[\eta]/[\eta]_0$  of cationic-P(AAm-DMC)-2. The  $[\eta]/[\eta]_0$  is the ratio of  $[\eta]$  of the flocculant sample in water with and without the added salt, which was obtained by the single point method  $[\eta]$  equation. As expected, the  $[\eta]$  of cationic-P(AAm-DMC)-2 depressed greatly with increasing concentration of  $\text{CaCl}_2$  in the water  $C_{\text{CaCl}_2}$ , especially at the beginning.

Figure 8 shows the variation of flocculation performance of cationic-P(AAm-DMC)-2 in 1.0 wt % Kaolin suspension with dose of flocculant at constant  $C_{\text{CaCl}_2}$  of 0.004 mol/L in two flocculation systems. One is that the added salt  $\text{CaCl}_2$  was added to the parent solution (Method-1) to get curve 1 and the other is that the added salt  $\text{CaCl}_2$  was added to the 1.0 wt % Kaolin suspension (Method-2) to get curve 2.



**Figure 7** Effect of the concentration of added salt  $\text{CaCl}_2$  on  $[\eta]/[\eta]_0$  of the cationic-P(AAm-DMC)-2 at 30°C.



**Figure 8** Variation of flocculation performance of cationic-P(AAm-DMC)-2 in 1.0 wt % Kaolin suspension with dose of flocculant  $C_d$  at constant  $C_{CaCl_2}$  of 0.004 mol/L in two flocculation systems. The added salt  $CaCl_2$  was added to the parent solutions (method-1, curve 1) and to the 1.0 wt % Kaolin suspensions (method-2, curve-2).

For both method-1 and method-2, the final  $CaCl_2$  concentrations in two kinds of Kaolin suspensions are the same, 0.004 mol/L. Table II shows experimental conditions for the method-1 in detail. According to the program in Table II, the  $CaCl_2$  concentration,  $C_{CaCl_2} = 0.004$  mol/L, in Kaolin suspensions with different  $C_d$ , is constant, and the same as in the method-2.

Apparently, the light transmittance (T%) of the supernatants in the method-1 is higher compared to that in the method-2 at any dose of the flocculant. This experimental result indicates that the flocculation performance of cationic-P(AAm-DMC)-2 rises significantly in the method-1. Combining both results from Figures 7 and 8, it is clearly seen that the chain shape of cationic-P(AAm-DMC)-2 in the method-1 changes from compact to expanded due to the addition of added salt 0.004 mol/L  $CaCl_2$  to the parent solution and the dilution in the Kaolin suspension, i.e., a largely expanded chain deformation. It is good for the interaction between the polymer chain and the Kaolin particles during the flocculation process. In the method-2, however, the chain shape of cationic-P(AAm-DMC)-2 changes from expanded to compact, a negative chain deformation, due to the addition of added salt 0.004 mol/L  $CaCl_2$  to the Kaolin suspension directly. In this case the flocculation performance becomes worse at each dose.

Figure 9 shows the effect of salt concentration in parent solutions on the flocculation performance of cationic-P(AAm-DMC)-2 at different doses of flocculant for 1.0 wt % Kaolin suspension with constant  $CaCl_2$  concentration 0.004 mol/L in the Kaolin suspensions ultimately. As can be clearly seen from Figure 9, the flocculation performances are improved

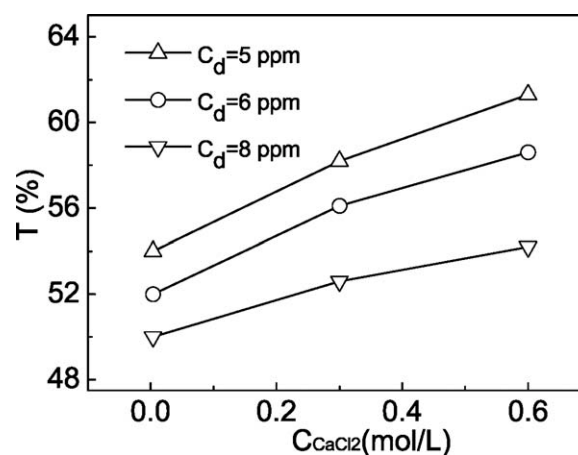
**TABLE II**  
Experimental Conditions for Adding Added Salts in Method-1 of Figure 8

$C_d$ Dose(ppm)	$C_{CaCl_2}$ in Parent solutions (mol/L)	Volume used (mL)	$C_{CaCl_2}$ in Kaolin suspensions (mol/L)
3	1.3333	0.3	0.004
4	1	0.4	0.004
5	0.8	0.5	0.004
6	0.667	0.6	0.004
8	0.5	0.8	0.004

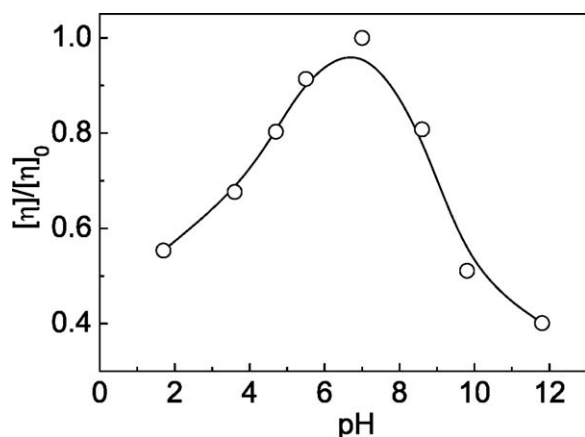
greatly with increasing  $C_{CaCl_2}$  in the parent solution at different dose, in which  $C_d = 5$  ppm is the optimal dose. The larger the  $C_{CaCl_2}$  is in the parent solution, the more shrinkage the polymer chains in the parent solution. It leads to a larger chain expansion extent or larger expanded chain deformation, and greater interaction with Kaolin particles and finally results in the better flocculation performance.

### Effect of pH

The chain size or chain shape of a polyelectrolyte in water is also changed greatly with the pH of solutions. Therefore, the flocculation performance of cationic-P(AAm-DMC)-2 would be changed with the pH of the parent solutions. Figure 10 shows the effect of the pH of solutions on the  $[\eta]/[\eta]_0$  of cationic-P(AAm-DMC)-2. Here, the  $[\eta]_0$  represents the  $[\eta]$  measured in the solvent with pH = 7.0. As can be seen from Figure 10, the curve of  $[\eta]/[\eta]_0$ -pH displays a maximum  $[\eta]$  at about pH = 7.0, i.e., the  $[\eta]$  always decreases rapidly in spite of in the region of pH < 7.0 and pH > 7.0. The HCl or NaOH, used



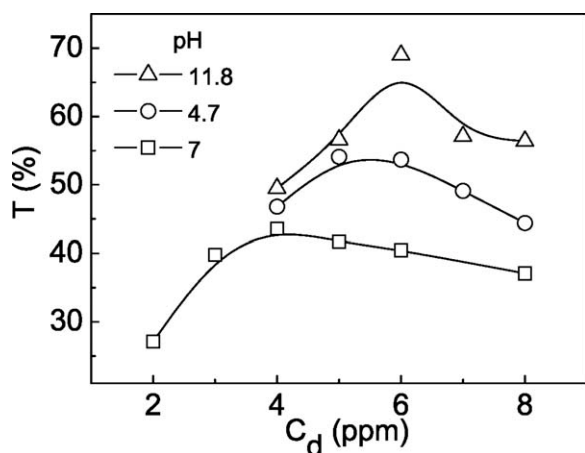
**Figure 9** The effect of the concentration of added salt,  $C_{CaCl_2}$ , in parent solutions on the flocculation performance of cationic-P(AAm-DMC)-2 at different doses of flocculant in 1.0 wt % Kaolin suspension. (Note:  $C_{CaCl_2}$  in the Kaolin suspensions ultimately is constant, 0.004 mol/L).



**Figure 10** Effect of the pH of solutions on  $[\eta]/[\eta]_0$  of cationic-P(AAm-DMC)-2 at 25°C.

for adjusting the pH of the solutions, acts as salt-like to shield the charges on the polymer chains, which depresses the  $[\eta]$  of the cationic-P(AAm-DMC)-2.

Figure 11 shows the effect of dose on the flocculation performance of cationic-P(AAm-DMC)-2 with different pH of parent solutions in 1.0 wt % Kaolin suspension at 25°C. As can be seen from Figure 11, the flocculation performance of the cationic-P(AAm-DMC)-2 flocculant from both parent solutions with acid or alkali is much better than that from the neutral parent solution. The HCl or NaOH here play the part of added salt. This is because both the effect of pH and added salt for polyelectrolytes are approximately the same in nature. Again, it is believed that the chain deformation from compact to expanded, caused by changing the pH of parent solutions, can also be effective to improve the flocculation performance of a polyelectrolyte flocculant.



**Figure 11** Variation of flocculation performance of cationic-P(AAm-DMC)-2 with  $C_d$  at different pH of parent solutions in 1.0 wt % Kaolin suspension.

## CONCLUSIONS

The relation between the chain dimension and the flocculation performance of both polymer flocculants, neutral-PAAm and cationic-P(AAm-DMC), has been investigated by changing the concentration of flocculant, solvent quality (for both flocculants), concentration of added salt, and pH (for cationic-P(AAm-DMC)) in parent solutions or in Kaolin suspensions.

A positive correlation between the flocculation performance of PAAm flocculants in Kaolin suspensions and the chain deformation extent, from compact chain to expanded chain during the flocculation process, is established. Higher concentration of PAAm, poorer solvent quality, stronger acidic or stronger alkali, and more addition of added salt in flocculant parent solutions could result in a larger chain deformation extent of the PAAms chains, and enhance their flocculation performance. It is because the chain expanded deformation of polymer flocculant during the flocculation process is good for the interaction between the flocculant and Kaolin particle.

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