

Kinetic Study of the Polymerization of Dimethyldiallylammonium Chloride and Acrylamide

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ABSTRACT: The kinetics of the polymerization of dimethyldiallylammonium chloride (DMDAAC) and acrylamide (AM) with different monomer molar ratios initiated by an ammonium persulfate–sodium bisulfate redox complex in an aqueous solution were studied. The polymerization rate (R_p) equation, the activation energy (E_a), and the reactivity ratio were measured. The results show that when the $n_{\text{DMDAAC}}:n_{\text{AM}}$ values were 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5, the copolymerization rate equation were $R_{p1} = k[M]^{2.61}[I_O]^{0.51}[I_R]^{0.52}$, $R_{p2} = k[M]^{2.70}[I_O]^{0.50}[I_R]^{0.53}$, $R_{p3} = k[M]^{2.73}[I_O]^{0.50}[I_R]^{0.56}$, $R_{p4} = k[M]^{2.77}[I_O]^{0.51}[I_R]^{0.59}$, and $R_{p5} = k[M]^{2.84}[I_O]^{0.51}[I_R]^{0.61}$ (where $[M]$ is the total monomer concentration, $[I_O]$ is the oxidant concentration, and $[I_R]$ is the reductant concentration), respectively

when the temperature was 45°C. The E_a values were $E_{a1} = 79.10$ kJ/mol, $E_{a2} = 81.39$ kJ/mol, $E_{a3} = 85.15$ kJ/mol, $E_{a4} = 88.88$ kJ/mol, and $E_{a5} = 90.61$ kJ/mol in the temperature range 35–55°C, respectively. The reactivity ratios of DMDAAC and AM were $r_{\text{DMDAAC}} = 0.14$ and $r_{\text{AM}} = 6.11$ when the temperature was 45°C. The structure of PDA was characterized by Fourier transform infrared spectroscopy and $^1\text{H-NMR}$. The results of the kinetic parameters explained the differences in the copolymerization rate and intrinsic viscosity of PDA with different cationicities. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 1636–1641, 2012

Key words: activation energy; kinetics (polym.); polyelectrolytes

INTRODUCTION

Cationic water-soluble polymers are finding increasing applications in various fields. One of the most widely applied polymers is the copolymer (PDA) of dimethyldiallylammonium chloride (DMDAAC) and acrylamide (AM),¹ which has the characteristics of a controlled positive charge and stable cation unit and molecular weight, and its distribution can be controlled by different preparation technologies,² so it can be used in petroleum exploitation, papermaking, mining, textiles and dyeing, daily chemical and water treatment,³ and so on.⁴

The method of aqueous solution polymerization to prepare PDA has the advantages of simple technologies, low cost, safe operation, no solvent recovery, and so on, and it is used widely.⁵ There have been many studies on the technology of the aqueous solution polymerization of DMDAAC and AM, but there is a lack of research on the polymerization kinetics. Tanaka⁶ and Brand et al.⁷ studied the reactivity ratios of DMDAAC and AM under different polymerization conditions separately. Liu⁸ studied the kinetics of copolymerization of DMDAAC and AM initiated by ammonium persulfate in aqueous solution. The poly-

merization rate (R_p) equation was $R_p = k[M]^{2.23}[I]^{0.70}$ (where k is the reaction rate constant, $[M]$ is the total monomer concentration, and $[I]$ is the initiator concentration) when $n_{\text{DMDAAC}}:n_{\text{AM}}$ (where n_{DMDAAC} and n_{AM} are the molar amounts of DMDAAC and AM in the initial monomer solution) was 4 : 1 and the temperature was 55°C, the activation energy (E_a) was 58.211 kJ/mol when the temperature was 45–60°C. Zhang et al.⁹ studied the kinetics of copolymerization of DMDAAC and AM initiated by an $\text{Na}_2\text{S}_2\text{O}_8$ – Na_2SO_3 redox system in an aqueous solution. The R_p equation was $R_p = k[\text{K}_2\text{S}_2\text{O}_8]^{0.55}[\text{Na}_2\text{SO}_3]^{0.56}[M]^{1.14}$ when $m_{\text{DMDAAC}}:m_{\text{AM}}$ (the mass ratio of DMDAAC to AM in the initial monomer solution) was 1 : 2, the temperature was 40°C, and E_a was 39.84 kJ/mol. These results were obtained only at a single monomer molar ratio, and the kinetics change rule with the monomer molar ratio changing were not studied; the relationship between the kinetics and the properties of the product, mainly the difference of molecular weight of PDA with different cationicities, was also not obtained.

On the basis of the preparation technologies,¹⁰ the polymerization kinetics of DMDAAC and AM with five monomer molar ratios was studied with the dilatometer method; the R_p equation, E_a , and reactivity ratio were determined. The change rule with the changing monomer molar ratio was primarily explained. The studies supplied the experimental bases of the kinetics for the reaction control of PDA production.

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EXPERIMENTAL

Materials

DMDAAC was prepared according to the literature.^{11,12} AM was purchased from Jiangsu Nantian Agricultural Technology Chemical Industry Co., Ltd. (Jiangsu, China); Analytical reagents included ammonium persulfate, sodium bisulfate, anhydrous ethanol, silver nitrate, and potassium chromate.

Preparation of the reaction solution

Exactly weighted amounts of DMDAAC and AM with a certain molar ratio were put into a four-necked flask equipped with a thermometer, a stirrer, a nitrogen inlet tube, and an outlet tube. Distilled water was added to form a homogeneous solution. Then, the reaction mixture was stirred at room temperature under a nitrogen atmosphere for 20 min. The initiator solution was added to the flask. After 10 min of stirring, the reaction solution was obtained.¹⁰

Determination of the volume shrinkage factor (K)

A certain amount of reaction solution was put into the dilatometer and installed into a thermostatic water bath. The primary volume (V_0) was read. The polymerization started, and the volume began to shrink. The volume (V_t) was read at a certain point, and the dilatometer was taken swiftly out of the water bath. Then, the reaction solution was precipitated by ethanol, filtered *in vacuo*, rinsed with ethanol, and dried *in vacuo* to a constant weight. The percentage conversion (α) was determined by gravimetry (the mass ratio of the isolated polymer to the initial monomer), and the value of K was obtained according to eq. (1).¹³ The values of K at different temperatures were determined by means of the same procedure.

$$\alpha = \frac{1}{K} \times \frac{V_t - V_0}{V_0} \quad (1)$$

Determination of R_p

The value of $d\alpha/dt$ was obtained from the slope of the plot of α versus t . R_p was calculated according to eq. (2).¹³

$$R_p = -\frac{d[M]}{dt} = [M] \frac{d\alpha}{dt} \quad (2)$$

Determination of E_a

According to the Arrhenius eq. (3)

$$k = Ae^{-E_a/RT} \quad (3)$$

E_a could be calculated according to eq. (4):

$$R_p = Ae^{-E_a/RT} [M]^x [I_O]^y [I_R]^z \quad (4)$$

With a fixed total monomer concentration, the oxidant concentration, reductant concentration, and relation of α and t at different polymerization temperatures were obtained. R_p could be calculated according to eq. (2). E_a could be calculated from the slope of the plot of $\ln R_p$ versus $1/T$ (where T is the temperature).¹³

Determination of the reactivity ratio

The copolymerization was carried out when the $n_{\text{DMDAAC}}:n_{\text{AM}}$ values were 1 : 9, 2 : 8, 3 : 7, 4 : 6, 5 : 5, 6 : 4, and 7 : 3; the total monomer concentration was 2.5 mol/L. The conversion of about 5% was controlled. The copolymer component was analyzed by the titration of chloride. The reactivity ratio was calculated according to eq. (5).¹³

$$R - \frac{R}{\rho} = \frac{R^2}{\rho} r_{\text{AM}} - r_{\text{DMDAAC}}, R = \frac{[M_1]}{[M_2]}, \rho = \frac{d[M_1]}{d[M_2]} \\ = \frac{1 - \text{CD}}{\text{CD}} \quad (5)$$

where R is the molar ratio of AM and DMDAAC in initial monomer solution; ρ is the molar ratio of the AM unit and DMDAAC unit in the copolymer; r_{AM} and r_{DMDAAC} are the reactivity ratios of AM and DMDAAC, respectively; $[M_1]$ and $[M_2]$ are the molar amounts of AM and DMDAAC, respectively, in the initial monomer solution; $d[M_1]$ and $d[M_2]$ are the molar amounts of the AM unit and DMDAAC unit, respectively, in the copolymer; and CD is the cationicity of the copolymer.

Determination of the intrinsic viscosity ($[\eta]$) of PDA

The determination of $[\eta]$ was carried out according to a literature method (1.0 mol/L NaCl as a solvent and measured with a Ubbelohde viscometer (Shanghai Liangjing Glass Instrument Plant, Shanghai, China) in a $30.0 \pm 0.1^\circ\text{C}$ water bath).¹⁴

Characterization

Fourier transform infrared (FTIR) spectroscopy of PDA was performed on an FTIR-8400S spectrophotometer (Shimadzu, Tokyo, Japan). $^1\text{H-NMR}$ spectrum of PDA was obtained on an Avance III 500-MHz spectrometer (Bruker, Rheinstetten, Germany) with D_2O as a solvent.

RESULTS AND DISCUSSION

Determination of the R_p equation of DMDAAC and AM

There was some interaction between the two monomers. Because the monomer with a higher reactivity

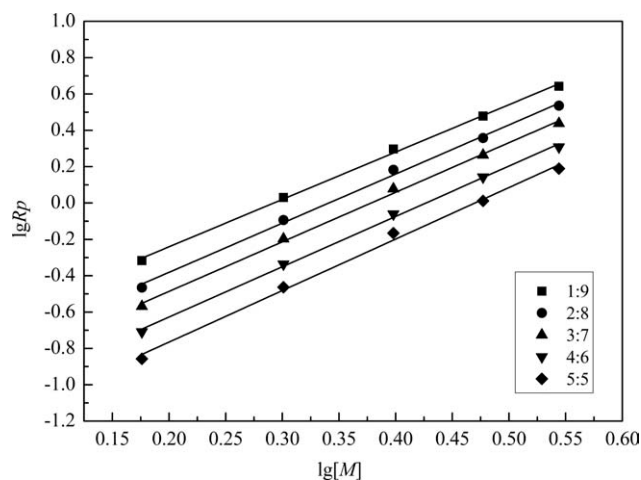


Figure 1 Plot of $\ln R_p$ versus $\ln [M]$ under different monomer molar ratios.

reacted quickly, the composition of the copolymer obtained changed with increasing polymerization time. To avoid this phenomenon, the conversion had to be less than 10%.¹⁵

Effect of the total monomer concentration on R_p

With $n_{\text{DMDAAC}}:n_{\text{AM}}$ fixed at 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5, a temperature of 45°C, an oxidant concentration ($[I_O]$) of 2.0×10^{-4} mol/L, and a reductant concentration ($[I_R]$) of 2.0×10^{-4} mol/L, the effect of the total monomer concentration on R_p was studied by the variation of the total monomer concentration in the range 1.5–3.5 mol/L. According to eq. (2), the R_p values under different monomer concentrations were determined. The results are shown in Figure 1.

Figure 1 shows the linear dependence of $\ln R_p$ on $\ln [M]$ under different monomer molar ratios. The dependence of R_p on the total monomer concentration was as follows: $R_{p1} \propto [M]^{2.61}$, $R_{p2} \propto [M]^{2.70}$, $R_{p3} \propto [M]^{2.73}$, $R_{p4} \propto [M]^{2.77}$, and $R_{p5} \propto [M]^{2.84}$, respectively. Exponent numbers greater than 1.0 have been found by many researchers over the past few decades; this result may have been due to the complicated interaction between comonomers, such as ion-pair and polarity effects.¹⁵ Furthermore, when the value of $n_{\text{DMDAAC}}:n_{\text{AM}}$ increased, the deviation increased; this indicated that DMDAAC had a greater impact. Jaeger et al.¹⁶ studied the kinetics of polymerization of DMDAAC initiated by ammonium persulfate; the overall rate equation was $R_p = k[M]^{2.9}[I]^{0.8}$, and the exponent number of R_p on monomer concentration was 2.9; this indicated that there was a big interaction between the DMDAAC monomers that affected the exponent number.

Effect of the oxidant concentration on R_p

With $n_{\text{DMDAAC}}:n_{\text{AM}}$ fixed at 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5, a temperature of 45°C, $[M]$ of 2.5 mol/L,

and $[I_R]$ of 2.0×10^{-4} mol/L, the effect of the oxidant concentration on R_p was studied by the variation of the oxidant concentration in the range 1.0×10^{-4} to 3.0×10^{-4} mol/L. According to eq. (2), the R_p values under different oxidant concentrations were determined. The results are shown in Figure 2.

Figure 2 shows the linear dependence of $\ln R_p$ on $\ln [I_O]$ under different monomer molar ratios. The dependence of R_p on the oxidant concentration was as follows: $R_{p1} \propto [I_O]^{0.51}$, $R_{p2} \propto [I_O]^{0.50}$, $R_{p3} \propto [I_O]^{0.50}$, $R_{p4} \propto [I_O]^{0.51}$, and $R_{p5} \propto [I_O]^{0.51}$, respectively. The exponent number was about 0.5; this indicated that termination occurred through the bimolecular interaction of the growing chain radicals,¹³ as also indicated by some previous works.^{8,9}

Effect of the reductant concentration on R_p

With $n_{\text{DMDAAC}}:n_{\text{AM}}$ fixed at 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5, a temperature of 45°C, $[M]$ of 2.5 mol/L, and $[I_O]$ of 2.0×10^{-4} mol/L, the effect of the reductant concentration on R_p was studied by the variation of the reductant concentration in the range 1.0×10^{-4} to 3.0×10^{-4} mol/L. According to eq. (2), the R_p values under different reductant concentrations were determined. The results are shown in Figure 3.

Figure 3 shows the linear dependence of $\ln R_p$ on $\ln [I_R]$ under different monomer molar ratios. The dependence of R_p on the reductant concentration was as follows: $R_{p1} \propto [I_R]^{0.52}$, $R_{p2} \propto [I_R]^{0.53}$, $R_{p3} \propto [I_R]^{0.56}$, $R_{p4} \propto [I_R]^{0.59}$, and $R_{p5} \propto [I_R]^{0.61}$, respectively. The exponent number was slightly higher than 0.5; this indicated that main termination occurred through the bimolecular interaction of growing chain radicals, but some others occurred through unimolecular termination. Furthermore, with increasing monomer molar ratio, the exponent number increased from 0.52 to 0.61; this indicated that the

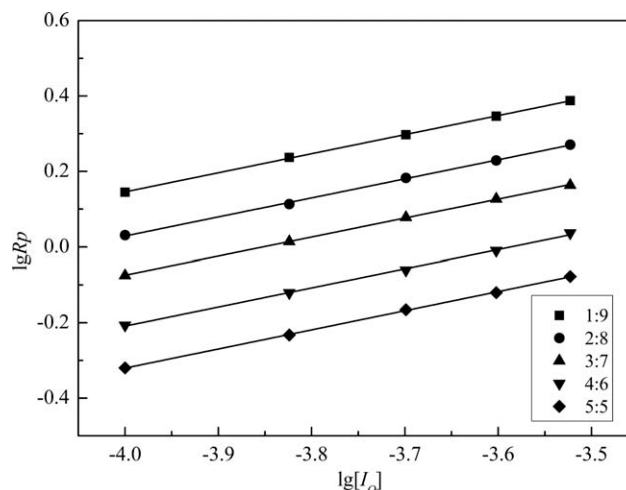


Figure 2 Plot of $\ln R_p$ versus $\ln [I_O]$ under different monomer molar ratios.

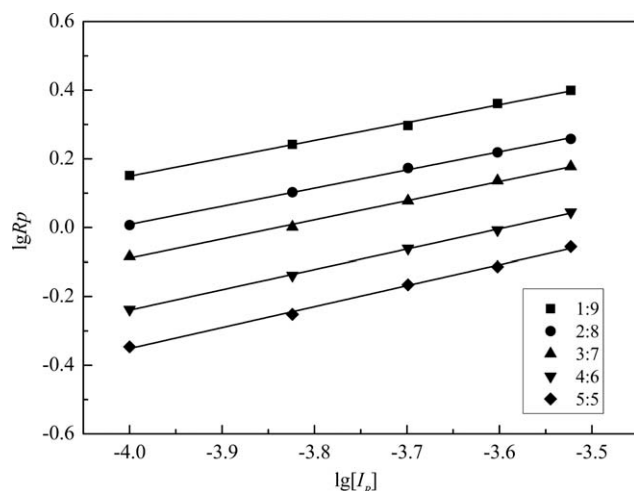


Figure 3 Plot of $\ln R_p$ versus $\ln [I_R]$ under different monomer molar ratios.

amount of comonomer occurred with increasing unimolecular termination, which resulted in a copolymer with a lower molecular weight.¹³

Determination of E_a of the polymerization of DMDAAC and AM

With $n_{\text{DMDAAC}}:n_{\text{AM}}$ fixed at 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5, $[M]$ of 2.5 mol/L, $[I_0]$ of 2.0×10^{-4} mol/L, and $[I_R]$ of 2.0×10^{-4} mol/L, the effect of the reaction temperature on R_p was studied by the variation of the reaction temperature in the range 35–40°C. According to eq. (2), the R_p values under different temperatures were determined. The results are shown in Figure 4.

The slopes of plots in Figure 4 are -9.514 , -9.789 , -10.242 , -10.690 , and -10.899 . These indicate that the E_a values were 79.10, 81.39, 85.15, 88.88, and 90.61 kJ/mol, respectively, when the $n_{\text{DMDAAC}}:n_{\text{AM}}$ values were 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5.

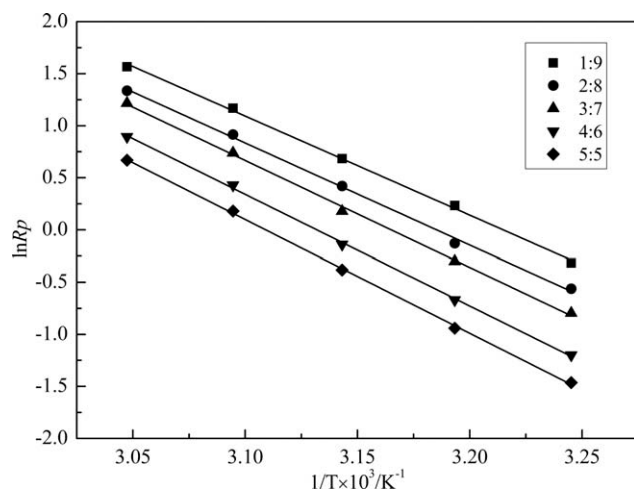


Figure 4 Plot of $\ln R_p$ versus $1/T$ under different monomer molar ratios.

E_a increased from 79.10 to 90.61 kJ/mol as the molar ratio of DMDAAC to AM increased from 1 : 9 to 5 : 5. This result was mainly because the activity of DMDAAC was much lower than that of AM. When the content of DMDAAC in the reaction system increased, the rate of chain initiation and chain propagation decreased; this resulted in the increase of E_a .

Determination of the reactivity ratios of DMDAAC and AM

The reactivity ratios of DMDAAC and AM were studied under low conversion when the $n_{\text{DMDAAC}}:n_{\text{AM}}$ values were 1 : 9, 2 : 8, 3 : 7, 4 : 6, 5 : 5, 6 : 4, and 7 : 3. The results according to eq. (5) are shown in Figure 5.

The slope of the plot in Figure 5 is 6.11, and the intercept is -0.14 ; this indicates that the reactivity ratios of AM and DMDAAC were 6.11 and 0.14, respectively.

The results indicate that the activity of DMDAAC was much lower than that of AM. This was mainly because DMDAAC had two allyl units, so it had a strong chain-transfer and self-inhibition effect; On the other hand, DMDAAC had a large volume and positive charge, so it had a large end effect and penultimate effect because of large steric and electric repulsion; this resulted in the increase of E_a and the decrease in reaction activity.¹⁵

Comparison of the optimal process conditions and $[\eta]$ of the products

The optimal process conditions were obtained under different monomer molar ratios, and the $[\eta]$ values of the product PDA with different cationicities (the ratio of n_{DMDAAC} to the total n_{DMDAAC} and n_{AM}) were determined. The results are shown in Table I.

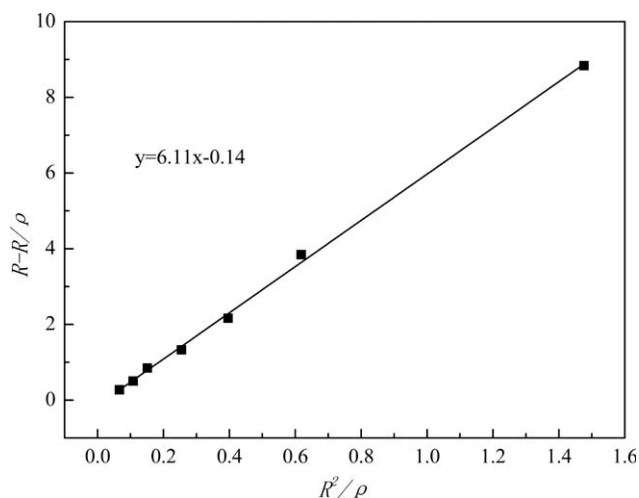


Figure 5 Reactivity ratios of AM and DMDAAC: $[M] = 2.5$ mol/L, $[I_0] = 2.0 \times 10^{-4}$ mol/L, $[I_R] = 2.0 \times 10^{-4}$ mol/L, and $T = 45^\circ\text{C}$.

TABLE I
Optimal Process Conditions and $[\eta]$ Values of the Products

No.	$n_{\text{DMDAAC}}:n_{\text{AM}}$	[M] (mol/L)	[I _o] (mol/L)	[I _r] (mol/L)	Temperature (°C)	$[\eta]$ (dL/g)
1	1 : 9	2.5	2.6×10^{-4}	2.6×10^{-4}	45	17.1
2	2 : 8	4.2	8.4×10^{-5}	8.4×10^{-5}	45	12.2
3	3 : 7	4.8	1.3×10^{-4}	1.3×10^{-4}	45	9.5
4	4 : 6	4.9	2.1×10^{-4}	2.1×10^{-4}	45	7.7
5	5 : 5	5.2	2.8×10^{-4}	2.8×10^{-4}	45	6.4

As can be seen from the results presented in Table I, $[\eta]$ of product PDA decreased with increasing monomer molar ratio; this result may have been to the lower reactivity of DMDAAC. In this work, we found that the reactivity ratios of AM and DMDAAC were 6.11 and 0.14, respectively, so with increasing content of DMDAAC, we needed to increase the total monomer concentration or initiator concentration to ensure optimal progress of the polymerization. From Table I, we can see that the total monomer concentration increased from 4.2 to 5.2 mol/L and the oxidant concentration and reductant concentration increased from 8.4×10^{-5} to 2.8×10^{-4} mol/L when monomer molar ratio was increased from 2 : 8 to 5 : 5. However, when $n_{\text{DMDAAC}}:n_{\text{AM}}$ was 1 : 9, the oxidant concentration and reductant concentration were 2.6×10^{-4} mol/L. This higher value may have occurred because the total monomer concentration (2.5 mol/L) was much lower than that (>4.2 mol/L) under other monomer molar ratios, so a high initiator concentration was needed for successful polymerization. The interaction between the comonomers was reflected directly from the kinetics; the exponent number of R_p on the total monomer concentration increased from 2.61 to 2.84, and E_a increased from 79.10

to 90.61 kJ/mol with increasing monomer molar ratio.

Characterization

Figure 6 shows the FTIR spectrum of PDA. The band at 3445 cm^{-1} was associated with the stretching of the N—H bond of amino. The bands at 2932 and 2870 cm^{-1} were associated with the stretching of the C—H bond of methylene and methyl, respectively. The band at 1655 cm^{-1} was associated with the stretching of the C=O bond of carbonyl. The bands at 1458 and 1358 cm^{-1} were associated with the bending of the C—H bond of methylene and methyl, respectively. The bands at 1126 and 617 cm^{-1} were associated with the stretching and bending of the N—C bond of heterocycle. The band at 968 cm^{-1} was the characteristic absorption peak of quaternary ammonium.

Figure 7 shows the $^1\text{H-NMR}$ spectrum of PDA. The peaks at $\delta = 1.2$ and 1.4 were the absorptions of $-\text{CH}_2-$ of DMDAAC in the main chain. The peaks at $\delta = 1.6$ and 1.7 were the absorptions of $-\text{CH}_2-$ of AM in the main chain. The peaks at $\delta = 2.1$ and 2.3 were the absorptions of $-\overset{\text{H}}{\text{C}}-$ of AM in the main chain. The peaks at $\delta = 2.6, 3.2,$ and 3.7 were the

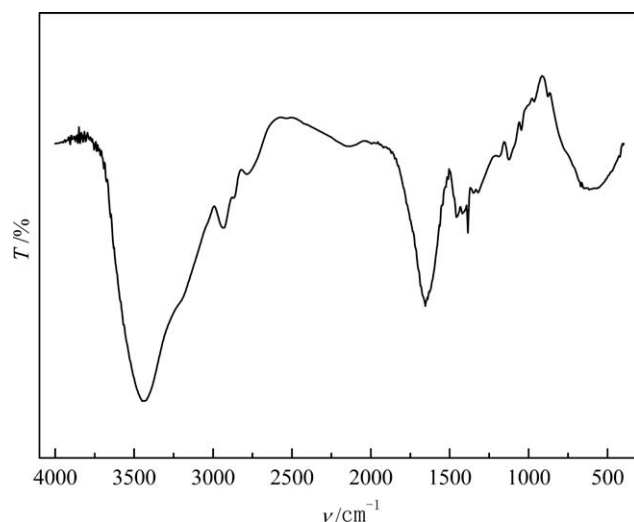


Figure 6 FTIR spectrum of PDA ($n_{\text{DMDAAC}}:n_{\text{AM}} = 3 : 7$).

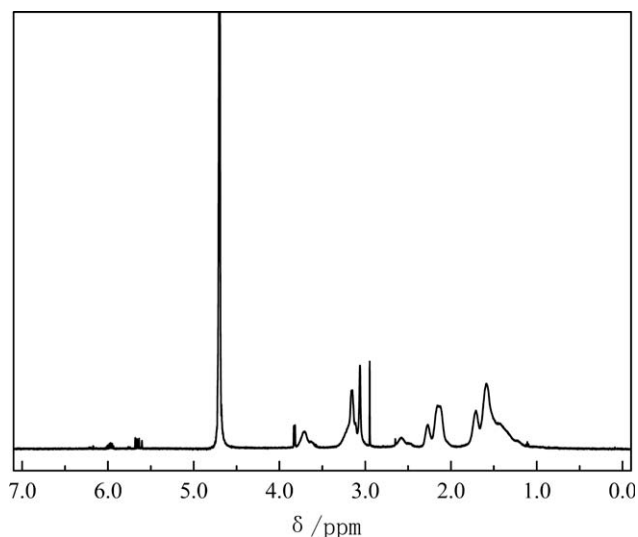


Figure 7 $^1\text{H-NMR}$ spectrum of PDA ($n_{\text{DMDAAC}}:n_{\text{AM}} = 3 : 7$).

absorptions of $\begin{array}{c} -\text{CH}- \\ | \end{array}$, $-\text{CH}_3$, and $-\text{CH}_2-$ linking with N of DMDAAC.

CONCLUSIONS

The kinetics of the polymerization of DMDAAC and AM under different monomer molar ratios initiated by an ammonium persulfate–sodium bisulfate redox complex in aqueous solution were studied. The R_p equations were $R_{p1} = k[\text{M}]^{2.61}[\text{I}_\text{O}]^{0.51}[\text{I}_\text{R}]^{0.52}$, $R_{p2} = k[\text{M}]^{2.70}[\text{I}_\text{O}]^{0.50}[\text{I}_\text{R}]^{0.53}$, $R_{p3} = k[\text{M}]^{2.73}[\text{I}_\text{O}]^{0.50}[\text{I}_\text{R}]^{0.56}$, $R_{p4} = k[\text{M}]^{2.77}[\text{I}_\text{O}]^{0.51}[\text{I}_\text{R}]^{0.59}$, and $R_{p5} = k[\text{M}]^{2.84}[\text{I}_\text{O}]^{0.51}[\text{I}_\text{R}]^{0.61}$, respectively, when the $n_{\text{DMDAAC}}:n_{\text{AM}}$ values were 1 : 9, 2 : 8, 3 : 7, 4 : 6, and 5 : 5 and the temperature was 45°C.

The E_a values were $E_{a1} = 79.10$ kJ/mol, $E_{a2} = 81.39$ kJ/mol, $E_{a3} = 85.15$ kJ/mol, $E_{a4} = 88.88$ kJ/mol, and $E_{a5} = 90.61$ kJ/mol, respectively, in the temperature range 35–55°C under the same monomer molar ratios.

The reactivity ratios of DMDAAC and AM were $r_{\text{DMDAAC}} = 0.14$ and $r_{\text{AM}} = 6.11$, respectively; these values indicated that the reactivity of DMDAAC was much lower than that of AM, so the copolymerization was nonideal, and the copolymer obtained was a random copolymer.

The results of the kinetic parameters explained the differences of the optimal preparation conditions and $[\eta]$ of the produced PDAs with different cationicities.

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