Tl(III) Initiated Polymerization of Methyl Methacrylate in Micellar Phase

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ABSTRACT: The polymerization kinetics of methyl methacrylate (MMA) was studied, using Tl(III)-cyclohexanone (CH) redox system as initiator, in the presence of emulsifier [i.e., sodium dodecyl sulfate (SDS), cetyltrimethylammonium bromide (CTAB), and thallium triacetate (TX-100)] over a temperature range of 25–45°C. The effect of various concentrations of MMA, Tl(III), cyclohexanone, H^+, and varying ionic strengths on the rate of polymerization, rate of Tl(III) consumption ($-R_{Tl}$), and the percentage of monomer conversion were examined in the presence of 0.015M SDS. The kinetic results of polymerization in the absence and presence of 0.015M SDS were compared in terms of overall activation energy (E_a) for the process. The viscosity-average molecular weight (M_V) of the polymers, obtained in the presence of varying concentrations of anionic surfactant (SDS), was also determined. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 92: 2480–2485, 2004

Key words: kinetics; methyl methacrylate; polymerization; surfactant; hydrophobic and electrostatic interaction

INTRODUCTION

It is well known that Tl(III) oxidizes various organic substrates (i.e., cyclohexanone,[1] alcohol,[2] lactic acid,[3] etc.) in aqueous solution through a free-radical intermediate. Santappa et al.[4] first studied Tl(III) as a redox initiator for acrylamide polymerization in aqueous medium. Misra et al.[5] used Tl(ClO$_4$) as initiator for polymerization of acrylonitrile and acrylamide. However, the effect of surfactant on the methyl methacrylate polymerization by using Tl(III) as initiator was not reported, although a lot of work was done with various metal ions [e.g., Ce(IV), V(V), Cr(VI), Mn(VII), etc.] for redox polymerization of vinyl monomer in the presence of surfactants.[6,7] The present piece of work mainly deals with the kinetics of methyl methacrylate polymerization by using Tl(III)–cyclohexanone redox system as initiator, in the presence of ionic surfactants, above their CMC.

EXPERIMENTAL

Materials

Methyl methacrylate (MMA; Merck) and cyclohexanone (Fluka) were distilled under reduced pressure. Sodium dodecyl sulfate (SDS) and cetyltrimethylammonium bromide (CTAB; Sisco Chemicals) were purified by standard methods. Thallium triacetate (TX-100; Sigma), sulfuric acid, and sodium bisulfate were quality chemicals and used as such without further purification.

Methods

The polymerization was carried out in a Pyrex tube of 150-mL capacity, which was sealed at the bottom, and the top was fitted with a standard joint stopper having inlet and outlet tubes for the passage of nitrogen. A microburette was fitted vertically on the stopper. The aqueous solutions of MMA, cyclohexanone, sulfuric acid, sodium bisulfate (to maintain proper ionic strength), and SDS in appropriate concentrations were taken in the reaction tube. Nitrogen gas was bubbled for about half an hour and stopcocks were closed. The solution was thermostated to attain desired temperature followed by addition of Tl(III) solution from the microburette and mixed by shaking. The volume of the reaction mixture was adjusted to 20 mL. The polymerization started immediately after the addition of Tl(III) solution. After a definite interval of time, the reaction was quenched by addition of excess standard ferrous ammonium sulfate solution. The polymer formed was filtered off through a G_4 crucible and kept at 50–60°C until constant weight was attained. The filtrate along with washings after separation of polymer was estimated for residual Tl(III) by titrating with Ce(IV) by using feroin as indicator. The rate of polymerization and rate of Tl(III) consumption were calculated from the initial slope of the curve of the percentage of monomer conversion versus time and Tl(III) consumption versus time curve, respectively.[6,7]
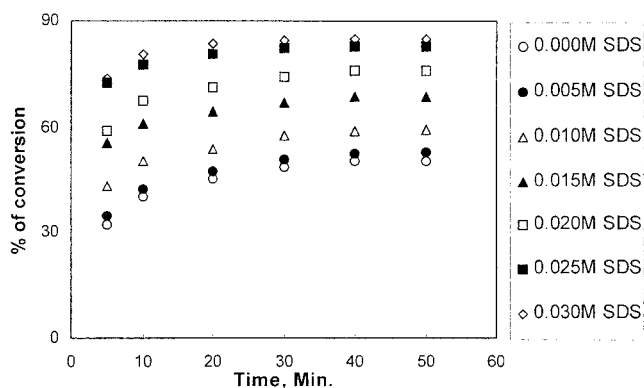
Figure 1 Percentage of methyl methacrylate conversion with time in the presence of SDS.

TABLE I
Effect of Concentration of Surfactant on the Rates

[SDS] $\times 10^3$ (mol l^{-1})	$R_P \times 10^5$ (mol l^{-1} s^{-1})		$-R_{Tl} \times 10^6$ (mol l^{-1} s^{-1})	
	SDS	CTAB	SDS	CTAB
00	5.43	—	4.76	—
05	5.47	2.33	4.82	1.98
10	7.29	1.11	5.94	1.05
15	9.33	0.84	7.04	0.71
20	11.27	0.69	7.91	0.53
25	13.86	0.69	8.57	0.52
30	14.05	0.69	8.68	0.52

[MMA] = 0.05 mol l^{-1}; [Tl(III)] = 5.08 mol l^{-1}; [CH] = 0.108 mol l^{-1}; [H$^+$] = 3.98 mol l^{-1}; [μ] = 4.2 mol l^{-1}; Temp. = 35°C.

Molecular weight determination

The viscosity-average molecular weight, M_V, was determined viscometrically. A solution of polymer (1%) in acetone was placed in a Ubbelohde suspended-level viscometer. The flow times of solution as well as the solvent were measured at 25°C. The intrinsic viscosity [η] for the solution was computed with the help of Huggins and Kramer's relationship[8] and viscosity-average molecular weight of the polymers were calculated by using the following expression[9]

$$[\eta] = 7.5 \times 10^{-4} M_v^{-0.70}$$

RESULTS AND DISCUSSION

Effect of surfactants

On enhancement of the concentration of the anionic surfactant (SDS) from 0.00 to 0.03M, an increase in the polymerization rate [$R_{P(obs)}$] as well as the percentage of monomer conversion were observed (Fig. 1). Such a trend can be attributed to the hydrophobic interaction and electrostatic interaction of micellar core and micellar surface of the anionic micelles for the monomers, cyclohexanone and Tl(III), respectively. However, in the presence of CTAB, the cationic surfactant, the rate of polymerization as well as the percentage of conversion of monomers were found to be decreased as compared to the absence of surfactant (Table I). Due to electrostatic repulsion, the approach of Tl(III) was hindered by a positively charged Stern layer of CTAB micelles toward the micellar solubilized cyclohexanone. TX-100 had no effect on the rates. A similar type of trend was also reported earlier for polymerization of acrylic and methacrylic acid,[10] acrylamide,[11–13] and vinyl benzoate[14] with different redox systems. The rate of Tl(III) consumption ($-R_{Tl}$) also increased with increasing SDS concentration, while decreasing with CTAB (Table I).

Effect of monomer concentration

The rate of polymerization, $R_{P(obs)}$, was found to be increased on increasing concentration of the MMA, in the presence of 0.015M SDS in reaction mixture. The double log plot of $R_{P(obs)}$ versus [Monomer] (Fig. 2) resulted the slope values of 1.52. This value is in good agreement with earlier reports for other redox systems.[15–17] At higher concentration of monomer, its interaction with SDS micelles is increased, which leads to the probability of their presence at reaction site (i.e., micellar core) in larger numbers, resulting in rate enhancement. The rate of Tl(III) disappearance, $-R_{Tl}$, remained constant with the change in the monomer concentration (Table II), showing that Tl(III) was not directly involved in the initiation process. This finding was very much similar to the observation of Viswanathan and Santappa[18] and Rocek and Radkowsky[19] for the Cr(VI) redox system.

Effect of Tl(III) concentration

In 0.015M SDS solution, on increasing the concentration of Tl(III) from 2.5 to 12.5 mM, the rate of polymerization as well as the percentage of the monomer
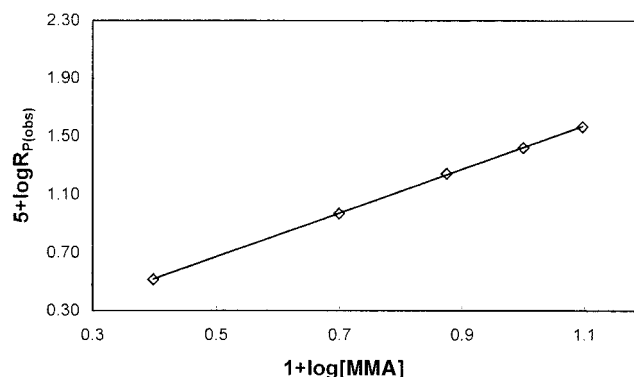

Figure 2 Bilogarithmic plot of $R_{P(obs)}$ versus [Monomer].

TABLE II
Effect of Methyl Methacrylate Concentration on the Rates

[MM] (mol l^{-1})	$R_P \times 10^5$ (mol l^{-1} s^{-1})	$-R_{Tl} \times 10^6$ (mol l^{-1} s^{-1})
0.025	3.29	6.99
0.050	9.33	7.03
0.075	17.14	7.07
0.100	26.37	7.03
0.125	36.82	7.05

[SDS] = 0.015 mol l^{-1}; [Tl(III)] = 5.08 mol l^{-1}; [CH] = 0.108 mol l^{-1}; [H$^+$] = 3.98 mol l^{-1}; [μ] = 4.2 mol l^{-1}; Temp. = 35°C.

Figure 4 Bilogarithmic plot of $R_{P(obs)}$ versus [CH] (○) and $-R_{Tl}$ versus [CH] (●).

conversion were increased. The order in Tl(III) for $R_{P(obs)}$, obtained from the plot of log $R_{P(obs)}$ versus log[Tl(III)] (Fig. 3), was found to be 0.512. Such types of square-root dependence of $R_{P(obs)}$ on other metal ions were also reported earlier.[15–17] The resulting rate enhancement may be attributed to the preference of Tl(III) to be present at the micellar surface because of the electrostatic attraction of Tl(III) for the Stern region. The rate of Tl(III) consumption indicated linear dependence on Tl(III) concentration and is in good agreement with the findings of Santappa et al.[4] for Tl(III)-initiated acrylamide polymerization.

Effect of cyclohexanone concentration

The rate of polymerization and the percentage of monomer conversion were observed to be increased with increasing concentration of cyclohexanone (0.05 to 0.25M) in the presence of 0.015M SDS in solution. The $R_{P(obs)}$ had square-root dependence on the [CH], confirmed from the value of slope (i.e., 0.514) obtained from the double-logarithmic plots of $R_{P(obs)}$ versus [cyclohexanone] (Fig. 4). The solubilization of cyclohexanone in the micellar phase was more at its higher concentration, leading to enhancement of free-radical formation as a result of which rapid polymerization took place. The Tl(III) reacted with cyclohexanone to
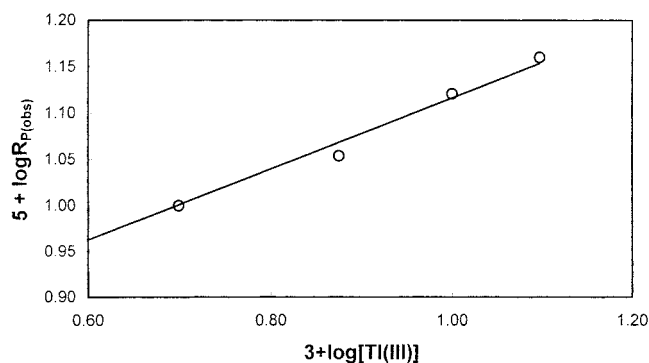
produce free radical, through intermediate complex formation.[1,4] The rate of Tl(III) consumption was linearly dependent on the [CH], which was confirmed from the value of slope of the plot of log $-R_{Tl}$ versus log[CH] (Fig. 4). The first-order rate dependence also supports the complex formation of Tl(III) with cyclohexanone.[20]

Effect of [H$^+$] and ionic strength

On increasing the [H$^+$] from 3.2 to 4.0M in the reaction medium, the rate of polymerization as well as rate of Tl(III) consumption were found to be increased (Table III) at a constant ionic strength (μ = 4.2M). The enhancement of the rates at higher acid concentration may be due to the increased activity of Tl(III). Such types of rate enhancement for Tl(III) initiated graft polymerization were also reported previously by Misra et al.[21] Both rates were observed to decrease on increasing the ionic strength (4.2 to 4.8) at constant [H$^+$] (Table III).

Effect of temperature

An enhancement of polymerization rate was observed in both the absence and the presence of 0.015M SDS in

Figure 3 Bilogarithmic plot of $R_{P(obs)}$ versus [Tl(III)].

TABLE III
Effect of [H$^+$] and Ionic Strength on the Rates

[H$^+$] (mol l^{-1})	μ (mol l^{-1})	$R_P \times 10^5$ (mol l^{-1} s^{-1})	$-R_{Tl} \times 10^6$ (mol l^{-1} s^{-1})
3.98	4.2	9.33	7.04
3.82	4.2	9.07	6.91
3.61	4.2	8.88	6.78
3.39	4.2	8.69	6.69
3.17	4.2	8.44	6.51
3.98	4.4	9.28	6.94
3.98	4.6	9.21	6.83
3.98	4.8	9.14	6.75

[SDS] = 0.015 mol l^{-1}; [MMA] = 0.05 mol l^{-1}; [Tl(III)] = 5.08 mol l^{-1}; [CH] = 0.108 mol l^{-1}; Temp. = 35°C.
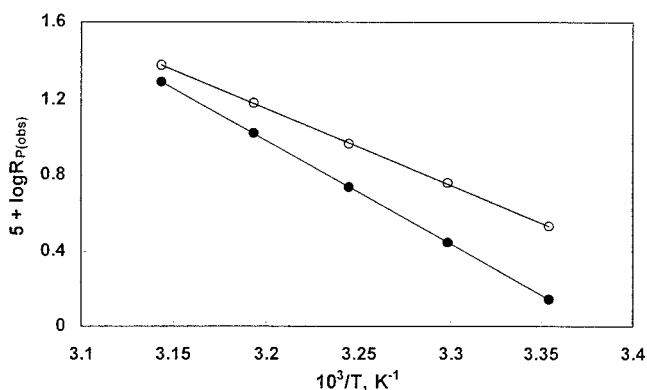
Figure 5 Arrhenius plot: $\log R_{P(\text{obs})}$ versus $1/T$ (●) $0.000M$ SDS (○) $0.015M$ SDS.

the reaction medium, on increasing the temperature from 25 to 45°C. At the higher temperature, the availability of MMA as well as cyclohexanone are more at the reaction site due to their greater solubilization in the micellar phase, causing rate acceleration. The overall activation energy for the polymerization process derived from the Arrhenius plots (Fig. 5) were found to be decreased considerably in the presence of $0.015M$ SDS (16.34 k cal mol^{-1}) in the reaction mixture as compared to that in the absence of surfactant (19.95 k cal mol^{-1}). The decrease in the activation energy indicates a positive catalytic role of anionic surfactant (SDS) micelles in the process.[6–7]

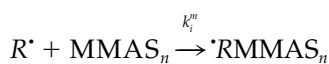### Reaction mechanism and kinetic scheme

Taking into account all the aforementioned facts, it was concluded that the polymerization process mostly occurs in the micellar phase of anionic surfactants (SDS). To explain it, the free-radical mechanistic scheme was proposed as
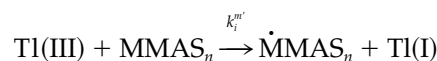
$$nS \rightleftharpoons S_n$$

$$MMA + S_n \overset{K_1}{\rightleftharpoons} MMAS_n$$

$$CH + S_n \overset{K_2}{\rightleftharpoons} CHS_n$$
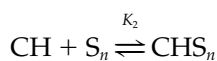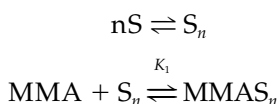
Formation of free radical

$$[\text{Tl(III)}] + CHS_n \overset{K_3}{\rightleftharpoons} Complex \overset{k^{m'}}{\longrightarrow} {}^{\bullet}R + [\text{Tl(I)}] + H^+$$

Initiation by primary radical

$$R^{\bullet} + MMAS_n \overset{k_i^m}{\longrightarrow} {}^{\bullet}RMMAS_n$$

Initiation by [Tl(III)]

$$\text{Tl(III)} + MMAS_n \overset{k_i^{m'}}{\longrightarrow} M\dot{M}AS_n + \text{Tl(I)}$$

Propagation

$$R M\dot{M}AS_n + MMAS_n \overset{k_p^m}{\longrightarrow} R\dot{M}MA_2S_n$$

$$R\dot{M}MA_2S_n + MMAS_n \overset{k_p^m}{\longrightarrow} R\dot{M}MA_3S_n$$

$$R\dot{M}MA_{(x-1)}S_n + MMAS_n \overset{k_p^m}{\longrightarrow} R\dot{M}MA_xS_n$$

Linear termination

$$R\dot{M}MA_xS_n + \text{Tl(III)} \overset{k_t^{m'}}{\longrightarrow} \text{Polymer} + \text{Tl(I)} + H^+$$

Mutual termination

$$R\dot{M}MA_xS_n + R\dot{M}MA_yS_n \overset{k_t^m}{\longrightarrow} \text{Polymer}$$
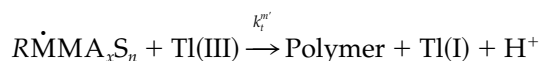
Reaction of primary radical

$$\dot{R} + [\text{Tl(III)}] \overset{k_o^m}{\longrightarrow} \text{oxidation product}$$
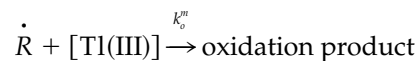
where MMA is methyl methacrylate, S is the surfactant (SDS), S_n is the micelles, K_1 and K_2 are association constants, k_i^m, k_p^m, and k_t^m are respective rate constants, and superscript m indicates the micellar phase.

Making a usual steady-state assumption for the free-radical formation, the rate expression for polymerization, R_P^m, and Tl(III) consumption, $-R_{\text{Tl}}^m$, can be derived as,

For linear termination

$$R_P^m = \frac{k_p^m[MMAS_n]^2}{k_t^{m'}}\left(\frac{k'[CHS_n]}{[MMAS_n] + \left(\frac{k_0^m}{k_i^m}\right)[\text{Tl(III)}]} + k_i^{m'}\right) \quad (1)$$

$$-R_{\text{Tl(III)}}^m = 2[\text{Tl(III)}]\{k'[CHS_n] + k_i^{m'}[MMAS_n]\} \quad (2)$$

For mutual termination

$$R_P^m = \frac{k_p^m[\text{MMAS}_n]^{3/2}[\text{Tl(III)}]^{1/2}}{(k_t^m)^{1/2}}$$

$$\times \left(\frac{k'[\text{CHS}_n]}{[\text{MMAS}_n] + \left(\dfrac{k_0^m}{k_i^m}\right)[\text{Tl(III)}]} + k_i^{m'}\right)^{1/2} \quad (3)$$

$$-R_{\text{Tl(III)}}^m = [\text{Tl(III)}]\{k'[\text{CHS}_n] + k_i^{m'}[\text{MMAS}_n]\} \quad (4)$$

It was observed that no polymerization took place in the absence of cyclohexanone. It means that Tl(III) alone could not initiate polymerization and hence the term $k_1^{m'}$ was eliminated. Because the polymerization rate was one and one-half-dependent on the monomer concentration, the linear termination is ruled out (i.e., the termination is a mutual one). Thus, the rate expression with proper substitution can be written as,

$$R_P^m = \frac{k_p^m K_1[\text{MMA}]^{3/2}[S_n]^{3/2}[\text{Tl(III)}]^{1/2}}{(k_t^m)^{1/2}}$$

$$\times \left(\frac{k'K_2[\text{CH}][S_n]}{K_1[\text{MMA}][S_n] + \left(\dfrac{k_0^m}{k_i^m}\right)[\text{Tl(III)}]}\right)^{1/2} \quad (5)$$

$$-R_{\text{Tl(III)}}^m = k'K_2[\text{Tl(III)}][\text{CH}][S_n] \quad (6)$$

where

$$k' = K_3 k^{m'} \quad (7)$$

The overall polymerization rate, $R_{P(\text{obs})}$, can be considered as the sum of the rate of polymerization in the bulk phase (R_P^b) and that in the micellar phase (P_p^m) (i.e., $R_{P(\text{obs})} = R_P^b + R_P^m$). However, at a higher concentration of SDS, above its CMC, $R_{P(\text{obs})}$ can be assumed to be equal to R_P^m for higher solubilization of monomer and cyclohexanone in the micellar phase (i.e., $R_{P(\text{obs})} \approx R_P^m$).

On squaring eq. (5) with suitable substitution and reciprocation, it can be rearranged as

$$\frac{[\text{MMA}]^2}{(R_P^m)^2} = \frac{k_t^m}{K_1^2 K_2 k_p^{m2} k'[\text{Tl(III)}][\text{MMA}]^2[\text{CH}][S_n]^3}$$

$$+ \frac{(k_0^m/k_i^m)}{k_p^{m2} k' K_1^3 K[\text{MMA}]^3[\text{CH}][S_n]^4} \quad (8)$$

Rate parameters

The k' values were determined to be 11.25×10^{-3} and 11.75×10^{-3}, respectively, from the plots of $-R_{\text{Tl}}$ versus [Tl(III)] and $-R_{\text{Tl}}$ versus [CH] (not shown), by substituting the values of K_1 and K_2 (attained previ-
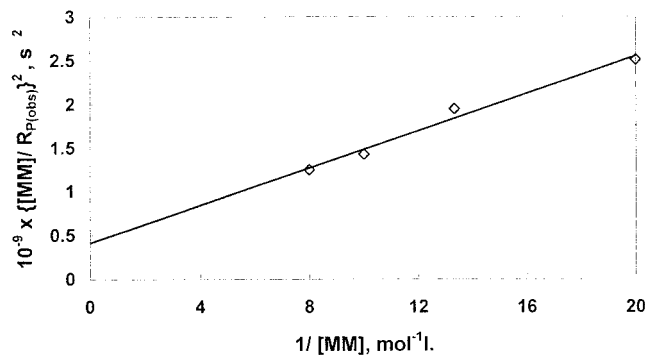

Figure 6 Plot of $\{[\text{MMA}]/R_{P(\text{obs})}\}^2$ versus $1/[\text{MMA}]$.

ously[6]). The value of S_n was calculated by using the expression, $S_n = (C_D - \text{CMC})/N$, where the aggregation number N is taken to be 62 for SDS[22] and C_D is the concentration of SDS (i.e., 0.015M). The values of k_o^m/k_i^m and $k_p^m/k_t^{m0.5}$ were calculated to be 4.76×10^{-3} and 0.466 from the plot of $[\text{MMA}]^2/R_{P(\text{obs})}$ versus $1/[\text{MMA}]$ (Fig. 6) and 4.62×10^{-3} and 0.533 from the plot of $1/[\text{Tl(III)}]$ (not shown) by using the following relationship.

From the plot $[\text{MMA}]^2/R_{P(\text{obs})}$ versus $1/[\text{MMA}]$,

$$\frac{k_o^m}{k_i^m} = \frac{K_1[S_n]\text{Slope}}{[\text{Tl(III)}]\text{Intercept}} \quad (9)$$

$$\frac{k_p^m}{k_t^{m0.5}} = \frac{1}{(\text{Intercept} \times k'K_1^3[\text{Tl(III)}][\text{CH}][S_n]^3)^{0.5}} \quad (10)$$

From the plot $[\text{MMA}]^2/R_{P(\text{obs})}$ versus $1/[\text{Tl(III)}]$,

$$\frac{k_0^m}{k_i^m} = \frac{K_1[\text{MMA}][S_n] \times \text{Intercept}}{\text{Slope}} \quad (11)$$

$$\frac{k_i^m}{k_t^{m0.5}} = \frac{(k_0^m/k_i^m)}{\text{Intercept} \times k'K_1^3 K_2[\text{MMA}][\text{CH}][S_n]^3} \quad (12)$$

Molecular weight

The viscosity-average molecular weight M_V of the polymers, obtained in the presence of SDS in the reaction mixture, was found to increase on increasing the concentration of SDS (Table IV). This can be attributed to the favorable environment, created by the SDS micelles, for lengthening the chain length. Such type of increase in molecular weight with increasing concentration of surfactant was also reported earlier by Kim[23] and Badran et al.[24]

TABLE IV
Changes in Viscosity Average Molecular Weight (M_v)
with Varying Concentration of SDS

[SDS] $\times 10^3$ (mol l^{-1})	$[\eta]$	$M_v \times 10^5$
00	5.43	4.76
05	5.47	4.82
10	7.29	5.94
15	9.33	7.04
20	11.27	7.91
25	13.86	8.57
30	14.05	8.68

CONCLUSION

The presence of anionic surfactant (SDS) in the reaction medium enhanced the rate of polymerization along with the percentage of the monomer conversion. This is due to the micellar coulombic and hydrophobic interactions for Tl(III), monomers, and cyclohexanone. The CTAB micelles reduced the rates considerably because of electrostatic repulsion of Tl(III) by positively charged Stern layer. The viscosity-average molecular weight of the polymers also increased with increasing concentration of anionic surfactant micelles.

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