Journal of Chemical Technology and Metallurgy, 58, 4, 2023, 672-681

OXIDATION OF ANILINE BY IRON (III) IN THE PRESENCE OF BIPYRIDYL IN SDS/TRITON X-100 MIXED MICELLES. APPLICABILITY OF BEREZIN PSEUDO PHASE MODEL

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Received 29 December 2022
Accepted 20 January 2023

ABSTRACT

SDS/Triton X-100, a mixed anionic and non-ionic surfactant system, has been chosen to study the oxidation of aniline by Fe(III) in the presence of bipyridyl. The reaction obeys pseudo first order kinetics with respect to aniline and Fe(III). The reaction has a square dependence on the concentration of bipyridyl and inverse square dependence on hydrogen ion concentration. There is no considerable effect of Triton X-100 (TX-100) micelles on the rate of the reaction. In contrast to this observation the reaction is markedly accelerated in the presence of SDS. The effect of varying mole fraction of Triton X-100 and total surfactant concentration has been carried out in the presence of mixed micelles (SDS/TX-100) and it was found that rate decreases as mole fraction of Triton X-100 (α_{TX-100}) increases. The extent of interaction (β) between the surfactant molecules were also determined which could explain synergistic behavior in SDS/Triton X-100 mixed micelles. Based on kinetic observations a reaction scheme has been proposed. The applicability of the Berezin pseudo - phase model has been examined in the case where the number of equilibria comes before the rate-determining step. The values of K_S determined were in good agreement with values obtained from spectrophotometric method showing the applicability of Berezin pseudo - phase model to kinetic analysis of reactions involving pre-equilibria in the presence of mixed micelles.

Keywords: *aniline, Fe(III), mixed micelles, SDS, triton-X 100, berezin pseudo-phase model.*

INTRODUCTION

Mixed normal micelles are defined as normal micelles formed from monomers of two different surfactants in aqueous solutions. The most important effect is lowering of interfacial tension due to preferential adsorption of surfactant molecules at solution interfaces. Therefore rates of chemical reactions can be significantly influenced by the presence of mixed micelles due to their synergistic and antagonistic effects [1 - 6]. Physico-chemical aspects such as, micelle composition, and aggregation number, CMC measurements, micelle modeling were studied in the presence of mixed micelles but very few studies were reported on kinetics and catalytic effect of mixed micelles on reactions [7 - 10]. Therefore to study kinetics and catalytic effects of mixed anionic (SDS)/nonionic (Triton-X 100) micelles on the rates, oxidation of aniline by iron (III) has been chosen. Aniline is a toxic compound used in the preparation of paints, rubbers, pharmaceutical preparation, dyes, plastics. The chemical oxidation of aniline by iron (III) in the presence of mixed micelles can result in almost complete oxidation of aniline with greater rate. The kinetics of this reaction has been carried in the presence of 2, 2'- bipyridyl at different mole fractions of Triton X-100 (α_{TX-100}) and at different total surfactant concentration values C_t. The binding constants of aniline in mixed micelles have been determined and kinetic results were analysed by using Berezin pseudo phase model. The extent of interaction (b) between the surfactant molecules were also determined which could explain synergistic behavior in SDS/Triton X-100 mixed micelles.

EXPERIMENTAL

Materials

The chemicals used were of the analytical reagent grade, and all solutions were made in double-distilled water. SDS and Triton X-100 were used without further purification.

0.1 mol dm^{-3} iron (III) solution was prepared by dissolving 4.822 gm of ammonium ferric sulphate in aqueous perchloric acid (Merck) medium. The iron (III) solution was standardized by titrating with EDTA using variamine blue B as indicator [11]. A 0.05 mol dm^{-3} 2, 2'- bipyridyl (BDH AnalaR) solution was prepared by dissolving 3.203 gm of 2, 2'-bipyridyl in 100 ml double distilled water. A 0.1 mol dm^{-3} of aniline (Sigma - Aldrich, analytical reagent grade) solution was prepared in 1 % v/v methanol-water mixture. The solution was standardized by the method described by Vogel [12].

Determination of interaction parameter (β) and calculation of critical micellar concentrations (CMC) of mixed surfactants

CMC obtained from experiment is known as C_{exp} which were determined by using surface tension method and CMC obtained from calculation known as C_{cal} were calculated by Rubingh method [13]. Results were tabulated in Table 1. The value of b is a measure of the extent of interaction between the surfactants where negative values indicate synergism (unlike interactions, i.e, anionic-nonionic) and positive values antagonism. The b values proved that there is marked interaction between SDS and Triton X-100 surfactants. The synergistic behavior in SDS/Triton X-100 system is due to the ethoxylated chains of the Triton X-100 surfactants coil around the charged head groups of SDS, screening the electrostatic repulsion and favouring the micelle formation.

Kinetic Measurement

Kinetic runs have been carried out at different concentrations of SDS and Triton X-100 keeping the concentrations of aniline, bipyridyl, Fe(III) and hydrogen ion constant {Under the pseudo-first order conditions [aniline] >> [bipy] >> [Fe(III)]}. A series of kinetic runs were also carried out at different mole fractions of Triton X-100 (α_{TX-100}) and at different total surfactant concentration values C_t, keeping the concentrations of Fe(III), aniline, bipyridyl and hydrogen ion constant.

A Shimadzu UV-1800 spectrophotometer has been used to measure the product $[Fe(bipy)_3]^{2+}$ absorbance's at

Table 1. CMC_{12}, X_1, b and Average b for SDS/Triton X-100 system. $[NaClO_4] = 0.08$ mol dm^{-3}, $[HClO_4] = 5 \times 10^{-3}$ mol dm^{-3}, T = 30° ± 0.1°C

Mole fraction of Triton X-100 α_{TX-100}	$C_{12}\times10^3$ mol dm^{-3} exptl	$C_{12}\times10^3$ mol dm^{-3} ideal	$C_{12}\times10^3$ mol dm^{-3} cal	X_1	β	f_1	f_2	$C_{mon}\times10^3$ mol dm^{-3}
0.0	1.20	-	-	-	-	-	-	-
0.1	0.71	0.932	0.71	0.371	-1.22	0.618	0.849	0.71
0.2	0.57	0.762	0.57	0.495	-1.16	0.743	0.752	0.57
0.4	0.43	0.559	0.54	0.645	-1.20	0.860	0.606	0.43
0.5	0.40	0.493	0.40	0.709	-1.11	0.910	0.572	0.40
0.6	0.39	0.441	0.39	0.216	-0.82	0.603	0.962	0.39
0.8	0.32	0.364	0.32	0.148	-1.41	0.360	0.970	0.32
0.9	0.26	0.335	0.26	0.173	-3.04	0.125	0.913	0.26
1.0	0.31	-	-	-	-	-	-	-
Average interaction parameter (β_{avg}) is -1.42								

$C_{12exptl}$: Experimental values of CMC of mixed surfactants; $C_{12ideal}$: Ideal values of CMC of mixed surfactants from Rubingh method; C_{12cal}: Calculated values of CMC of mixed surfactants from Rubingh method; α_{TX-100}: Stoichiometric mole fraction of TX-100; X_1: Actual mole faction of TX-100; f_1 and f_2: activity coefficients of SDS and TX-100; C_{mon}: Critical mononmer concentration of mixed surfactants obtained from Rubingh method

520 nm in order to track the progress of the reaction. All other materials involved have a negligible absorbance at this wavelength. The rate constants were consistently tested using duplicate kinetic runs, and it was observed that they were repeatable to within 3 %. The pseudo first order rate constants, k', were calculated from plots of log (A_∞-A_t) versus time and presented in Table 2.

The product was found to be azo compound and confirmed by the spot test prescribed by Feigl [14]. The stoichiometry for the oxidation of aniline by Fe(III) in the presence of bipyridyl was determined by spectrophotometry using mole ratio method, the results indicate that one mole of aniline reacts with two moles of iron(III).

$$2[Fe(bipy)_2]^{3+} + 2Hbipy^+ + 2ArNH_2 \rightarrow Ar - N = N - Ar + 2[Fe(bipy)_3]^{2+} + 6H^+$$

Determination of binding constant of aniline:

To determine the binding constant of bipyridyl and aniline in the presence of mixed micelles of SDS/Triton X-100, spectra of bipyridyl and aniline (under the conditions $[H^+] = 0.02$ mol.dm^{-3} and ionic strength (μ) = 0.08 mol dm^{-3} maintained by $NaClO_4$) have been scanned at various mole fractions of Triton X-100 and at various C_t (total surfactant concentrations) .

A_m is the absorbance in the presence of mixed micelles, A_W^0 is the absorbance in the absence of mixed micelles, and A_M^0 is the limiting absorbance in the presence of mixed micelles, and these values have been determined at wavelengths of 255 nm. Equation (1) was used to calculate the binding constant (K_B, K_S) [15] of bipyridyl and aniline.

$$\frac{1}{(A_M - A_W^0)} = \frac{1}{(A_M^0 - A_W^0)} + \frac{1}{(A_M^0 - A_W^0) K_S C_M}$$

Plotting $1/(A_M - A_W^0)$ against $1/C_M$, where $C_M = C_t$ [SDS] + [Triton X-100] - C_{mon} is derived from micellization studies. The binding constants, K_B, K_S, of bipyridyl and aniline were determined at various

Table 2. The pseudo-first order rate constants, k', were calculated from plots of log (A_∞-A_t) at various conditions at constant temperature $30^0 \pm 0.1^oC$.

[SDS] ×10^3 mol dm^{-3}	[TX-100] ×10^3 mol dm^{-3}	[C_t] ×10^3 mol dm^{-3}	α_{TX-100}	[Aniline] ×10^3 mol dm^{-3}	[Fe (III)]×10^5 mol dm^{-3}	[Bipy] ×10^3 mol dm^{-3}	[H$^+$] ×10^3 mol dm^{-3}	μ mol dm^{-3}	k′×10^4 s^{-1}
8.00	8.00	16.0	0.50	4.00	4.00	1.60	4.00	0.08	29.9
					6.00				30.1
					8.00				30.4
12.0	12.0	24.0	0.50	0.80	6.00	1.60	4.00	0.08	4.00
				2.00					15.7
				4.00					30.1
				6.00					46.4
				8.00					61.9
				4.00	6.00	0.16	4.00	0.08	1.49
						0.40			2.53
						0.80			9.13
						1.20			19.1
						1.60			30.1
						2.00			48.9
4.00	4.00	8.00	0.50	4.00	6.00	1.60	4.00	0.08	30.1
							5.00		19.4
							6.00		13.4
							8.00		7.58
							10.0		4.98
							12.0		3.56

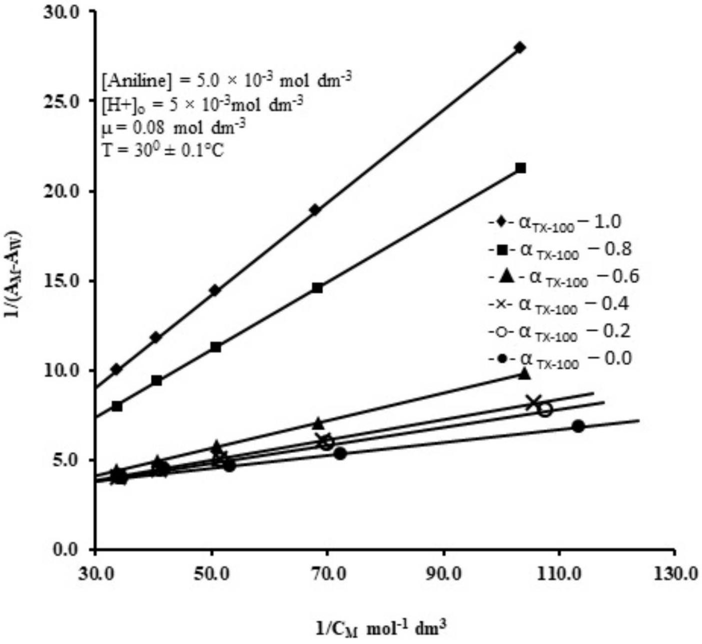

Fig. 1. Plot of $1/(A_M-A_W)$ vs. $1/C_M$ for aniline.

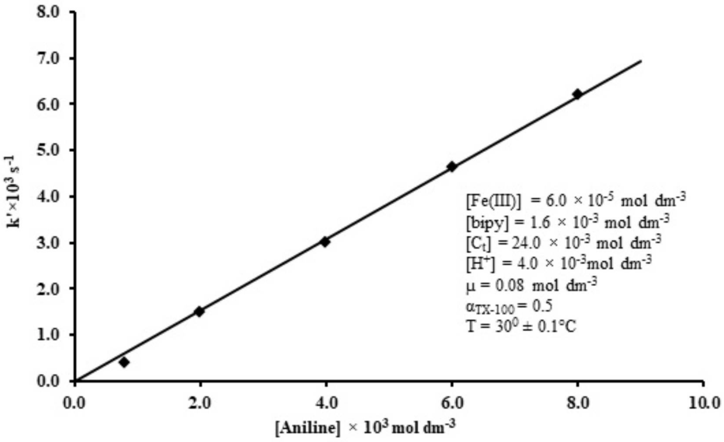

Fig. 2. Plot of k' vs. [aniline].

mole fractions of Triton X-100 based on the slope and intercept of this plot (Fig. 1).

RESULTS AND DISCUSSION

Kinetic runs have been carried out at different concentrations of SDS and Triton X-100 and at different mole fractions of Triton X-100 (α_{TX-100}), under the pseudo-first order conditions [aniline] >> [bipy] >> [Fe(III)]}. Good linear plots between log (At) versus time were obtained indicating first order kinetics with respect to [Fe(III)]. Plot of k' versus [aniline] was found to be passing through origin showing that the reaction obeys first order kinetics with respect to [aniline] (Fig. 2). The reaction has square dependence on the concentration of bipyridyl (Fig. 3), inverse square dependence on hydrogen ion concentration (Fig. 4). There is no effect of Triton X-100 micelles on the rate of the reaction (Table 3). This is probably due to absence of electrostatic interactions with the reactants.

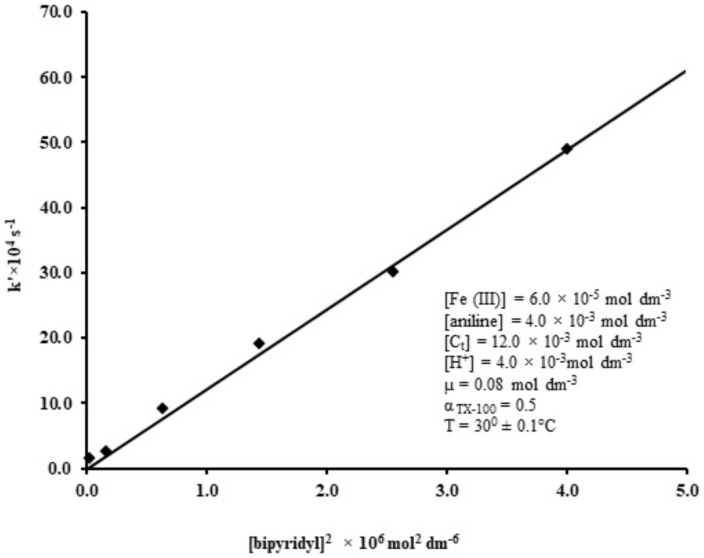

Fig. 3. Plot of k' vs. $[bipyridyl]^2$.

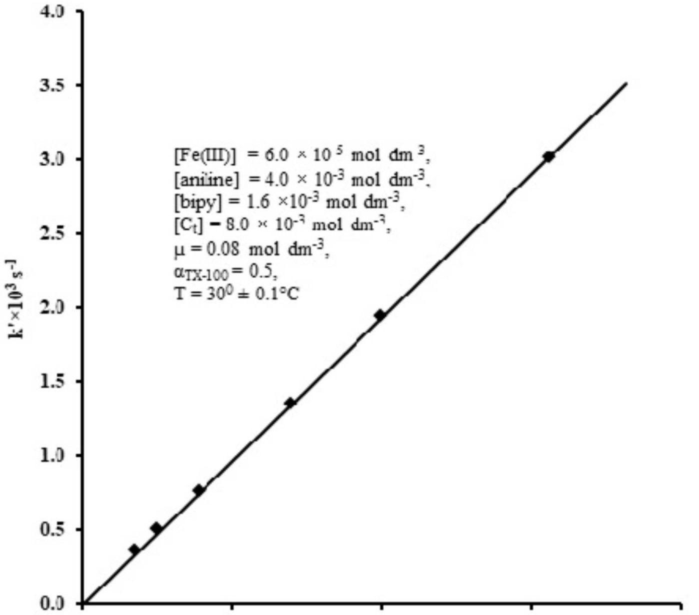

Fig. 4. Plot of k' vs. $1/[H^+]^2$ on rate.

In contrast to this observation the reaction is markedly accelerated in the presence of SDS due to the binding of the reactants in a small volume of Stern layer of the micelle thus leading to considerable concentration effect. There is a possibility that the transition state having a net charge of +4 is stabilised more than the less positively charged initial state in the micellar pseudo-phase leading to the lowering of activation energy. In the range of H^+

concentration employed in the present study (4.0 - 12.0) × 10^{-3} mol dm^{-3}, all bipyridyl can be considered to be in the protonated form $Hbipy^{+}$, (pKa of $Hbipy^{+}$ is 4.35). The pKa value of $C_6H_5NH_3^{+}$ is 4.54. [16 - 17]. Hence almost all aniline is present in the protonated form under these conditions.

The interaction between the concentration of total surfactant and the mole fraction of Triton X-100 in the presence of mixed micelles shows that as the mole fraction of Triton X-100 increases, rate of reaction decreases. Electrostatic interactions and rate both decrease as the negative charge density brought on by SDS reduces with increasing TX-100 concentration. Electrostatic repulsion between the SDS head groups is reduced by the presence of non-ionic surfactant molecules.

To explain the kinetic features, the author suggests the scheme 1.

In this scheme, M and W stand for the micellar and aqueous phases, respectively.

$[H^{+}]_M$ is the hydrogen ion concentration in moles per litre of the Stern layer. $[Hbipy^{+}]_t$, $[Fe(III)]_t$, and $[ArNH_3^{+}]_t$, represents total concentrations. Binding constants of Fe(III), $Hbipy^{+}$, and $ArNH_3^{+}$ are represented by K_A, K_B, and K_S. Corresponding partition coefficients are represented by P_A, P_B, and P_S. V is the molar volume. C_t is the total surfactant concentration of mixed surfactants. C_{mon} is the cmc of mixed surfactants obtained from micellisation studies. C_M is the micellar concentration of mixed surfactants (C_t-C_{mon}).

Scheme 1. Mechanism of the reaction.

$$\begin{array}{lll} Fe^{3+}_{W} & Hbipy^{+}_{W} & ArNH^{+}_{3W} \\ \upharpoonleft\!\downharpoonright K_A & \upharpoonleft\!\downharpoonright K_B & \upharpoonleft\!\downharpoonright K_S \\ Fe^{3+}_{M} & Hbipy^{+}_{M} & ArNH^{+}_{3M} \end{array} \tag{2}$$

$$Fe^{3+}_{M} + Hbipy^{+}_{M} \overset{K_{1M}}{\rightleftharpoons} [Fe(bipy)]^{3+}_{M} + H^{+}_{M} \tag{3}$$

$$[Fe(bipy)]^{3+}_{M} + Hbipy^{+}_{M} \overset{K_{2M}}{\rightleftharpoons} [Fe(bipy)_2]^{3+}_{M} + H^{+}_{M} \tag{4}$$

$$Fe^{3+}_{W} + Hbipy^{+}_{W} \overset{K_{1W}}{\rightleftharpoons} [Fe(bipy)]^{3+}_{W} + H^{+}_{W} \tag{5}$$

$$[Fe(bipy)]^{3+}_{W} + Hbipy^{+}_{W} \overset{K_{2W}}{\rightleftharpoons} [Fe(bipy)_2]^{3+}_{W} + H^{+}_{W} \tag{6}$$

$$[Fe(bipy)_2]^{3+}_{M} + ArNH^{+}_{3M} \xrightarrow{k_M} [Fe(bipy)_2]^{2+}_{M} + ArNH^{\bullet}_{M} + 2H^{+}_{M} \tag{7}$$

(rate determining step)

$$[Fe(bipy)_2]^{3+}_{W} + ArNH^{+}_{3W} \xrightarrow{k_W} [Fe(bipy)_2]^{2+}_{W} + ArNH^{\bullet}_{W} + 2H^{+}_{W} \tag{8}$$

(rate determining step)

$$ArNH^{\bullet}_{M/W} + [Fe(bipy)_2]^{3+}_{M/W} \rightarrow ArN^{\bullet}_{M/W} + [Fe(bipy)_3]^{2+}_{M/W} + H^{+}_{M/W} \tag{9}$$

$$2ArN^{\bullet} \rightarrow Ar\ N{=}N\ Ar \tag{10}$$

$$[Fe(bipy)_2]^{2+}_{M/W} + Hbipy^{+}_{M/W} \rightarrow [Fe(bipy)_3]^{2+}_{M/W} + H^{+}_{M/W} \tag{11}$$

Berezin's method can be used to illustrate how the mechanism results in the rate law.

$$\text{Rate} = \frac{k_M K_{1M} K_{2M}\left\{P_A[Fe^{3+}]_t/(1+K_A C_M)\right\}\left\{P_B^2[bipy^+]_t^2/(1+K_B C_M)^2\right\}\left\{P_S[ArNH_3^+]_t/(1+K_S C_M)\right\}(\bar{V}C_M)}{[H^+]_M^2\{\left\{\left(K_{1M}P_B[Hbipy^+]_t[H^+]_M\right)/(1+K_B C_M)\right\}+\left\{\left(k_{1M}K_{2M}P_B^2[Hbipy^+]_t^2\right)/(1+K_B C_M)^2\right\}\}}$$

$$+\frac{k_W K_{1M} K_{2M}\left\{[Fe^{3+}]_t/(1+K_A C_M)\right\}\left\{[Hbipy^+]_t^2/(1+K_B C_M)^2\right\}\left\{[ArNH_3^+]_t/(1+K_S C_M)\right\}(1-\bar{V}C_M)}{[H^+]_M^2\{\left\{\left(K_{1M}P_B[Hbipy^+]_t[H^+]_M\right)/(1+K_B C_M)\right\}+\left\{\left(k_{1M}K_{2M}P_B^2[Hbipy^+]_t^2\right)/(1+K_B C_M)^2\right\}\}} \quad (12)$$

Given that the K_A for iron (III) is 9.28×10^4 dm^{-3}/mol, the extent of the reaction in the aqueous phase compared to the micellar phase is negligible. As a result, the second term can be neglected. In the equation (12), the denominator of the first term $[H^+]_M >> K_{1M}P_B[H^+]_M[Hbipy^+]_t/(1+K_BC_M) + K_{1M}K_{2M}P^2{}_B[Hbipy^+]^2{}_t/(1+K_BC_M)^2$ is negligible compared to $[H^+]_M$.

As a result, the equation (12) has become an equation (13).

$$\text{Rate}=\frac{k_M K_{1M}K_{2M}P_A P_B^2 P_S[Fe^{3+}]_t[Hbipy^+]_t^2[ArNH_3^+]_t\bar{V}C_M}{\left([H^+]_M^2(1+K_AC_M)(1+K_BC_M)^2(1+K_SC_M)\right)} \quad (13)$$

The equation explains the aniline and iron(III) first order behavior as well as the direct dependence of the rate on $[bipy]^2$ and $1/[H^+]^2$. This concentration-dependent rate further proves that equation (7) is the only step in the process that determines rate. Otherwise, under the circumstances of [aniline] >> [Fe(III)], log $(A-A_t)$ vs. time plots should not have been linear and should have shown biphasic or multiphasic trends.

In equation (13) $[H^+]_M$ is the concentration of H^+ ion in moles per liter of micellar phase $(M^S{}_{H+}/\bar{V})$ and is given by the expression $(0.82 \times [H^+]_t/[Na^+]_t\bar{V})$ the equation modifies to

$$\text{Rate}=\frac{k'_M K_{1M}K_{2M}P_A P_B^2 P_S[Fe^{3+}]_t[Hbipy^+]_t^2[ArNH_3^+]_t\bar{V}C_M}{\left([H^+]_M^2+(1+K_AC_M)(1+K_BC_M)^2(1+K_SC_M)\right)} \quad (14)$$

where $k'_M = k_M/(0.82/[Na^+]_t\bar{V})$.

Since K_A is the binding constant of Fe^{3+} has a value in the order of 10^4 at $[C_t] >> CMC$, $K_AC_M>>1$, the equation

$$k' = \frac{k'_M K_{1M}K_{2M}P_A P_B^2 P_S[Fe^{3+}]_t[Hbipy^+]_t^2[ArNH_3^+]_t}{\left([H^+]_M^2(1+K_AC_M)(1+K_BC_M)^2(1+K_SC_M)\right)}$$

The equation (15) rearranges to

$$\frac{1}{k'(1+K_BC_M)^2}=$$

$$=\frac{[H^+]_t^2}{k'_M K_{1M}K_{2M}P_A P_B^2 P_S[Fe^{3+}]_t[Hbipy^+]_t^2[ArNH_3^+]_t}+\frac{K_SC_M[H^+]_t^2}{k'_M K_{1M}K_{2M}P_A P_B^2 P_S[Fe^{3+}]_t[Hbipy^+]_t^2[ArNH_3^+]_t} \quad (16)$$

In order to determine the value of K_S, the binding constant of $C_6H_5NH_3{}^+$, a plot of $1/k'(1+K_BC_M)^2$ vs. C_M should be linear with a positive intercept. These plots have been obtained for all α_{TX-100} value ranges (Fig. 5). The results demonstrate the applicability of the Berezin pseudo-phase model to kinetic analysis of reactions involving pre-equilibria in the presence of mixed micelles because they are in good agreement with the value of K_S determined independently using the spectrophotometric method (Table 3).

Synergism of mixed surfactants on rate of the reaction and kinetic parameters

The values in Table 4 demonstrate that as the value of α_{TX-100} decreases from 1 to 0, there is a continuous change in the mixed micellar characteristics from pure Triton X-100 to pure SDS. However, the K_S values obtained in pure Triton X-100 micellar solutions and the K_S values in mixed micellar solutions with α_{TX-100} 0.6 and 0.8 are comparable. Because of synergism in mixed micellar solutions, the values should have been significantly higher than the actual values. The negative value indicates synergism as well (Table 1).

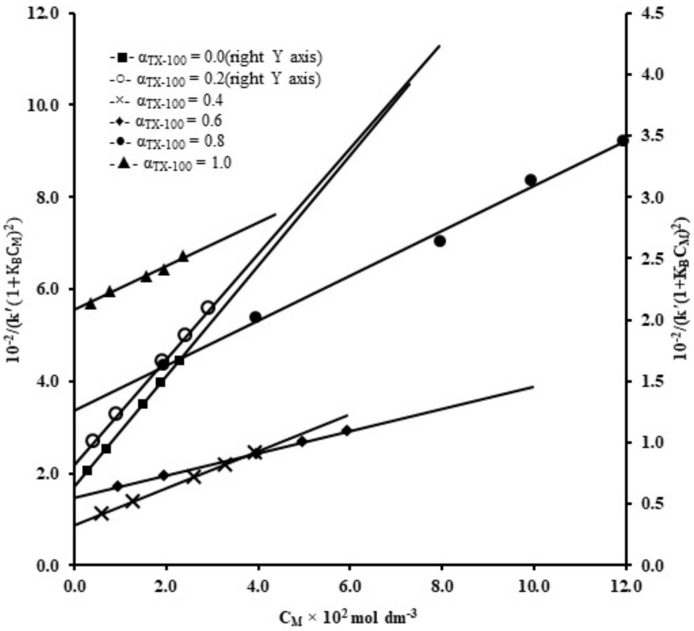

Fig. 5. Plots of $1/(k'(1+K_BC)^2)$ vs. C_M for aniline.

Table 3. Effect of mole fraction (α_{TX-100}) and C_t at different mole fractions of Triton X-100.
[Fe(III)] = 4.0×10^{-5} mol dm^{-3}, [aniline] = 4.0×10^{-3} mol dm^{-3}, [bipy] = 1.6×10^{-3} mol dm^{-3}, [H$^+$] = 4.0×10^{-3} mol dm^{-3}, μ = 0.08 mol dm^{-3}, T = $30^0 \pm 0.1$°C

[SDS] × 10^3 mol dm^{-3}	$k' \times 10^4$ s^{-1}					
	α_{TX-100}					
	0.0	0.2	0.4	0.6	0.8	1.0
4.0	110	85.6	78.9	51.5	20.0	1.75
8.0	72.5	60.2	55.7	39.8	13.3	1.67
16.0	36.6	33.6	31.5	25.1	7.23	1.58
20.0	27.4	26.2	24.8	20.4	5.64	1.54
24.0	21.1	20.8	19.9	16.9	4.52	1.47

Table 4. Comparison of binding constant of aniline (K_S) obtained from kinetics and from spectrophotometric data at different mole fractions of Triton X-100.

α_{TX-100}	K_S (Aniline) mol^{-1} dm^3 from spectrophotometry	K_S (Aniline) mol^{-1}dm^3 from kinetics
0.0	78.9	69.4
0.2	46.9	54.7
0.4	37.8	47.3
0.6	24.1	16.9
0.8	8.77	13.2
1.0	5.04	8.50

The synergistic behavior is explained by assuming that in SDS/Triton X-100 system, the ethoxylated chains of the Triton X-100 surfactants coil around the charged head groups of SDS, screening the electrostatic repulsion and favouring the micelle formation.

CONCLUSIONS

- The reaction follows first-order kinetics with respect to aniline and Fe(III). The concentration of bipyridyl has a square dependence on the reaction, and the concentration of hydrogen ions has an inverse square dependence.
- The mechanism of the reaction proposed involves the rate-determining step which is preceded by a number of equilibria in mixed micelles of SDS and Triton X-100.
- As the mole fraction of Triton X-100 (TX-100) increases, the rate of reaction decreases in mixed micelles.
- The binding constant of aniline K_S, obtained from spectrophotometry are in good agreement with the binding constants from Berezin pseudo-phase model, indicating applicability of the Berezin pseudo-phase model to kinetic analysis of reactions involving pre-equilibria in the presence of mixed micelles.

Acknowledgements

One of the authors (R S S Srikanth Vemuri) expressed his gratitude towards Dr. J. Sudhakar, Principal, Vignan's institute of engineering for women for his continuous encouragement throughout writing of this article. Authors expressed sincere thanks to Andhra University for providing lab facilities to conduct experiments.

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