Surfactant Catalyzed Oxidation of Ethanolamines by Cerium(IV)

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Effect of surfactant medium on the kinetics of oxidation of amino alcohol by cerium(IV) has been reported. Two amino alcohols namely, monoethanolamine (MEA) and triethanolamine (TEA) are chosen for kinetic study. Sizeable changes in reaction rate are noted only in presence of sodium lauryl sulphate (NaLS) as surfactant. Both the amino alcohols exhibit rate maxima at around the cmc of NaLS, beyond which the k_ψ-[NaLS] profile shows slow increase in rate constant with increasing NaLS concentration. Suitable model has been used to explain the kinetic pattern post CMC and from this the micelle-reactant binding constant values have been evaluated. From the temperature dependence study, the activation parameters for the oxidation reactions have been computed and these are compared against those obtained for aqueous medium. Based on all information, plausible mechanism for micellar catalysis has been presented.

Keywords: Micelles, Amino alcohol, Monoethanolamine, Triethanolamine, Sodium lauryl sulphate, Activation parameters.

INTRODUCTION

Most biochemical processes are known to proceed in a micro-heterogeneous system containing an aqueous and a lipophilic moiety [1]. Among biochemical processes, electron transfer reactions are of importance [2]. Respiration and photosynthesis provide the bulk energy requirement for maintenance of life on earth belong to this category. Kinetic studies of electron transfer reactions in micro heterogeneous medium help in understanding the mechanism of electron transport in such media [3]. Conversion of alcohols in to aldehydes or acids is one of the fundamental and most studied process for industrial production of fine chemicals and from biological point of view also. In this connection, oxidation of amino alcohols to yield amino aldehydes or amino acids has been studied recently [4-7].

The products obtained out of these are the precursors of various organic moieties having antiviral, anticancer and antidiabetic effects [8-11]. Enzymatic oxidation or aerial oxidation processes require Ru, Au, Pt or Pd as catalysts and hence makes these processes non-economical [12-14]. In continuation to our earlier works [15-17], we present here the effect of micellar medium of sodium lauryl sulphate on the Mn(II) catalyzed oxidation of amino alcohols by Ce(IV). The choice of Ce(IV)

an as oxidant in the present work is well justified because it is a known oxidant in acid media (sulphuric acid, nitric acid, perchloric acid) with a reduction potential of 1.7 volts for (Ce^{3+}, Ce^{4+}) couple [18-21]. Surface active agents, *i.e.* surfactants consisting of hydrophilic head and hydrophobic tail reduce the surface tension of liquids when dissolved in water [22-25]. Micelle formation at CMC that depends on the nature of surfactant head or tail, temperature, ionic strength of solvents, additives, *etc.* is an important phenomenon since they have the ability to influence the reaction dynamics [26,27]. Surfactants find wide range of applications in industries like agriculture, pharmaceuticals, food and has influence on day-to-day activities of human life [28-30] hence our present work on the effect of micellar medium of sodium lauryl sulphate on Mn(II) catalyzed oxidation of amino alcohols by Ce(IV) bears importance.

Amino alcohols, *viz.* monoethanolamine (MEA) and triethanolamine (TEA) were used as substrates in the kinetic study. The redox reactions were carried out under pseudo-first order conditions with concentration of amino alcohols at least ten times greater than that of the oxidant Ce(IV). In aqueous medium, these reactions showed some common kinetic features. The reactions were first order with respect to [Ce(IV)], the dependence of rate on substrate concentration showed Michaelis-

Menten type kinetic pattern, the rate constant values increased with increasing dielectric constant of the medium but exhibited inverse [H⁺] dependence and the entropy of activation was negative. Kinetic information suggested that the reactions proceeded through intermediate complex formation. Such observations are in conformity with reports published elsewhere [31]. The reactivity order was found to be TEA > MEA. With TEA, the oxidation reaction was too fast to require any Mn(II) for catalysis, which was otherwise necessary for oxidation of MEA. It was further verified that in the concentration range studied (10 to 100 times less than [Ce(IV)]), Mn(II) did not reduce Ce(IV). As such their standard reduction potentials are comparable [32].

Three types of surfactants, *viz.* anionic (sodium lauryl sulphate, NaLS), cationic (cetyltrimethylammonium bromide, CTAB) and non-ionic (Triton X-100) were tried to study the effect of micellar medium. Of these, the last mentioned surfactant was excluded from the study due to immediate precipitation under conditions of study. With CTAB, there was hardly any kinetic change; measurable effects were noticed only in NaLS surfactant medium. The present paper deals with the results of kinetic studies on the effect of sodium lauryl sulphate on the oxidation of monoethanolamine (MEA) and triethanolamine (TEA) under varying conditions of surfactant concentration and temperature.

EXPERIMENTAL

Amino alcohols (Qualigen), ceric ammonium sulphate (Qualigen), manganese sulphate tetrahydrate (S.D. Fine Chemicals) and sulphuric acid (E. Merck), all analytical grade samples were used. Acidic solution of ceric ammonium sulphate was standardized by literature method [33]. Sodium lauryl sulphate (BDH) was used after recrystallization from ethanol. All solutions were prepared in doubly distilled conductivity water.

Kinetic measurements were performed spectrophotometrically under pseudo-first order conditions using a Shimadzu UV 1700A spectrophotometer and 1cm quartz cell. The reactions in aqueous medium were initiated by mixing previously thermostated solutions in the order acid, amino alcohol, Mn(II) and finally Ce(IV). The course of reaction was followed by monitoring the decrease in absorbance of Ce(IV) at 380 nm as a function of time. There was negligible interference from other reagents at this wave length. In the concentration range of Ce(IV) studied, Beer's law was obeyed with $\lambda = 533 \pm 20$ dm³ mol⁻¹ cm⁻¹. For reactions in micellar medium, the surfactant solution, previously thermostated at the same temperature as other reagents, was first mixed with the Ce(IV) solution, and this mixture was added to the mixture of other reagents to initiate the reaction. The wavelength of maximum absorption of Ce(IV) was hardly altered and hence all kinetic measurements were carried out at 380 nm in micellar medium also.

RESULTS AND DISCUSSION

Oxidation reactions of amino alcohols have been carried out at different concentrations of sodium lauryl sulphate starting from below CMC ($\approx 5 \times 10^{-3}$ mol dm⁻³ for the present study)

up to a high concentration under constant conditions of other reaction parameters. Disappearance of Ce(IV) in each case is observed to be first order. Plot of log A_t against time is linear and from the slope of this plot pseudo-first order rate constant (k_ψ) is computed. Further, temperature effect on the reactions in surfactant medium is studied by monitoring the reactions at constant concentration of NaLS in the temperature range from 30-50 ºC. A number of constant concentrations of NaLS ranging from 2×10^{-3} to 5×10^{-2} mol dm⁻³ have been used for the purpose.

For both the amino alcohols, variation of rate constant of the oxidation reactions with increase in [NaLS] is found to be more or less same. With increasing NaLS concentration, the rate constant at first increases, almost rapidly and this continues up to [NaLS]= 8-10×10^{-3} mol dm⁻³. Beyond this value, there is a rapid fall in rate constant so that the k_ψ-[NaLS] profile shows a maximum in each case (Fig. 1). After the maximum, the rate constant slowly but distinctly increases with further increase in NaLS concentration. Increase in temperature increases k_ψ values monotonically. The increase is compounded when there is simultaneous increase in NaLS concentration (Table-1). The maximum value of k_ψ, *i.e.* the value of k_ψ at around [NaLS] = 1.0×10^{-3} mol dm⁻³, also increases systematically with rise in temperature. When k_ψ values (s⁻¹) are converted to k_2 (mol⁻¹ s⁻¹), Arrhenius plots at each NaLS concentration are found to be linear. However, the activation parameters obtained from such plots are of little use; instead the activation parameters for the reactions in pure micellar medium have been computed using different models. It is suffice to mention here that the activation entropy and enthalpy in the pure micellar medium of NaLS are comparable to those obtained in the absence of surfactant.
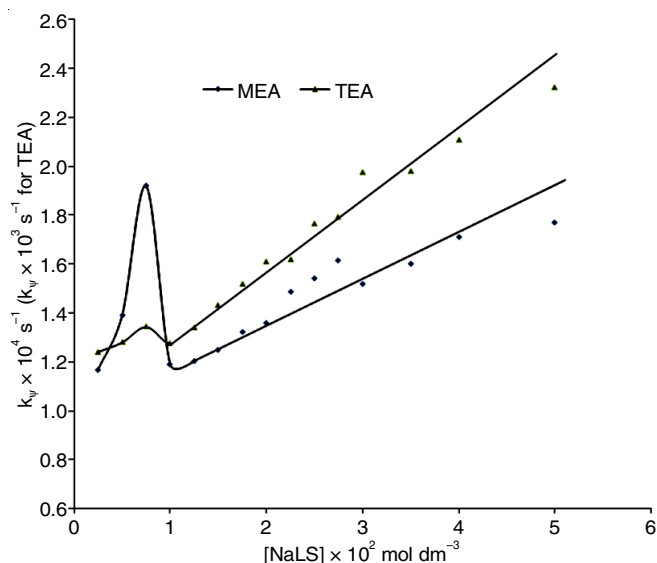

Fig. 1. Plot of k_ψ against [NaLS] for MEA and TEA. Oxidant: Ce(IV), Solvent: Water, Temperature: 303 K

Mechanism: As discussed earlier, the reactivity pattern in presence of NaLS, a brief description regarding the mechanism for the oxidation reactions in aqueous medium should be considered. For oxidation in aqueous medium the main kinetic features are mentioned earlier. These indicate inter-

TABLE-1
VARIATION OF RATE CONSTANT WITH [NaLS] FOR
THE OXIDATION OF MEA BY Ce(IV) AT DIFFERENT
TEMPERATURES. [Ce(IV)] = 3×10^{-3}, [MEA] =
3×10^{-2} AND [H$^+$] = 4.0 mol dm^{-3}

| $10^2 \times$ [SDS] (mol dm^{-3})	$10^4 \times k_\psi$ (s^{-1})				
	303 K	307 K	311 K	315 K	319 K
0.00	0.541	0.776	1.109	1.396	1.952
0.25	1.169	1.640	2.290	3.162	4.365
0.50	1.391	1.972	2.754	3.845	5.248
0.75	1.919	2.754	3.845	5.308	7.328
1.00	1.190	1.717	2.426	3.349	4.623
1.25	1.201	1.778	2.511	3.467	4.786
1.50	1.250	1.823	2.540	3.507	4.841
1.75	1.322	1.883	2.630	3.630	5.011
2.00	1.360	1.927	2.691	3.715	5.128
2.25	1.486	2.098	3.053	4.027	5.588
2.50	1.543	2.180	3.123	4.205	5.813
2.75	1.614	2.264	3.227	4.303	5.952
3.00	1.520	2.137	3.259	4.168	5.754
3.50	1.600	2.372	3.404	4.516	6.262
4.00	1.709	2.426	3.427	4.731	6.606
5.00	1.770	2.562	3.571	4.917	6.830

mediate complex formation between the substrate and the oxidant. The intermediate complex then breaks down in a slow step and culminates in product formation through a number of subsequent fast reactions.

As mentioned earlier, the activation entropy and enthalpy in micellar medium are comparable to those in the aqueous medium. Further, for both the amino alcohols, the oxidation reaction in presence of NaLS retains the same kinetic features as in the absence of NaLS. The reaction is first order in [Ce(IV)], fractional order with respect to [amino alcohol] and shows inverse order of dependence on [H$^+$], *etc*. Therefore, the mechanism of oxidation of amino alcohols in presence of NaLS would broadly remain the same, *i.e.* a bimolecular transition state of amino alcohol and Ce(IV) would be involved. At low concentration of NaLS, the k_ψ values increase with [NaLS] and this observation is attributed to increasing solubilization of the reactant species which increases with NaLS concentration and reaches a limiting value in the vicinity of CMC. As surfactant concentration is further increased, a dilution effect is exerted on the reactants due to increase in concentration of the micellar phase in the reaction medium. This in turn decreases the rate of reaction resulting in a maximum in the plot of k_ψ against [NaLS]. Similar observations are common in literature [34-37].

The slow acceleration of the rate of oxidation by Ce(IV) beyond the maxima, in either amino alcohol presents an interesting case and needs explanation. Under conditions of study (4N H$_2$SO$_4$), the reductant amino alcohols are predominantly in the protonated form due to high pK_a values of the latter [38]. With regard to reactive Ce(IV) species, it has been reported [18,39,40] that in sulphuric acid medium with [HSO$_4^-$] or [SO$_4^{2-}$] >> [Ce(IV)], the Ce(IV) may exist in a number of sulphate or protonated sulphate complex species, which may be neutral, [*e.g.* Ce(SO$_4$)$_2$] or bear either positive [*e.g.* Ce(SO$_4$)$^{2+}$, Ce(HSO$_4$)$^{3+}$] or negative [*e.g.* Ce(SO$_4$)$_3^{2-}$] charges.

On the basis of the equilibrium constant values [41], the predominant sulphate complex species are found to be in the

order Ce(SO$_4$)$_3^{2-}$ > Ce(SO$_4$)$_2$ > Ce(SO$_4$)$^{2+}$ with trace amounts of Ce^{4+}, Ce(OH)$^{3+}$, *etc*. The fact that the oxidation reaction is accelerated in anionic surfactant medium suggests Ce(SO$_4$)$^{2+}$ to be the reactive species although the role of Ce(SO$_4$)$_2$ cannot be ruled out. Such suggestions are consistent with kinetic findings and supported by the mentioned effects of cationic surfactant CTAB on the oxidation rate. The observed effects of micellar CTAB medium on the redox reaction are marginal and not opposite to what observed in micellar SDS medium. This excludes involvement of the negatively charged Ce(SO$_4$)$_3^{2-}$ species in the redox process. Thus, in SDS medium partitioning of both the cationic Ce(IV) species and the substrate between water and micellar pseudo-phase is considered and accordingly Menger and Portnoy model [42-44] (**Scheme-I**) is extended to explain the kinetic data. It may be noted that in cationic surfactant of CTAB, no such partitioning is expected on electrostatic grounds and hence the reaction predominantly proceeds in the aqueous medium, which accounts for the observed marginal effect.

Scheme-I
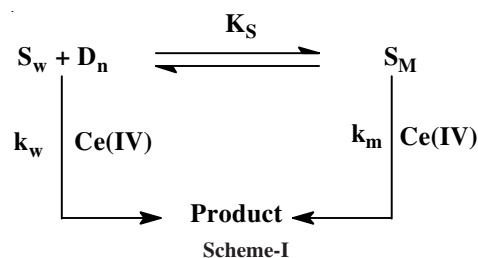
Here D$_n$ represents the micellized surfactant, subscripts w and m denote aqueous and micellar pseudo-phases respectively and k_w and k_m are the first order rate constants in the respective phases [45]. The substrate S may be either MEA or TEA and K$_S$ is the binding constant of the substrate with the micelle. The observed first order rate constant is given by

$$k_\psi = \frac{k_w + k_m K_S[D_n]}{1 + K_S[D_n]} \quad (1)$$

which can be modified to

$$\frac{1}{k_\psi - k_w} = \frac{1}{k_m - k_w} + \frac{1}{(k_m - k_w)K_S[D_n]} \quad (2)$$

When the kinetic data are fitted into eqn 2 linearity in the plot of $(k_\psi - k_w)^{-1}$ *versus* $[D_n]^{-1}$ $(= ([NaLS]-CMC)^{-1})$ is observed (Fig. 2) implying that the model is adequate to explain the oxidation of both the amino alcohols in micellar medium. From the intercept values of such plots, the micellar rate constants (k_m) are computed at different temperatures and the values are presented in Table-2. The rate constant k_m in pure micellar phase is then used to calculate the entropy of activation (Table-2). A comparison of $\Delta S^\#$ values for aqueous and micellar medium shows that entropy of activation in pure micellar phase is less negative. This indicates that the intermediate complex is relatively less stable and undergoes easy fragmentation in micellar phase resulting in products.

The ratio of intercept to the slope of the plot obtained using eqn. 2 yields the binding constant of the substrate MEA with
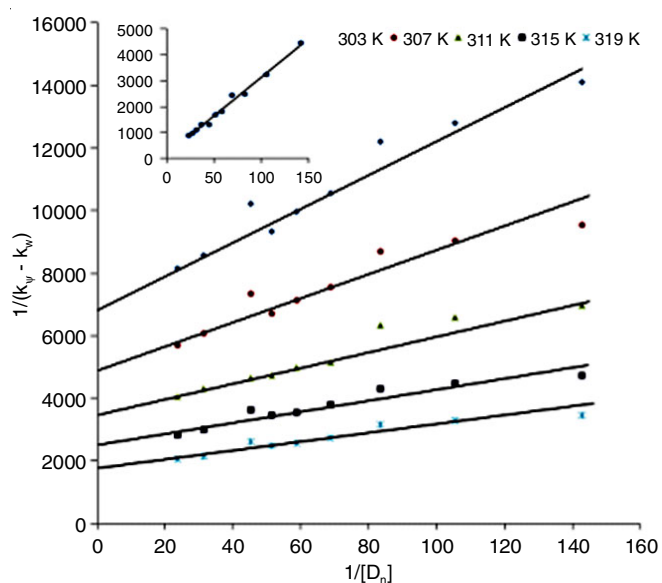
Fig. 2. Plot of $1/(k_\psi - k_w)$ against $1/[D_n]$ for the oxidation of MEA by Ce(IV) at different temperatures. The inset shows the same plot for TEA at 303 K

TABLE-2
RATE CONSTANT (k_m), BINDING CONSTANT (K_S), ACTIVATION PARAMETERS (ΔE & $\Delta S^\#$) AND TRANSFER FREE ENERGY ($-\Delta\mu^o$) FOR THE PURE MICELLAR PHASE OXIDATION OF MEA BY Ce(IV)

Temp. (K)	$10^4 \times k_m$ (mol^{-1} dm^3 s^{-1})	K_S	$-\Delta S^\#$ (JK^{-1} mol^{-1}) (Micellar medium)	$-\Delta S^\#$ (JK^{-1} mol^{-1}) (Aqueous medium)	$10^{-3} \times \Delta\mu^o$
303	2.038	114.51	-110.95	-133.99	22.06
307	2.881	116.92	-110.96	-133.72	22.40
311	4.050	125.93	-110.93	-133.41	22.89
315	5.563	126.90	-113.68	-134.09	23.20
319	7.508	130.91	-108.76	-133.83	23.58

Energy of activation, ΔE (kJ mol^{-1}): Aqueous medium = 60.92, Micellar medium = 64.56.

the micelle. The K_S data in Table-2 indicate that the binding constant increases with increase in temperature. Transfer free energy [46] per mole of the solute from water to the micellar phase is calculated by the relationship

$$\Delta\mu^o = \mu_w^o - \mu_m^o = RT \ln (55.5 \text{ K}) \tag{3}$$

where K is equal to the binding constant K_S. $\Delta\mu^o$ values computed by the above equation for monoethanolamine are also given in Table-2. The high values of $\Delta\mu^o$ indicate that amino alcohol is involved in hydrophobic interaction with the micelle in its core [47]. The positively charged oxidant species are expected to be oriented on the outer surface of the micellar phase due to electrostatic attraction. The differential orientation of the reactant species in the same micellar phase explains the cause of slow acceleration in the rate of reaction.

A glance at the kinetic data indicates that for both MEA and TEA, the rate constant values in pure micellar medium increases approximately three to four times compared to that in aqueous medium. Further, the micellar binding constant of the substrate and the transfer free energy are moderately large. Relative basicity of the amino component of the two amino

alcohols (MEA > TEA) is reflected in their decreasing order of K_S and $\Delta\mu^o$ values (Table-3). The basicity of the amino group drives the amino alcohol away from the micellar head; rather it is attracted to the core of the micelle. The high $\Delta\mu^o$ value is indi-cative of it and together with the high K_S value it probably means deep penetration into the core of the micelle. The oxidant, on the other hand, resides in the stern layer or at best at the micellar head. This separation between the substrate and the oxidant on their preference for hydrophobic or electrostatic attraction suppresses micellar acceleration of the rate, although both are present at the micellar surface and hence a large acceleration is otherwise expected.

TABLE-3
COMPARISON OF MICELLAR BINDING CONSTANT AND TRANSFER FREE ENERGY OF MEA AND TEA AT 303 K

Amino alcohol	$10^4 \times k_w$	$10^4 \times k_m$	K_S	$\Delta\mu^o$ (kJ mol^{-1})
MEA	0.541	2.038	114.51	22.06
TEA	12.09	37.09	14.63	16.87

Conclusion

The oxidation kinetics of amino alcohols by cerium(IV) in surfactant media was investigated. Anionic surfactant, sodium lauryl sulphate (NaLS) was found to affect the reaction rate. Upto the CMC of NaLS, the rate of oxidation reaction increases with increasing NaLS concentration. In post cmc region, the rate constant falls rapidly showing a maximum in k_ψ-[NaLS] profile for all the amino alcohols. The rate constant increases slowly with further rise in NaLS concentration for both mono-ethanolamine and triethanolamine after maximum. The kinetic data computed using various models reveals that there is consi-derable rise in rate constant in pure micellar medium compared to that in aqueous medium for both monoethanolamine and triethanolamine.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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