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ORIGINAL CONTRIBUTION

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Effect of anionic and cationic micelles on the oxidation of D-glucose by cerium(IV) in presence of H_2SO_4

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Abstract The influence of surfactants (anionic and cationic) on the reactivity of the redox couple cerium(IV) and D-glucose was examined spectrophotometerically. Various kinetic parameters have been determined in the absence and presence of surfactants. The kinetics were followed by monitoring the disappearance of the absorbance of cerium(IV) at 385 nm. The reaction obeyed first-order kinetics with respect to [D-glucose] in both media. No effect of anionic micelles of sodium dodecyl sulfate (SDS) was observed due to electrostatic repulsion between the negative head group of SDS and reactive species of cerium(IV) ($Ce(SO_4)_3^{2-}$). A twofold increase in the oxidation rate was observed in the presence of cationic micelles of cetyltrimethylammonium bromide (CTAB). The observed catalytic role has been analyzed in terms of the Menger–Portnoy model. The effects of various inorganic salts (Na_2SO_4, $NaNO_3$ and NaCl) were also studied in micellar media.

Keywords Anionic micelles · Cationic micelles · D-Glucose · Oxidation · Cerium(IV) · Surfactant · Catalysis

Introduction

Though the use of surfactant structures to alter or enhance reaction rates has been known for several decades [1], its use in the control of reaction pathways is a fairly recent development. Of widespread interest is the use of ionic surfactants to facilitate charge separation and retard back reactions in sensitized photochemical generation of hydrogen from water [2]. Micellar effects upon reaction rates and equilibria have generally been discussed in terms of the pseudophase model, and this approach will be followed here. The model aids in the interpretation of the catalytic activity of functionalized micelles used as models for enzymatic sites [3] and is applicable to the effects of reverse micelles, microemulsions, and vesicles on reaction rates and equilibria. Micellar catalysis and inhibition have received considerable attention in view of the analogies drawn between micellar and enzyme catalysis [4–7]. Micelles increase the rate of bimolecular reactions by concentrating both the reactants at their surfaces.

Mechanistic work on micellar effects is typically done using reactant concentrations much below those required for normal preparative work. Micellar solutions, in some cases, have proved superior to organic solvents as reaction media to obtain better yield. The studies of chemical reactions in micellar media can provide understanding about the reactions which take place at the interface [1, 8–11]. Electron transfer processes in micellar systems can be considered as models to gain insight into electron transport occurring in biological phenomena.

Cerium(IV) is used as an oxidant not only in analysis, but also in synthetic organic chemistry including carbohydrate chemistry. The hydrated ion $[Ce(H_2O)_n]^{4+}$ is a strong acid, which undergoes hydrolysis and polymerization. Kinetic and mechanistic studies of cerium(IV) oxidation of monosaccharides abound [12–15], but micellar effect on such redox systems has not been studied so far. We have now initiated a systematic study on the effect of organized assemblies, such as micelles, on the metal ion oxidation of carbohydrates [16–21], whereby the observed catalysis has been found to be dependent on the nature of the reactant species and surfactant type (cationic, anionic, nonionic). Many factors may account for the rate enhancement of a chemical reaction when the reactants are incorporated into or onto a micelle: approximation, electrostatic, hydrophobic, hydrogen bonding and medium effects. Thus, to understand the role of these factors on the metal ion oxidation of aldohexoses, we present here the effect of anionic and cationic micelles on cerium(IV) oxidation of D-glucose. Though D-glucose–cerium(IV) reaction kinetics has been studied on many occasions [14, 15, 22–24], this study appears to be the first on the effect of surfactants and added electrolytes.

Experimental

Materials

Standard solutions of D-glucose (99%, Merck, India) and ammonium ceric nitrate (99%, Qualigens, India) were prepared in doubly distilled deionised water. Cerium(IV) solution was standardized by titration and kept in a dark bottle. All other reagents like sodium dodecyl sulfate (99%, Sigma-Aldrich, USA), cetyltrimethylammonium bromide (99%, BDH, England), sodium nitrate (99%, Merck, India), sodium chloride (99.9%, BDH, India), sodium sulfate (98%, Merck, India) and sulfuric acid (98%, Merck, India) were used without further purification.

Kinetic measurements

Known volumes of both cerium(IV) in H_2SO_4 and D-glucose, in separate containers, were placed in a thermostat maintained at the desired temperature (±0.1 °C) for sufficient time to attain thermal equilibrium. Required volume of the D-glucose solution was then added to the acidic cerium(IV) solution and zero time noted when half of the D-glucose solution had been added. D-glucose was always in excess (ca. ten times) of cerium(IV) solution in order to maintain pseudo-first-order conditions. The progress of the reaction was followed at 385 nm by monitoring the absorbance changes of cerium(IV) with a Bausch & Lomb Spectronic-20 Spectrophotometer. For this purpose, aliquots of the reaction mixture were taken out at definite time intervals and cooled in an ice bath to quench the reaction before each measurement. The pseudo-first-order rate constants were estimated from the linear parts of the plots of log(absorbance) versus time by carrying out reactions up to 80% completion. The same procedure was used to evaluate the pseudo-first-order rate constants ($k_{\psi 1}$ or $k_{\psi 2}$, s^{-1}) in presence of surfactants.

Free radical detection

Presence of free radicals in the reaction mixtures was tested by adding acrylonitrile. Addition of the monomer solution (30%) to reaction mixture containing $[Ce(IV)]_T = 1.0 \times 10^{-3}$ mol dm^{-3}, [D-glucose]$_T = 4.0 \times 10^{-2}$ mol dm^{-3} and $[H_2SO_4]_T = 1.83$ mol dm^{-3} at 40 °C led to the appearance of a precipitate of a white polymeric product. The positive response indicated in situ generation of free radicals in the oxidation.

Stoichiometry

Several reaction mixtures with [Ce(IV)] > [D-glucose] ([oxidant] = 5.0×10^{-4} mol dm^{-3}:[reductant] = 0.5–4.5×10^{-4} mol dm^{-3}) at fixed [H^+] (= 1.83 mol dm^{-3}) were prepared. After completion of the reactions, the unconsumed oxidant was estimated spectrophotometerically. The consumption ratio, assuming that the D-glucose was totally consumed under these conditions, was found to be 2:1 (cerium(IV):D-glucose). However, due to the autoacceleration nature of the reaction (vide supra), the exact stoichiometry equation and products formed are difficult to predict.

Results and Discussion

General consideration

It is known that monosaccharide(s) oxidation by cerium(IV) proceed via the formation of a complex involving equatorial-OH of the reductant [14, 15, 24, 25]; this being a characteristic of common redox processes [16–21, 26]. It

Table 1 Dependence of pseudo-first-order rate constant on [Ce(IV)], [D-glucose] and [H_2SO_4] for the oxidation of D-glucose by cerium(IV) at 40 °C

10^3 [Ce(IV)] (mol dm^{-3})	[H_2SO_4] (mol dm^{-3})	10^2 [D-glucose] (mol dm^{-3})	Noncatalytic $10^4 k_{obs1}$(s^{-1})	Autocatalytic $10^4 k_{obs2}$(s^{-1})
0.2	1.83	4.0	6.1	Not observed
0.4			2.7	Not observed
0.6			2.2	Not observed
0.8			1.1	1.9
1.0			1.1	1.8
1.2			0.8	1.8
1.4			0.8	1.7
1.0	0.36	4.0	1.0	3.2
	0.73		1.0	3.8
	1.10		1.2	2.8
	1.47		0.9	1.9
	1.83		1.1	1.8
	2.20		0.9	Not observed
	2.57		0.8	Not observed
	2.94		0.7	Not observed
	3.30		0.6	Not observed
	3.67		0.5	Not observed
1.0	1.83	2.0	0.4	1.0
		3.0	0.8	1.4
		4.0	1.1	1.8
		5.0	1.3	2.4
		6.0	1.8	3.0
		7.0	1.9	3.5
		8.0	2.3	4.4
		9.0	2.5	4.7

should be emphasized here that before examining the effects of the two surfactants, namely, CTAB and SDS, on the D-glucose–cerium(IV) reaction, studies were also made without the addition of surfactant. No electrolyte (to maintain ionic strength) was used since Tondre and coworkers, in their pioneering work [27, 28], have advised avoiding the use of even buffer solutions to maintain the pH of micellar solutions.

Reaction in absence of surfactants

The oxidation kinetics of D-glucose by cerium(IV) was investigated at several initial [Ce(IV)], [D-glucose], [H_2SO_4] and temperatures. The plots of log(absorbance) versus time (Fig.1) were nonlinear, indicating that the kinetics was proceeding in two stages. The most interesting feature of this reaction is the autocatalysis, due to the catalytic role of one of the oxidation products. The pseudo-first-order rate constants were calculated for both the steps (noncatalytic and autocatalytic) from these plots (Table 1). The rate constants increased with increase in [D-glucose] (Fig. 2 and Table 1). The plot of log k_{obs} versus log [D-glucose] was linear with a slope of 1.0 indicating a first-order dependence on [D-glucose]. The k_{obs} values obtained at five different temperatures are listed in Table 2. The values of activation parameters were calculated using Arrhenius and Eyring equations (Table 2).

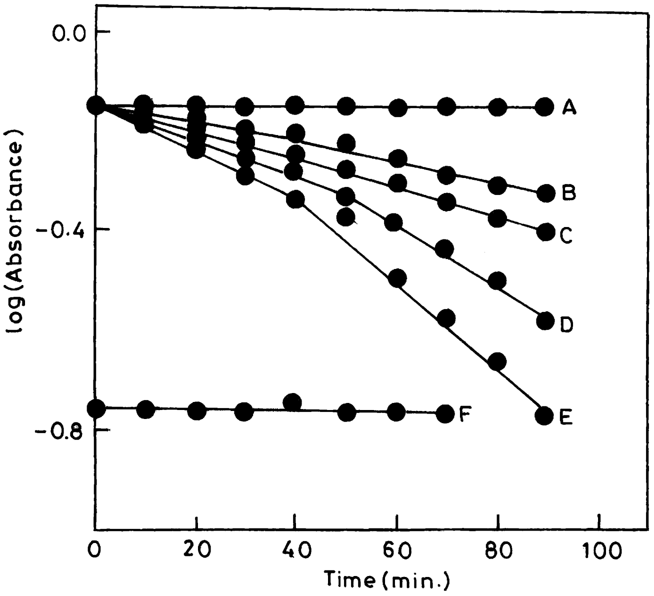
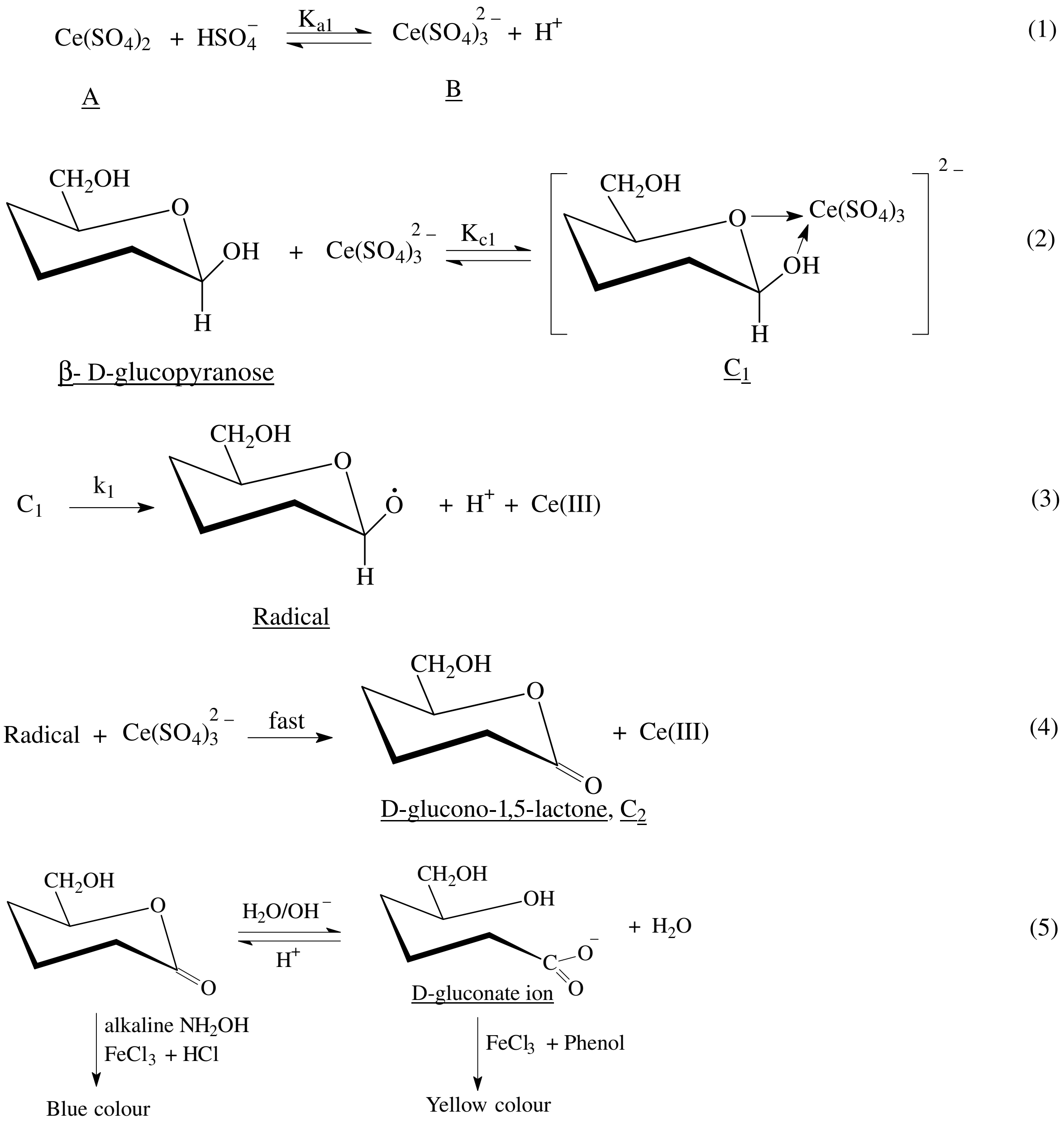


Fig. 1 Plots of log(absorbance) versus time as a function of [D-glucose]. Reaction conditions: [Ce(IV)] = 1.0 × 10^{-3} mol dm^{-3}, [D-glucose] = 0.0 **A**, 2.0 **B**, 3.0 **C**, 5.0 **D**, 6.0 **E** and 4.0 × 10^{-2} mol dm^{-3} **F**, [H_2SO_4] = 1.83 mol dm^{-3} **A**–**E** and 0.0 mol dm^{-3} **F**, temperature = 40 °C

At different $[H_2SO_4]$ (assuming $[H_2SO_4] = [H^+]$), in the range (0.36–3.67 mol dm^{-3}) at fixed [D-glucose]$(= 4.0 \times 10^{-2}$ mol dm^{-3}), [Ce(IV)] $(= 1.0 \times 10^{-3}$ mol dm^{-3}) and temperature (= 40 °C), the rate remains almost unchanged with increase in $[H_2SO_4]$ (≤ 1.83 mol dm^{-3}). Further increase in $[H_2SO_4]$ results in a decrease in k_{obs1}values (Table 1, also see Fig. 3 for graphical representation). Similar observations of decreasing k at higher $[H_2SO_4]$ were noted earlier by Sen Gupta et al. [15]. The inhibition of rate by the addition of H_2SO_4 may be explained as due to the removal of reactive species of cerium(IV) either by shifting equilibrium (1) to the left side or by protonation of the reactive species,$Ce(SO_4)_3^{2-} + H^+ \rightleftharpoons HCe(SO_4)_3^-$. Due to the involvement of a large number of protonic equilibria among cerium(IV) and H_2SO_4 [29–33], the exact dependence on $[H^+]$ cannot be made. The effect of adding sulfate ions was also studied. The values of $k_{obs1}(\times 10^4)$ were 1.1, 1.2, 1.5, 1.6, and 1.7 s^{-1} at $[SO_4^{2-}]$ values of 0.0, 1.0, 2.0, 4.0, 5.0 $\times 10^{-4}$ mol dm^{-3}, respectively. The increase is a definite indication of the involvement of a sulfato complex of cerium(IV) as the reactive species.

On the basis of observed results (first-order with respect to oxidant and reductant), and previous observa-



Scheme 1

Table 2 Pseudo-first-order rate constants and activation parameters for the oxidation of D-glucose by cerium(IV) in absence and presence of CTAB (= 50.0 × 10^{-4} mol dm^{-3})

Temperature(°C)	Aqueous[a]		Micellar[a]	
	$10^4\,k_{obs1}$ (s^{-1})	$10^4\,k_{obs2}$ (s^{-1})	$10^4\,k_{\psi 1}$ (s^{-1})	$10^4\,k_{\psi 2}$ (s^{-1})
30	0.3	Not observed	0.6	Not observed
35	0.9	1.5	1.2	1.9
40	1.1	1.8	1.7	3.6
45	2.0	6.1	2.7	7.3
50	3.3	7.6	4.3	9.9
Parameters				
Ea (kJ mol^{-1})	92 (0.989)[b]	89 (0.928)	88 (0.992)	76 (0.968)
$\Delta H^{\#}$ (kJ mol^{-1})	89	86	85	73
$\Delta S^{\#}$ (J K^{-1} mol^{-1})	−54	−63	−84	−86
$\Delta G^{\#}$ (kJ mol^{-1})	106	105	111	100

[a] [Ce(IV)] = 1.0 × 10^{-3} mol dm^{-3}, [H_2SO_4] = 1.83 mol dm^{-3}, [D-glucose] = 4.0 × 10^{-2} mol dm^{-3}

[b] The values of linear regression coefficients (r) are given in parentheses

tions of ca. 93% of the cerium(IV) being present as $Ce(SO_4)_3^{2-}$ in 1N H_2SO_4 [30], the Scheme 1 mechanism applies.

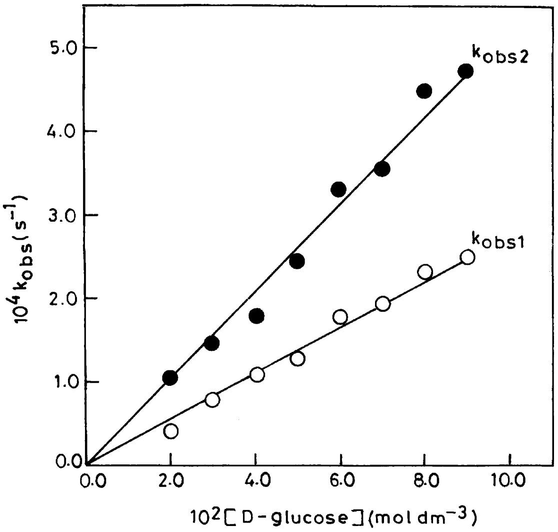


Fig. 2 Plots of k_{obs1} and k_{obs2} versus [D-glucose]. Reaction conditions: Same as in Fig. 1

The rate law for this reaction scheme is

$$k_{obs1} = \frac{k_1 K_{c1}[\text{D - glucose}]}{([H^+]/K_{a1}[HSO_4^-] + 1)} \qquad (6)$$

The equilibrium in Eq.1 lies well to the right, the inequality $1 \gg [H^+] / K_{a1}[HSO_4^-]$ will exist and the rate law, Eq. 6, reduces to Eq. 7, which explains the experimental results:

$$k_{obs1} = k_1 K_{c1}[\text{D - glucose}] \qquad (7)$$

that is, first-order dependence of [D-glucose] and independent on [H_2SO_4]. Interestingly, the second step oxidation (autocatalytic reaction path) is not a true reaction path for the oxidation of D-glucose by cerium(IV). It may be a mixture of the rates of D-glucose and its oxidation products (lactone and aldonic acid) (Scheme 2).

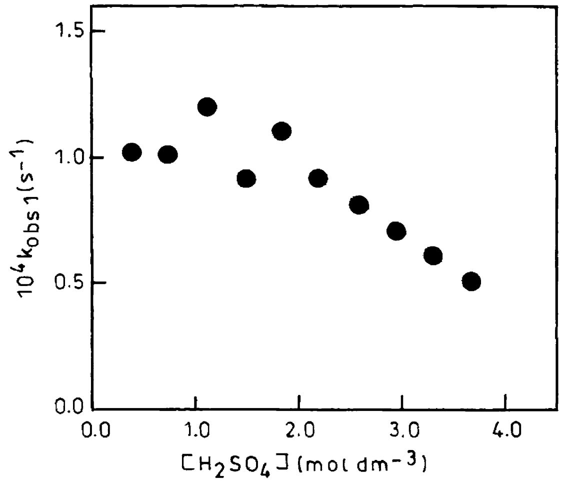


Fig. 3 Plot of k_{obs1} versus [H_2SO_4]. Reaction conditions: [Ce(IV)] = 1.0 × 10^{-3} mol dm^{-3}, [D-glucose] = 4.0 × 10^{-2} mol dm^{-3}, temperature = 40 °C

Thus, it is concluded that under the present kinetic conditions, the exact stoichiometry and product analysis cannot be predicted and the exact dependence of k_{obs2}on [D-glucose] cannot be estimated.

Reaction in the presence of surfactants

The effect of both anionic SDS and cationic CTAB surfactants on the reaction rate was investigated. The data depicted in Fig. 4 show that the anionic surfactant does not affect the rate of oxidation, which is not surprising. The negative head groups of SDS micelle

$$C_2 + Ce(IV) \longrightarrow Ce(III) + \text{2,3,4,6-tetrahydroxy-5-keto-hexanoic acid } (C_3) \qquad (8)$$

$$C_3 + Ce(IV) \longrightarrow Ce(III) + CO_2 + \text{other products} \qquad (9)$$

Scheme 2

simply repel the anionic species of cerium(IV). On the basis of these observations our conclusion that, out of various sulfate species of cerium(IV), only those with negative charge are the reactive species (i.e., $Ce(SO_4)_3^{2-}$, see Eq. 1) seems logical.

Cetyltrimethylammonium bromide, which produces cationic micelles in aqueous medium, has been found to increase the rate constants (Fig. 4). The catalytic activity of CTAB is due to association of the negatively charged species of cerium(IV) into or on the surface of the cationic micelles. In order to confirm the mechanism in micellar medium vis-à-vis aqueous medium, several kinetic runs were carried out at 50.0×10^{-4} mol dm^{-3} CTAB with varying experimental conditions of [Ce(IV)], [D-glucose], [H_2SO_4] and temperature. The results are given in Table 3. Just like aqueous medium, the same first-order dependence each in [D-glucose] and [Ce(IV)] are observed. Thus, the mechanism operative in the aqueous medium is also being followed in the CTAB micellar medium too. Activation energy values recorded in Table 2 clearly indicate that the CTAB acts as catalyst and provides a new reaction path with lower activation energies (as k_ψ does not represent a single elementary step, a complete account for all the factors that influence $\Delta H^\#$ and $\Delta S^\#$ is not possible).

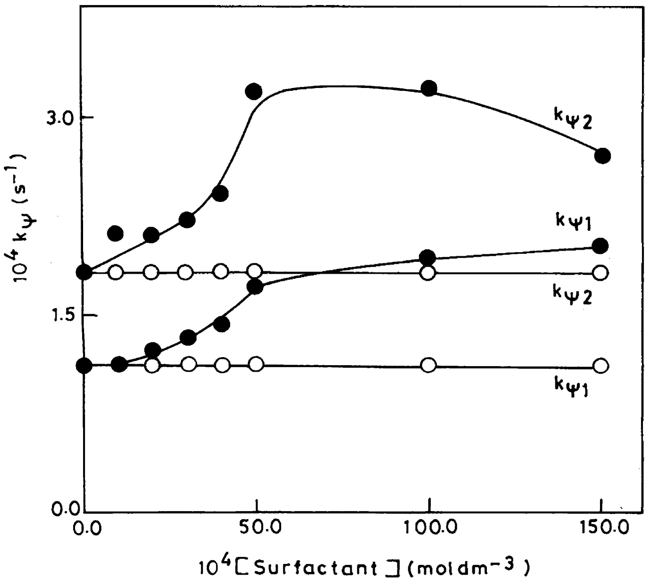


Fig. 4 Plots of rate constant versus [surfactant] (SDS ○, CTAB ●). Reaction conditions: [Ce(IV)] = 1.0×10^{-3} mol dm^{-3}, [D-glucose] = 4.0×10^{-2} mol dm^{-3}, [H_2SO_4] = 1.83 mol dm^{-3}, temperature = 40 °C

$$\begin{tikzcd}
(Ce(SO_4)_3)_w^{2-} \arrow[r, "k_w"] \arrow[d, shift left, "+D_n"] & \text{product} \qquad (10) \\
(Ce(SO_4)_3)_m^{2-} \arrow[r, "k_m"] \arrow[u, shift left, "K_S"] & \text{product} \qquad (11)
\end{tikzcd}$$

Scheme 3

The kinetic results of the effect of varying [CTAB] on the rate of reaction (Table 4, Fig. 4) can be explained by means of the pseudophase model proposed by Menger and Portnoy [5], which takes into consideration solubilization of only one reactant into the micellar phase (Scheme 3), where [D_n] = [CTAB]$_T$–cmc, k_w and k_m are, respectively, the pseudo-first-order rate constants in aqueous and micellar pseudophases. The observed rate law and Scheme 3 yield Eq. 12 which can be rewritten as Eq. 13. Accordingly, a plot of $1/(k_w–k_{\psi 1})$ versus

$$k_{\psi 1} = \frac{k_w + k_m K_S[D_n]}{(1 + K_S[D_n])} \qquad (12)$$

$$\frac{1}{(k_w - k_{\psi 1})} = \frac{1}{(k_w - k_m)} + \frac{1}{(k_w - k_m)K_S[D_n]} \qquad (13)$$

1/[D_n] should be linear. This has been found to be so (Fig. 5). The values of K_S and k_m obtained from the slope and intercept of Fig. 5, respectively, were found to be 50.5 mol^{-1} dm^3 and 3.3×10^{-4} s^{-1} (under the experimental conditions of [Ce(IV)] = 1.0×10^{-5} mol dm^{-3} and [D-glucose] = 4.0×10^{-2} mol dm^{-3}, the cmc value of CTAB, determined conductometrically at 40 °C, was found to be 10.65×10^{-4} mol dm^{-3}). These values were used to recalculate rates ($k_{\psi cal1}$) under various experimental conditions which were found to be in good agreement with experimentally observed rates (Table 4).

All micellar-mediated reactions are concluded to occur in the Stern layer [34]. Micellar surfaces are water rich [35] (activity of the water at the surfaces of ionic micelles is not different from water activity in the aqueous pseudophase). Electrostatic and hydrophobic interactions play an important role in the incorporation/association of substrates into micelles. Many ionic species of cerium(IV) are believed to be present in the

Table 3 Dependence of pseudo-first-order rate constant on [Ce(IV)], [D-glucose] and [H_2SO_4] for the oxidation of D-glucose by cerium(IV) at 40 °C in micellar medium ([CTAB] = 50.0×10^{-4} mol dm $^{-3}$)

10^3 [Ce(IV)] (mol dm^{-3})	[H_2SO_4] (mol dm^{-3})	10^2 [D-glucose] (mol dm^{-3})	Noncatalytic 10^4 $k_{\psi 1}$(s^{-1})	Autocatalytic 10^4 $k_{\psi 2}$(s^{-1})
0.2	1.83	4.0	4.6	Not observed
0.4			3.3	Not observed
0.6			2.6	4.0
0.8			2.4	4.1
1.0			1.7	3.2
1.2			1.1	2.5
1.4			1.0	2.1
1.0	0.36	4.0	Turbidity	
	0.73		Turbidity	
	1.10		Turbidity	
	1.47		Turbidity	
	1.83		1.7	3.2
	2.20		1.7	3.4
	2.57		1.5	3.1
	2.94		1.7	3.5
	3.30		1.5	3.3
	3.67		1.7	3.5
1.0	1.83	2.0	1.1	1.9
		3.0	1.4	2.3
		4.0	1.7	3.2
		5.0	2.0	3.8
		6.0	2.4	4.6
		7.0	2.6	4.9
		8.0	2.7	5.5
		9.0	2.9	7.0

Table 4 Effect of [CTAB] on the pseudo-first-order rate constant for the of oxidation of [D-glucose] (= 4.0×10^{-2} mol dm^{-3}) by cerium (IV) (= 1.0×10^{-3} mol dm^{-3}) in H_2SO_4 (= 1.83 mol dm^{-3}) at 40 °C

10^4 [CTAB] (mol dm^{-3})	Noncatalytic 10^4 $k_{\psi 1}$(s^{-1})	10^4 $k_{\psi \mathrm{cal1}}$(s^{-1})	$k_{\psi 1} - k_{\psi \mathrm{cal1}}/k_{\psi 1}$	Autocatalytic 10^4 $k_{\psi 2}$(s^{-1})
0	1.1			1.8
10	1.1			2.1
20	1.2	1.2	0.00	2.1
30	1.3	1.3	0.00	2.2
40	1.4	1.4	0.00	2.4
50	1.7	1.5	0.11	3.2
100	1.8	1.9	0.05	3.2
150	2.0	2.0	0.00	2.7

aqueous H_2SO_4 medium [29–33]. The positive catalytic effect of CTAB micelles indicates that the chemically active species must be anionic which may associate through electrostatic interaction with the positive head group of CTAB micelles. Under our experimental conditions ([H_2SO_4] = 1.83 mol dm^{-3}), the anionic species of cerium(IV) present in the reaction mixture is $Ce(SO_4)_3^{2-}$ [29, 36]. The second reactant, D-glucose, has no hydrophobicity due to the presence of 5-OH groups. As the reaction proceeds through the formation of a complex (see Eq. 2), associated $Ce(SO_4)_3^{2-}$ may form complex C_1 at the juncture region [16–21] (see Scheme 4 of a possible arrangement). The complex, having negative charge, may now orient in a manner suitable for continuing the reaction.

In order to see the effect of added electrolytes, a series of kinetic runs were performed in presence of inorganic salts (NaCl, $NaNO_3$and Na_2SO_4). It is observed that, by and large, all the added electrolytes have an inhibitory effect (Fig. 6). To discuss the effect of added electrolytes, it is necessary to consider two important factors, i.e., micellar structure and exclusion of reagent from the micellar pseudophase. By including changes in the properties of micelles, salts and organic additives control the extent of substrate solubilization and counterion binding to micelles [37, 38]. Inert salts, especially the inorganic ones, and organic additives have generally been found to decrease the rates of reactions. The micellar catalyzed reactions can be inhibited or micellar inhibition suppressed by the addition of electrolytes to

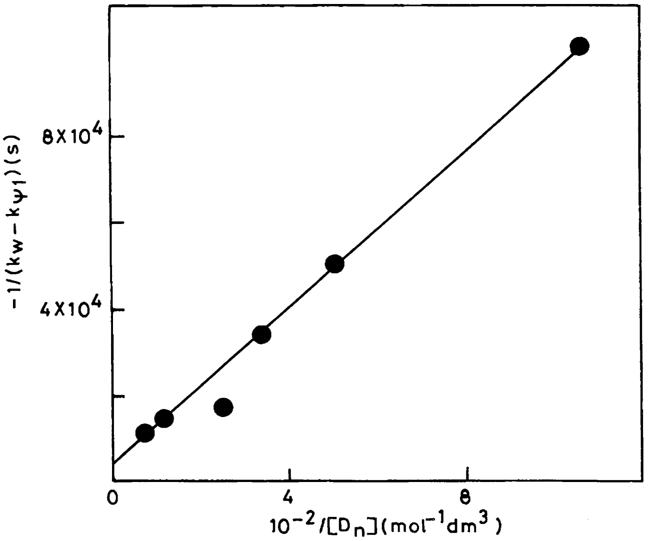
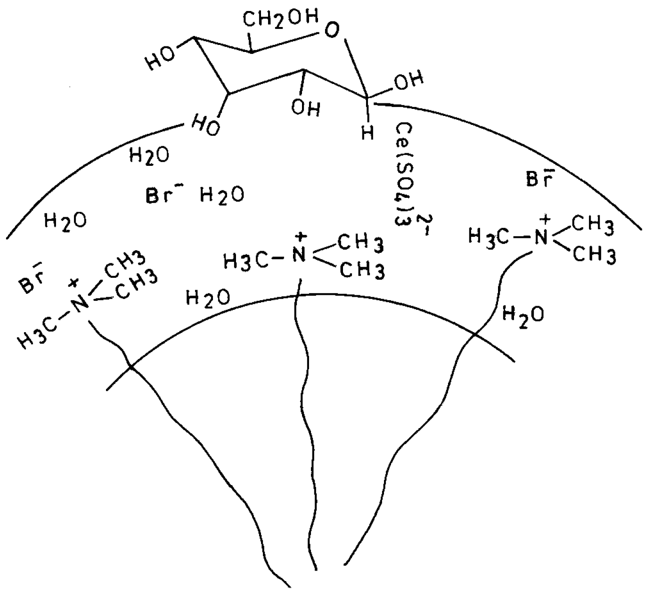


Fig. 5 Plot of $1/(k_w–k_{\psi 1})$ versus $1/[D_n]$ obtained in CTAB micellar medium. Reaction conditions: Same as in Fig. 4



Scheme 4

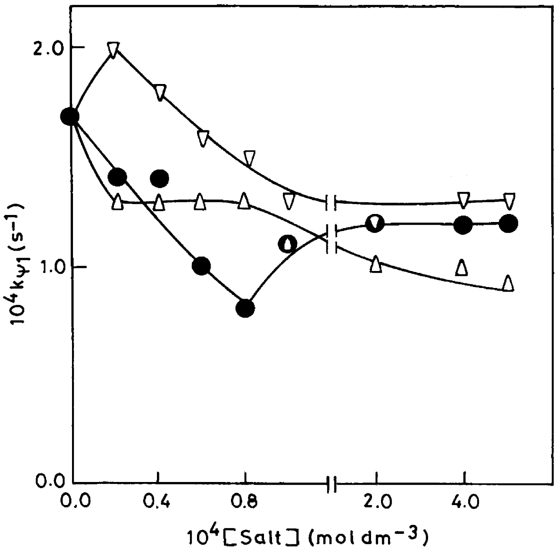


Fig. 6 Effect of $NaNO_3$ (▽), Na_2SO_4 (●) and NaCl (△) on $k_{\psi 1}$. Reaction conditions: same as in Fig.4 with $[CTAB] = 50.0 \times 10^{-4}$ - mol dm $^{-3}$

the medium [39]. Salt effect depends largely on the nature of counterion to the micelle. The counterions (Cl^-, NO_3^- and SO_4^{2-}) compete with $Ce(SO_4)_3^{2-}$ for the CTAB micelles so that inhibition typically increases. However, in presence of Na_2SO_4, an initial decrease followed by an increase is observed. As SO_4^{2-} contains a higher negative charge, its exclusion effect dominates in the beginning with a decrease in k_Ψ (Fig. 6). The kinetic results obtained at higher $[Na_2SO_4]$ (increase in k_Ψ) show that more than one factor may be involved. It will be recalled that, with the addition of Na_2SO_4, an increase in the rate constant resulted in the absence of surfactant, which was explained on the grounds of an increase in the active sulfato species: this effect may also become dominant in micellar media at higher $[Na_2SO_4]$.

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