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RESEARCH ARTICLE

HEXADECYLTRIMETHYL AMMONIUM BROMIDE EFFECT ON THE OXIDATION OF ETHYLENE GLYCOL BY PERIODATE

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Abstract

Kinetics of the oxidation of ethylene glycol (EG) by periodate (IO_4^-) has been studied in the presence of cationic surfactant cetyltrimethylammonium bromide (CTABr) using a conventional UV–visible spectrophotometric technique. The reaction is strongly inhibited by cationic micelle, (CTABr) with a progressive increase in CTABr concentration .This effect has been explained by the association of one of the reactants with the micelles leaving the other reactant in the bulk solution. The reaction follows first-order kinetics with respect to each [EG] and [IO_4^-] in micelle media and a mechanism which fitted the observed kinetic data was proposed. The micelle inhibiting oxidation reaction was shown to fit Arrhenius equation.

Keywords: Periodate, Ethylene glycol, CTABr, detergency, cationic

Introduction

Micellar catalysis has recently attracted a considerable interest in studies of hydrolysis [1-2], oxidation [3-9], nucleophile substitution reaction [10], and have been also recognized as a useful alternative route to improve analytical methodologies by shorten analysis time. Chemical reactivity in ionic colloidal self-assemblies (e.g., micelles, microemulsion droplets, and vesicles) has obtained importance owing to similarities in action with the enzymatic reactions. The similarities between the enzymatic reactions and the catalysis or inhibition by micelles include shape and size, polar surfaces, and hydrophobic cores. The micelles provide different microenvironments for different parts of the reactant molecules: that is, a nonpolar hydrophobic core can provide binding energy for similar groups while the outer charged shell can interact with the reactant polar groups [11].

Periodate has been used as oxidant for variety of substrate [12]. Oxidation with periodate is one of the most widely used reaction in organic chemistry [13]. It has the greatest application in the field of alcohol and carbohydrate chemistry [14]. Under controlled conditions, periodate will selectively oxidized 1, 2-diol, 1, 2- amino alcohols, 1, 2-hydroxyl aldehyde and ketones and various other grouping [15]. Periodate oxidation has a lot of advantages which is responsible for its being widely studied; (i) it can be applied in aqueous solution over a wide range of ${}_{P}^{H}$, (ii) it is quantitative and reasonably fast at room temperature[13-15].

Ethylene glycol is an organic compound primarily used as a raw material in the manufacture of polyester fibers and fabric industry. A small percent is used in industrial application like antifreeze formulation and other industrial product. The kinetic oxidation of ethylene glycol by various oxidizing agents have been investigated and were found to involve two electron transfers through the formation of a negatively charge cyclic intermediate.

The oxidation reaction of EG by periodate in micellar system remain however unexplored. In this work, kinetics and mechanism of the oxidation of EG through periodate in aqueous micellar solution of Hexadecyltrimethy ammonium bromide (CTABr) was undertaken in an effort to observed the effect of CTABr on the rate constant, substrate partitioning, change in critical micelle concentration (cmc) and determination of thermodynamic parameter.

Experimental

Sodium periodate (BDH, 99% pure) was used without further purification. Ethylene glycol (BDH) was purified by simple distillation. Hexadecyltrimethyl ammonium bromide (CTABr) was purified by recrystallization in Benzene/Ethanol (20:80) and dried in desiccator. All the solution were prepared with double distilled water.

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Kinetic measurement

The required volume of ethylene glycol and periodate were transfer to the reaction vessel, which is thermostated to the desired temperature. The kinetic data were obtained by monitoring the change in absorbance of periodate at absorption maximum ($^{a}{}_{\max}$= 225.4nm) as a function of time using SPS-100 UV-visible spectrophotometer. The concentration of ethylene glycol was kept in large excess over the concentration of periodate to achieved pseudo-first order conditions. The observed rate constant (k_{obs}) were obtained from slope of $\ln(A_t - A_\infty)$ versus time where A_t and A_∞ are the absorbance at a given time (t) and at infinity time respectively. The value of the pseudo-first order rate constant were reproducible within the experimental error.

Critical micelle concentration

The CMC values of the surfactant (CTAB) were measured in the presence and absence of reactants, (IO_4^- and EG). The CMC values were obtained from conductivity measurements and carried out with a digital conductivity meter (Jenway DDS – 307 Model). Temperature was varied between 20^0C and 50^0C. The CMC values were determined from plots of the specific conductivity versus surfactant concentration. The break points of nearly straight-line portions in the plot are taken as an indication of micelle formation, and this correspond to the CMC of surfactant. The CMC values of CTAB at different experimental conditions are summarized in Table 1a and 1b.

Result and Discussion

Evaluation of Observed Rate Constant (k_{obs}) (Micelle dependence)

The effect of micelles on the oxidation of [EG] by $[IO_4^-]$ was studied at [EG] = 1.790 and 3.85 x 10^{-3} mol dm^{-3}, $[IO_4^-]$ = 4.266 x 10^{-5} $moldm^{-3}$ and T = 25^0C. At fixed concentration of EG, IO_4^- and varying concentration of [CTABr], the observed rate constant in aqueous micelle (k_{obs}) increases slightly with increase in the concentration of CTABr from 4.683 to 5.028 x 10^{-3} s^{-1} with increase in [CTABr] from 0 to 1.829 x 10^{-4} $moldm^{-3}$. A plot of k_{obs} versus [CTABr] show a rate maximum at [CTABr] = 1.829 x 10^{-4} $moldm^{-3}$ (Fig. 1). A further increase in [CTABr] (above 1.829 x 10^{-4} $moldm^{-3}$) result in a decrease in the reaction rate compare to what is obtained in micelle free medium where k_{obs} increases geometrically. The $k_{.}$ -[CTABr] profiles (Fig. 2) at two different concentration of EG (1.790 and 3.580x10^{-3}mol/dm^{-3}) show that $k_{.}$ does not vary linearly with [CTABr] but decreases more rapidly at concentration below 9.2x10^{-4} mol/dm^3 the CMC of CTABr. After the CMC, the rate decrease in less rapid, approaching saturation at higher [CTABr] greater than CMC. Several models have been postulated to interpret the effect of the concentration of surfactant on the rate of the reactions. Pseudo-phase model suggested by Mengo and Bortany assumes that the substrate is distributed between water and micellar phases; hence, the overall rate will be the sum of the rates in water and in the micelles.

Catalytic effect occurs when both reactants bind to the micelle and react to give the product. However, when one of the reactants binds to the micelle while the other remains in the bulk solution, the reaction gets inhibited as EG binds to the micelle leaving periodate in the solution.

Observed Rate Constant (k_{obs}) Dependence on EG Concentration

The effect of [EG] was studied at constant [IO_4^-] (= 4.266 x 10^{-5}moldm^{-3}) and at four different [CTABr] concentration (= 0.914x 10^{-4}, 2.56 x10^{-4}, 7.316 x10^{-4} and 18.29 x10^{-4} mol.dm^{-3}) 25^{0}C. The observed rate constant (k_{obs}) on [EG] was examined. The profile obtained in Figure 3 show that the rate of reaction (k_{obs}) increases with increase in [EG] at fixed [CTABr] in aqueous solution according to the equation 1 but as [CTABr] increases, k_{obs} diminishes quickly till the concentration reaches the value of about 18.29 x 10^{-4} moldm^{-3} after which the rate constant becomes almost constant. Therefore, the inhibition effect of CTABr, which was observed in this reaction, can be explained by the fact that the ethylene glycol binds itself to CTABr leaving periodate in the bulk solution resulting in reducing the chance by which the two reactants can meet.

$k_{obs} = k_1[EG]$ ……………………………..[1]

Second Order Rate Constant Dependence on IO_4^- Concentration.

The dependence of second order rate constant ($k_{.}$) on [IO_4^-] at fixed [EG] with [CTABr] is shown in figure 4. The $k_{.} - [IO_4^-]$ profile show saturation at high [IO_4^-] for each fixed [EG] and [CTABr]. From Figure 4, $k_{.}$ decreases considerably as one approaches the cmc of CTABr (9.1x10^{-4}mol.dm^3), above the cmc, $k_{.}$ is essentially invariant with [IO_4^-].This observation show strong inhibition of pre-micellar activity due to the less bound of IO_4^- to the micelle. A comparative analysis show that the $k_{.}$ of oxidation of EG by the IO_4^- ion in the micellar medium decreases with increase in [CTABr] i.e the rate decreases with increase in [CTABr] .This is an indication that the substrate is less reactive when micelle is formed.

Effect of Temperature on Observed Rate Constant (k_{obs})

The effect of temperature on the second order rate constant were shown in figure 5. The $k_{.} - [CTABr]$ profiles were measured at constant substrate concentration and at each of the five temperature, 20, 25, 30, 35, and 40^{0}C. The [EG] was fixed at 3.580 x 10^{-3}mol.dm^{-3} and [IO_4^-] was fixed at 4.267x10^{-5}mol.dm^{-3}. A plot of $\ln(k_{.} / T)$ vs $(1/T)$ were linear (Fig.6). From the calculated intercept and slopes using Erying equation, activation parameters $\Delta G^*, KJ.mol^{-1}, \Delta H^*, KJmol^{-1}$, and $\Delta S^*, JK^{-1}mol^{-1}$ were obtained (Table 2). The linearity of the plots indicates that Arrhenius equation has been followed. The activation energies of the reaction in the absence/presence of CTABr have been calculated from the slopes of the straight lines and given in Table 2. The activation energy in the absence of CTABr is negative, which indicates that there must be at least one mechanistic step that possesses equilibrium constant, which decreases with raising the temperature. The negative activation energy can also be considered as an evidence for the involvement of free radicals in the mechanism of the reaction[16]. However, the presence of the surfactant in the reaction mixture increases the activation energy to a positive value to give another evidence for the inhibition effect of the micelles.

Effect of Substrate and Temperature on the CMC of CTABr

Temperature dependence of the cmc in the presence of [EG] and [IO_4^-] were shown in Table 3a 3b and 3c. Observation shows that the value of CMC increased with increase in temperature within the temperature range investigated. The effect of temperature on the CMC of surfactant in aqueous solution has been analyzed in terms of two opposing factors. First, as the temperature increases, the degree of hydration of the hydrophilic group decreases, which favours micellization, however, an increase in temperature also causes the disruptions of the water structure surrounding the hydrophobic group and this is unfavorable to micellization [17].

The plot of $\ln(cmc)$ vs $(1/T)$ show slight curvature as it has been reported by many researcher. The free energy ΔG_m^0, enthalpy ΔH_m^o and entropy ΔS_m^o of micellization were obtained from the slope and intercept using equation 2 a, b, and c as tabulated in Table 4.

$$\Delta G_m^0 = (2 - \sim) RT \ln \text{¡}_{CMC} \text{……………………[2a]}$$

$$\Delta H_m^0 = -RT^2[(2 - \sim)(\partial \ln \text{¡}_{CMC} / \partial T)_P - \ln \text{¡}_{CMC}(\partial \sim / \partial T)] \text{…………………..[2b]}$$

$$\Delta S_m^0 = \frac{\Delta \mathrm{H}_m^0 - \Delta G_m^0}{T} \text{…………………[2c]}$$

The free energy ΔG increases with increase in [CTABr]. This implies increase in energy barrier and hence reduction in rate which is also in agreement with the observed inhibition by surfactant.

The negative entropy value indicates that the transition state of the reactant become more rigid and order in the presence of micelle in respect to free reactant. The entropy of activation show a large variation at low CTABr concentration (pre-cmc) which is in agreement with the observed relatively strong pre-micellar activity. The high value of free energy of activation and enthalpy ΔH suggest that the transition state is highly solvated.

At low concentration (pre-cmc), entropy of activation is very low because the steric effect is less due to the fact that there are few monomer in the pre-micelle. The pre-micelle activity is a common feature of many micellar catalyzed reaction. IO_4^- inhibit micellization of CTABr slightly more than EG from the comparative values of their enthalpy of micellization (Table 2). This is due to the fact that IO_4^- causes a reduction in surface potential of the CTA$^+$ micelle by reducing the repulsion between the monomer head group.

Mechanism of CTABr-Inhibiting Oxidation of EG by Periodate

Inhibition of the reaction of periodate ion with ethylene glycol in CTABr media, suggesting that: (i) IO_4^- ion is bound in the micellar phase by electrostatic interaction with EG predominantly in the bulk water phase (ii) EG is solubilized in the micellar phase (below the stern layer) by hydrophobic interaction with IO_4^- bound at the micellar surface.
Equilibrium step (Eq 3) involves the formation of the periodate-glycol adduct $[EG.IO_4^-]$.

$$\mathrm{EG} + \mathrm{IO_4^-} \underset{k_{-1}}{\overset{k_1}{\rightleftharpoons}} [\mathrm{EG.IO_4^-}]^- \text{……………………………………………[3]}$$

Another equilibrium step involves the binding of IO_4^- to the micelle

$$\mathrm{M^n} + \mathrm{IO_4^-} \underset{k_{-n}}{\overset{k_2}{\rightleftharpoons}} [\mathrm{M.IO_4^-}]^{\,n-1} \text{…………………………………………………[4]}$$

Where n is the charge on the micelle. The reaction product of $(2HCHO + IO_4^- + Micelle)$ are formed in two phase. In the bulk water phase where $[EG.IO_4^-]$ adduct goes to the product and the micellar phase to which EG diffuse to react with micelle-bound IO_4^-. The following equation are relevant to this reaction.

$$EG.IO_4^- \xrightarrow{k_.} \Pr oduct \text{……………………………………………………[5]}$$

$$M.IO_4^- + EG \xrightarrow{k_m} \Pr oduct \text{……………………………………………….[6]}$$

$k_{.}$ is the first order dissociation constant of the adduct in the bulk water and k_m is the second order rate constant in the micellar phase.

From equation 3 to 6;

$[E_0] = \sim + \text{¤}$ ……………………………………………………………………………………….[7]

$[P_0] = \quad + \text{¤} + \text{ª}$ ……………………………………………………………………………………….[8]

$[M_0] = \text{¥} + \text{ª}$ ……………………………………………………………………………………….[9]

$\text{¤} = k_1 \sim$ ……………………………………………………………………………………….[10]

$\text{ª} = k_2 \ \text{¥}$ ……………………………………………………………………………………….[11]

The rate of formation of the product can be written as

$Rate = k_{.}\, \text{¤} + k_m \text{ª} \sim$ ……………………………………………………………………………………….[12]

The observed rate constant k_{obs} is given by,

$$k_{obs} = \frac{Rate}{[P_0]} = \frac{k_{.}\, \text{¤}}{[P_0]} + \frac{k_m \text{ª} \sim}{[P_0]} \quad \text{……………………………………………………..[13]}$$

From equation 7 and 10;

$\text{¤} = k_1(([E_0] - \sim) \quad)$ ……………………………………………………………………………………….[14]

$$= \frac{\text{¤}}{k_1([E_0] - \sim)} \quad \text{……………………………………………………..[15]}$$

From equation 8 and 11;

$\text{ª} = k_2 \quad ([M_0] - \text{ª})$ ……………………………………………………………………………………….[16]

$$\text{ª} = \frac{k_2 \quad [M_0]}{1 + k_2} \quad \text{……………………………………………………..[17]}$$

Substituting ª and in equation 8 yield,

$$P_0 = \frac{\text{¤}}{k_1([E_0 - \text{¤})} + \text{¤} + \frac{k_2 \quad [M_0]}{1 + k_2} \quad \text{……………………………………………..[18]}$$

$$= \frac{\text{¤}}{k_1([E_0] - \text{¤})} + \text{¤} + \frac{k_2[M_0]\text{¤}}{k_2\text{¤} + k_1([E_0] - \text{¤})} \quad \text{………………………………………[19]}$$

$$= \frac{\text{¤}}{k_1([E_0] - \text{¤})} + \text{¤} + \frac{k_2[M_0]\text{¤}}{\text{¤}(k_2 - k_1) + k_1[E_0]} \quad \text{………………………………………[20]}$$

Rearranging this equation and neglecting ¤^2 term and higher power yield;

$$\text{¤} = \frac{k_1[P_0][E_0]}{1 + k_1[E_0] + k_1[P_0] + k_2[M_0] - (k_1 - k_2)P_0} \ldots\ldots\ldots\ldots\ldots\ldots\text{[21]}$$

From equation 6 we have;

$$\sim\, = E_0 - \text{¤} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\text{[22]}$$

Substitute in equation 21 we obtained;

$$\sim\, = [E_0](1 + k_1[E_0]) - (k_2 - k_1)[P_0] \ldots\ldots\ldots\ldots\ldots\ldots\text{[23]}$$

Substituting for   in equation 17 yield;

$$a = \left(\frac{k_2[M_0][P_0]}{1 + k_1[E_0] + k_2[M_0] - (k_2 - k_1)[P_0]}\right) / \frac{k_2[P_0]}{1 + k_1[E_0] + k_2[M_0] - (k_2 - k_1)[P_0]} \ldots\ldots\text{[24]}$$

$$a = \frac{k_2[M_0][P_0]}{1 + k_1[E_0] + k_2[M_0] + k_1[P_0]}$$

The product of ~ and   is given by;

$$\sim a = \frac{k_2[M_0][E_0][P_0].(1 - \frac{k_2[P_0]}{1 + k_1[E_0] + k_2[M_0] + k_1[P_0]}}{1 + k_1[E_0] + k_2[M_0] + k_1[P_0] - (k_2 - k_1)[P_0]} \ldots\ldots\ldots\ldots\ldots\ldots\text{[25]}$$

Assuming that

$$k_2[M_0] + 1 >> k_1([E_0] + [P_0]) \ldots\ldots\ldots\ldots\ldots\ldots\text{[26]}$$

$$\sim a = \frac{k_2[M_0][E_0][P_0].(1 - \frac{k_2[P_0]}{1 + k_2[M_0]}}{1 + k_2[M_0] - (k_2 - k_1)[P_0]} \ldots\ldots\ldots\ldots\ldots\ldots\text{[27]}$$

Using equation 26, equation 27 reduced to;

$$\text{¤} = \frac{k_1[P_0][E_0]}{1 + k_2[M_0] - (k_2 - k_1)[P_0]} \ldots\ldots\ldots\ldots\ldots\ldots\text{[28]}$$

The observed rate constant (k_{obs}) then become,

$$k_{obs} = \frac{k_{\mu} k_1 + k_m k_2[M_0](1 - \frac{k_2[P_0]}{1 + k_2[M_0]})[E_0]}{1 + k_2[M_0] - (k_2 - k_1)[P_0]} \ldots\ldots\ldots\ldots\ldots\ldots\text{[29]}$$

The second order rate constant in micellar medium ($k_{.}$)

$$k_{.} = \frac{k_{\mu} k_1 + k_m k_2[M_0](1 + \frac{k_2[P_0]}{1 + k_2[M_0]}}{1 + k_2[M_0] - (k_2 - k_1)[P_0]} \ldots\ldots\ldots\ldots\ldots\ldots\text{[30]}$$

Equation 29 is in agreement with the observed linear dependence of k_{obs} with [EG] at fixed [CTABr] and [IO_4^-]

Table 1a Variation of the concentration of CTABr with [EG] Concentration

[EG] x 10^4 (mol.dm^{-3})	Conc x 10^4 (mol.dm^{-3}) of CTABr
0.00	9.30
7.168	9.80
17.920	10.71
25.088	12.41
35.840	14.86

Table 1b Variation of CMC of CTABr with [IO_4^-] concentration

$[IO_4^-]$ x 10^5 (mol.dm^{-3})	Conc x 10^4 (mol.dm^{-3}) of CTABr
0.00	9.30
1.051	9.70
2.012	11.65
3.153	12.14
4.204	13.50

Table 2 Activation energy at different temperature in the presence and absence of CTABr

Temperature	Activation Energy	
	Absence of CTABr	Presence of CTABr
0	14.23	3.84
20	14.94	6.11
25	20.11	4.23
30	16.18	1.18
35	21.16	2.19
40	23.18	6.80

Table 3a Temperature dependence of the concentration of pure CTABr

Temperature (K)	Conc x 10^4 (mol.dm^{-3})
293	9.00
298	9.20
303	9.80
313	11.00
323	12.05

Table 3b Temperature dependence of the concentration of CTABr in the presence of [EG] = 3.580 x 10^{-3}M

Temperature (K)	Conc x 10^4 (mol.dm^{-3})
293	9.50
298	9.90
303	10.10
313	10.90
323	12.95

Table 3c Temperature dependence of concentration of CTABr in the presence of $[IO_4^-]$ = 4.204 x 10^{-5}M.

Temperature (K)	Conc x 10^4 (mol.dm^{-3})
293	9.20
298	9.50
303	9.70
313	10.40
323	11.60

Table 4 Thermodynamic parameters of micellization of CTABr and CTABr – Substrate mixture

	$\Delta H_m^0 \pm 0.074 (kJ.mol^{-1})$	$\Delta S_m^0 \pm 0.557 (J.mol^{-1}.k^{-1})$	$\Delta G_m^0 \pm 0.058 (kJ.mol^{-1})$
Pure CTABr	-8.106	30.923	-17.321
CTABr + EG	-7.460	32.500	-17.145
CTABr + IO_4^-	-5.892	38.101	-17.244

Fig. 1. Plot of k_{obs} vs [CTABr] for oxidation of [EG] by $[IO_4^-]$ = [4.266X10^{-5} mol.dm^{-3}] at 25^{0}C.

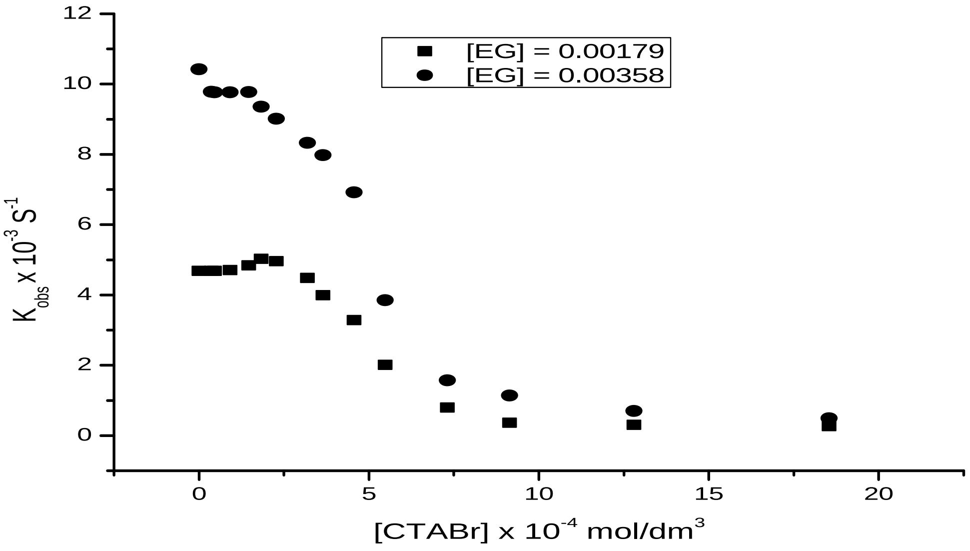


Fig. 2. Plot of K_{ψ} vs [CTABr] for oxidation of [EG] by $[IO_4^-]$ = [4.266X10^{-5} mol.dm^{-3}] at 25^{0}C.

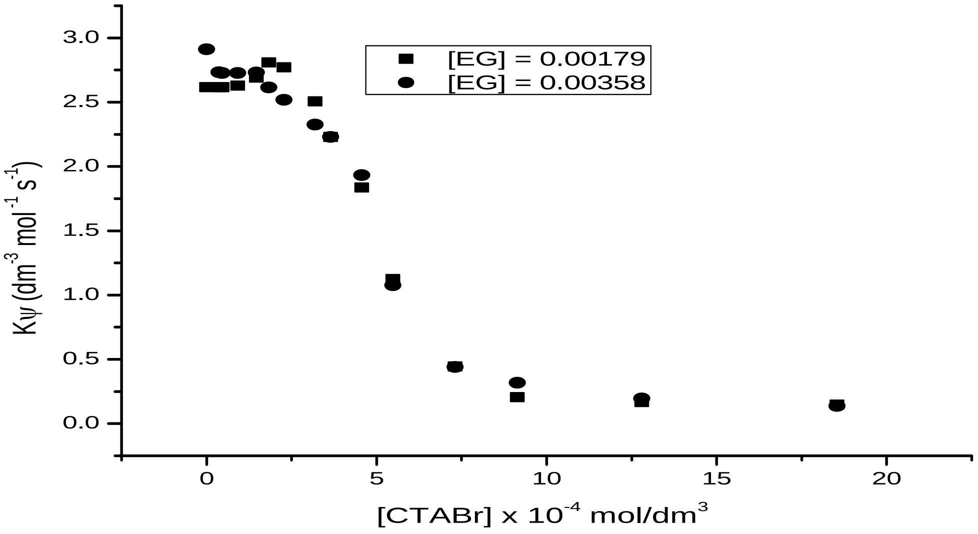


Fig. 3. Plot of k_Ψ vs $[IO_4^-]$ Reaction conditions [EG] = 3.580×10^{-3} mol. dm^{-3} at 25^0C

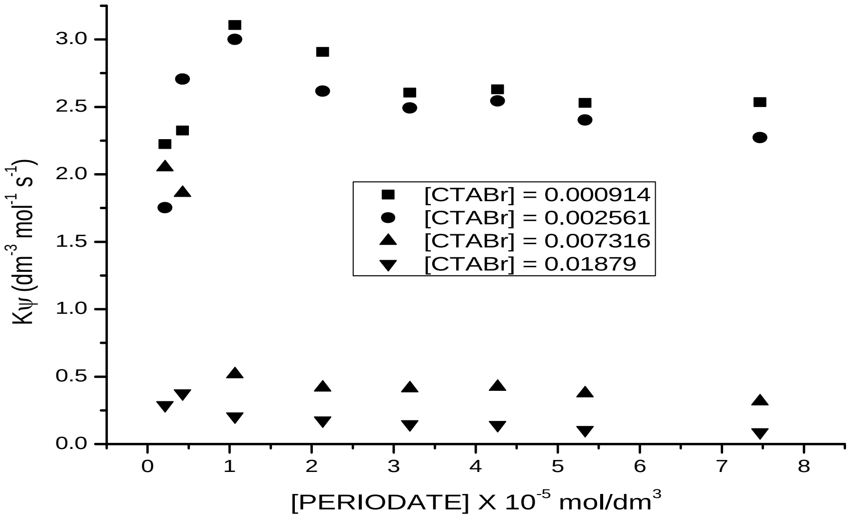
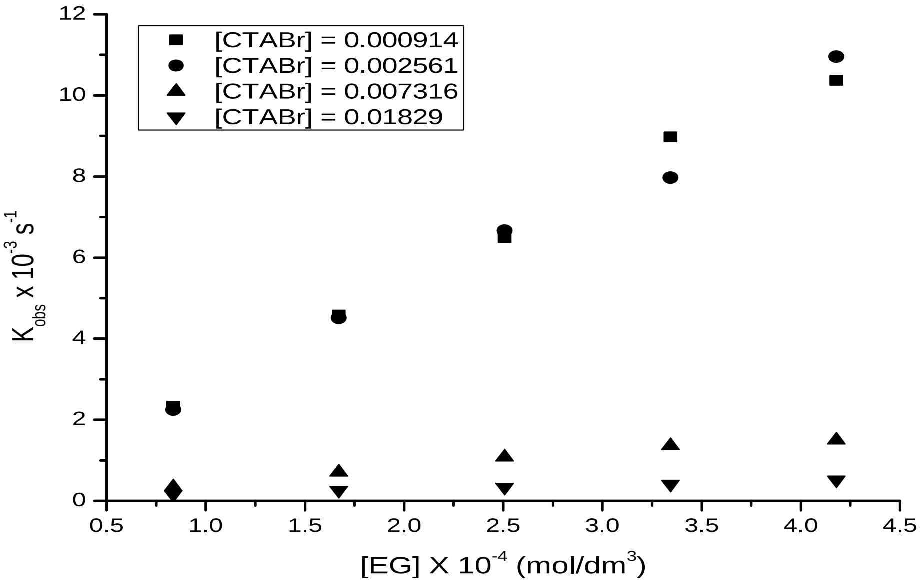


Fig. 4. A plot of kobs against [EG] concentration at 25^0C at various [CTABr] concentration $[IO_4^-]$ = 4.256×10^{-4} mol.dm^{-3}



Conclusion

The effect of CTABr micelle on the kinetic of oxidation of EG by periodate was studies. The activation parameters were evaluated using Arrhenius and Erying equation. The reaction show a first order reaction and CTABr micelles inhibit the rate of periodate oxidation of EG in aqueous solution. The observation was due to the favorable electrostatic binding of IO_4^- in the stern layer of the CTABr inhibiting the formation of $[EG.IO_4^-]$ intermediate adduct. There is a strong pre-micellar activity. The profile is highly structured due to the micellar evolution with respect to the cmc, size and number of monomer in each aggregate.

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