Micellar Effect on the Kinetics of Ethanol Oxidation by Potassium Permanganate in Acidic Medium

Dayo Felix Latona

Department of Pure & Applied Chemistry, Faculty of Basic and Applied Sciences, Osun State University, Osogbo, 210001, Nigeria
Corresponding author. E-mail address: dayo.latona@uniosun.edu.ng

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Abstract

The catalytic effect of sodium dodecyl sulphate (SDS) micelles on the oxidation of alcohols by potassium permanganate in an acidic medium was investigated using a double beam Unicam-1800 Schmadzu UV/Visible- spectrophotometer at λmax 525 nm under pseudo-first-order kinetics. The kinetic study followed a first-order dependence each on [$KMnO_4$], [Ethanol] and fractional order to [H^+]. The reaction was catalyzed by SDS at low concentration values and invariant at higher [SDS]. The catalytic effect may be attributed to the solubilization and incorporation of the reactants into the stern layer of the micelles and the inhibition at higher levels of SDS is attributed to the repulsion between the protonated oxidant accumulated on the surface of the micelle and the surfactant counter ions. The results are discussed in terms of the Pseudo-Phase Model as proposed by Menger-Portnoy and Piszkiewicz's positive cooperativity model. The activation parameters were obtained from Erying's equation as $\Delta H^{\#}$ 4.16 kJ mol^{-1}, $\Delta S^{\#}$ -0.198kJK^{-1} mol^{-1} and $\Delta G^{\#}$ 63.16 kJmol^{-1}. The negative $\Delta S^{\#}$ suggests an ordered transition state. The micellar rate constant, km and the binding constant Ks, were 7.45×10^{-3} s^{-1} and 40.35 mol dm^{-3} respectively. The binding constant indicates the existence of surfactant-substrate interaction and the value of n>1 suggests positive cooperativity. The study showed that electrostatic/hydrophobic interactions play a significant role in the micellar-influenced reaction and further affirmed the fact that all chemical reactions involving protonation are catalyzed by anionic surfactants.

Keywords: Kinetics, Sodium Dodecyl Sulphate (SDS), Oxidation, Potassium Permanganate, Ethanol

Introduction

Micelles are formed due to the aggregation of surfactant monomers in its thermodynamic stable equilibrium. They can be classified into cationic, anionic or neutral micelles depending on the constituent surfactants. Micelles are formed above the critical micelle concentration of the surfactant. (Hans-Friedrick & Andreas, 1978). Micellar effects on chemical reactions have received great attention recently (Menger & Portnoy, 1967). The influence of hydrophobic and electrostatic forces plays a vital role in micellar studies (Buton, 2006). The influence of sodium dodecyl sulphate (SDS) micelles on the oxidation of several organic substrates by different oxidants both in acidic and alkaline media has been investigated (Din, Hartani, & Khan, 2001; Perez-Benito & Rodenas, 1991). Sodium dodecyl sulphate micelles have been found to catalyse the addition-elimination interaction between o-toluidine and D-glucose (Dar, Andrabi, & Khan, 2006). The pre-micellar environment of SDS strongly inhibits the oxidation of D-fructose by diperiodatoargentate (Dar, Ilyas, Kumar, & Khan, 2006). Sodium dodecyl sulphate has been reported to inhibit the hydrolysis of Imine. The inhibition was attributed to the selective partitioning of the phenol form of the imine into the micellar pseudophase of Sodium dodecyl sulphate (Dash, Dash, & Panda, 1985). The hydrolysis of 3-hydroxy-carbofuran has also been reported to be inhibited by SDS (Astray et al., 2011). The catalytic effect of SDS on the oxidation of D-fructose by chromic acid in the presence of HClO4 has been reported (Kabir-ud-Din, Morshed, & Khan, 2002). Furthermore, SDS accelerates the oxidation of glycerol by picolinic acid-promoted hexavalent chromium (Ghose et al., 2012). Micelles have found great applications in modern chemistry reactions like Diels Alder (Mubofu & Engberts, 2007; Hunt & Johnson, 1991), redox (Rodriguez, delMarGraciani, Munoz, & Moya, 2000; Saha, Sarka, & Chowdbury, 2008), photochemical (Shi, Sigma, Ghosh, & Dabestani, 1997), decomposition (Brinchi et al., 2006), enzymatic (Ghosh & Verma, 2009; Moore & Flurkey, 1990), isomerization, ligand and radical (Hsieh, Nair, & Wu, 2006; Park, Kwom, & Choi, 2013; Gille, Knoll, & Quintzsch, 1999) and nucleophilic reaction (Al-Blewi, Al-Lohedan, Rafiquee, & Issa, 2013; Al-Shamay, Al-Lohedan, Rafiquee, & Issa, 2012; Samiey & Toosi, 2009; Latona & Akinola, 2022). Research on the kinetics of aliphatic alcohols by potassium dichromate in aqueous and micellar media has been reported (Hassan, Al-Hakimi, & Alahmadi, 2011). Further studies on the oxidation of alcohols in micellar medium include the oxidation of unsaturated alcohols byN-Clorosaccharin in micellar medium (Agnihotri & Tiwarii, 2021) However, the literature survey shows that micellar catalyzed oxidation of alcohols by $KMnO_4$ in the acidic medium has not been reported. Therefore, the purpose of this research was to study the micellar effect of sodium dodecyl sulphate on the oxidation of alcohols by potassium permanganate in an acidic medium with the view to explaining the results by Menger-Portnoy's Pseudo-Phase model and Piszkiewicz positive cooperativity model.

Materials and Methods

Chemicals

Ethanol (Merck, India), SDS (Fluka, Switzerland), H_2SO_4 (Merck, India), KNO_3, and $KMnO_4$ (BDH) were of Analar grade and used without further purification. All solutions were prepared in mole litre^{-1} using doubly distilled and CO_2-free water. The results were obtained in duplicates.

Kinetic runs

Requisite volumes of SDS, KNO_3, H^+, Ethanol and distilled water except $KMnO_4$ were introduced into the reaction vessel fitted with a double-wall spiral condenser to check evaporation and then placed in a water bath. At the constant temperature of the mixture in the reaction vessel, $KMnO_4$ solution, thermo-stated at the same temperature, was transferred to the mixture in the reaction vessel. In this study, aliquots of the reaction mixtures were withdrawn at definite time intervals, quenched in an ice bath and the absorbance was recorded. The progress of the reaction was monitored by measuring the decrease in absorbance of the reaction mixture at 525 nm using a double-beam Unicam-1800 Shimadzu spectrophotometer equipped with a thermo-regulated cell compartment. The pseudo-first-order rate constants k_ψ (s^{-1}) were calculated from the slope of the plot of log (a-x) versus time.

Results and Discussion

The observed pseudo-first-order rate constants (k_ψ) at various initial concentrations of the reactants are shown in Table 1. The log-log k_ψ versus [X]. where X = $KMnO_4$, Ethanol and H^+ showed first-order dependence each to

[$KMnO_4$] and [Ethanol] and fractional order to [H^+] with no effect on the ionic strength of the mixture. This suggests the presence of a neutral molecule in the rate-determining step.

Table 1 Effect of [Reactants]

10^5[$KMnO_4$] (mol dm^{-3})	10^3[Ethanol] (mol dm^{-3})	10^3[H^+] (mol dm^{-3})	$10^2 k\psi/s^{-1}$
1.00	3.00	1.00	0.21
2.00	3.00	1.00	0.42
3.00	3.00	1.00	0.62
4.00	3.00	1.00	0.82
5.00	3.00	1.00	0.95
6.00	3.00	1.00	1.18
3.00	1.00	1.00	0.18
3.00	2.00	1.00	0.30
3.00	3.00	1.00	0.42
3.00	4.00	1.00	0.59
3.00	5.00	1.00	0.72
3.00	3.00	1.00	0.42
3.00	3.00	2.00	0.53
3.00	3.00	3.00	0.67
3.00	3.00	4.00	0.78
3.00	3.00	5.00	0.84
3.00	3.00	6.00	0.95
3.00	3.00	7.00	1.14

At fixed ionic strength μ= 0.05 mol dm^{-3} maintained by KNO_3, [SDS] 3 x10^{-2} mol dm^{-3} at 298K

Activation parameters obtained from the temperature-dependent study using equations (1) and (2) are shown in Table 2.

$$In\left(\frac{k}{T}\right) = \frac{-\Delta H^{\#}}{RT} + In\left(\frac{k/}{\mathrm{h}}\right) + \left(\frac{\Delta S^{\#}}{R}\right) \qquad (1)$$

$$Where \;\; In\left(\frac{k/}{h}\right) = 23.76$$

$$\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#} \qquad (2)$$

k = rate constant; T= Temperature; $\Delta H^{\#}$ = Enthalpy of Activation; $\Delta S^{\#}$ = Entropy of Activation; $\Delta G^{\#}$ = Free Gibb's Energy of Activation; R = Molar gas constant; $k^{/}$ = Boltzmann constant; h= Plank's constant.

Table 2 Activation parameters

Substrate	$\Delta H^{\#}$ /kJ mol^{-1}	-$\Delta S^{\#}$ /kJ K^{-1} mol^{-1}	$\Delta G^{\#}$ /kJ mol^{-1}
Ethanol	4.16	0.198	63.16

[$KMnO_4$] 3 x 10^{-5} mol dm^{-3}, [Ethanol] 3 x 10^{-3} mol dm^{-3}, [H^+] 1 x 10^{-3} mol dm^{-3}, μ 0.05

Menger Portnoy model

A pseudo-phase kinetic model proposed by Menger and Portnoy was used to interpret the catalytic activity of sodium dodecyl sulphate and to evaluate the binding constant between the substrate and the surfactant. The model considered the partitioning of the substrate between the aqueous and the micellar pseudo phase as shown in Figure 1.

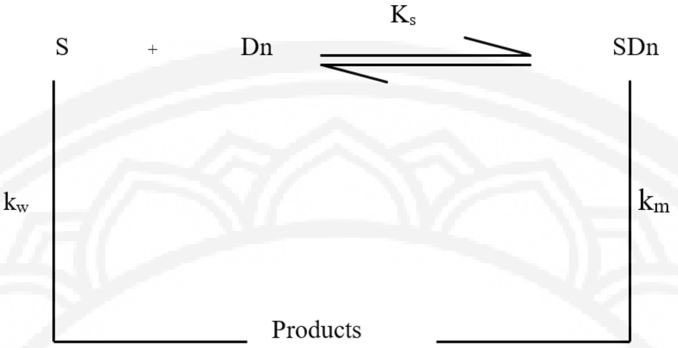

Figure 1 Menger-Portnoy model (D_n= micellar SDS surfactant; S = free substrate; SD_n= associated substrate)

Menger-Portnoy model shows that:

$$k_\psi = \frac{(k_w + k_m K_s[D_n])}{(1 + K_s[D_n])} \qquad (3)$$

k_w is the pseudo-first-order rate constant in the aqueous phase.

k_m is the pseudo-first-order rate constant in the micellar phase.

Ks is the binding constant of the substrate with the surfactant.

$[D_n]$ is the concentration of the micelle surfactant.

$[D]_T$ is the stoichiometric concentration of the surfactant.

$[D_n] = ([D]_T - cmc)$:(cmc is the critical micelle concentration)

The critical micelle concentration of SDS in the reaction mixture is 8.20×10^{-3} mol dm^{-3}(William, Phillips, & Mysel, 1985) and rearranging equation (3) gives:

$$\frac{1}{(k_\psi - k_w)} = \frac{1}{(k_m - k_w)} + \frac{1}{(k_m - k_w)K_s[D_n]} \qquad (4)$$

Figure 2 shows the validity of Menger-Portnoy's pseudo-phase. The value of km was 7.45×10^{-3}s^{-1} and Ks obtained equalled 40.35 mol dm^{-3}.

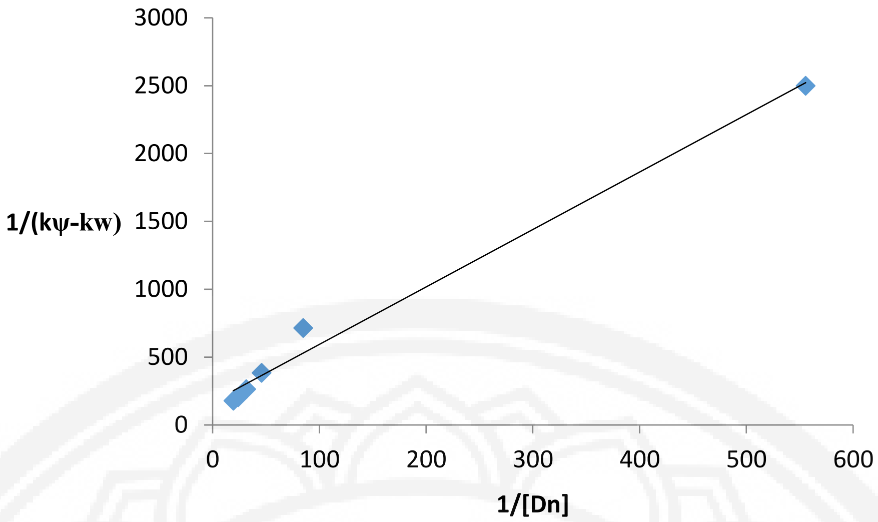

Figure 2 Validity of Menger Portnoy's model

The Piszkiewicz Cooperativity model

The Piszkiewicz Cooperativity model (Figure 3), which is analogous to the Hill model in enzymatic reactions, helps to explain the reactions in micellar systems. It gives a more accurate dependence of the observed rate constants on surfactant concentrations as shown in Figure 2.

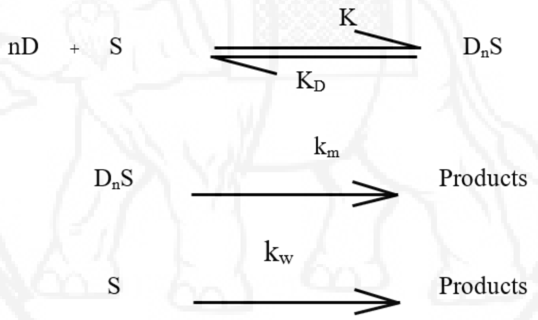

Figure 3 Piszkiewicz Cooperativity model

Piszkiewicz's cooperative model shows that:

$$k_\psi = k_m[D]^n + \frac{K_D k_w}{K_D+[D]^n} \qquad (5)$$

Re-arranging equation 5:

$$\log\frac{(k_\psi - k_w)}{(k_m - k_\psi)} = n\log[D] - \log K_D \qquad (6)$$

K_D is the dissociation constant of the micellized substrate back to its free component

K is the association constant of the micelle-substrate complex.

Applying equation 6 and using the earlier obtained value of k_m in Menger Portnoy's model, the plot of $\log\frac{(k_\psi - k_w)}{(k_m - k_\psi)}$ versus log[D] was linear for this reaction (Figure 4).

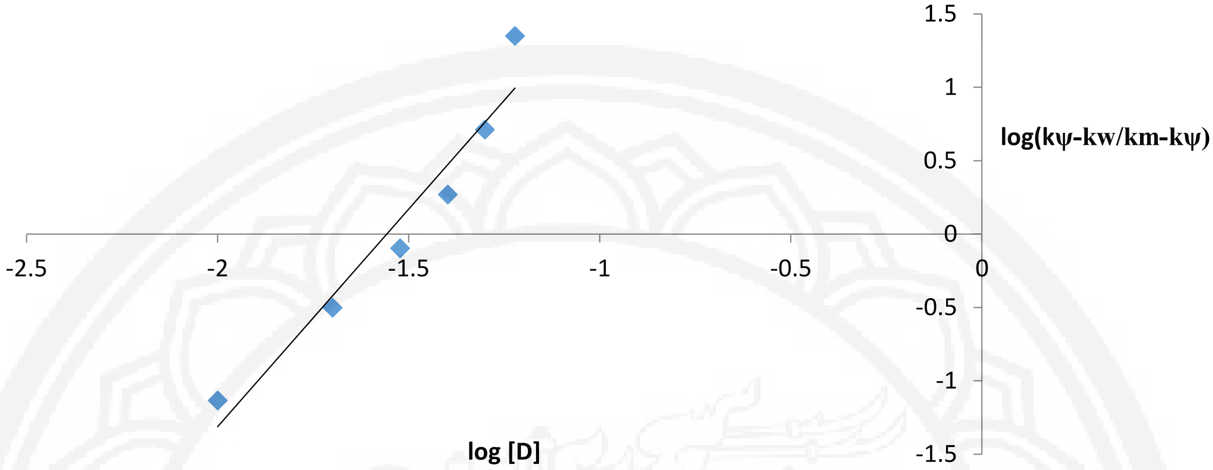

Figure 4 C Plot of $log\frac{(k_\psi - k_w)}{(k_m - k_\psi)}$ versus log[D]

Micelle is a porous cluster with a rough surface and water-filled cavities. The water activity at the surface of ionic micelles is not different from the water activity in the aqueous pseudo-phase (Tascioglu, 1996). The oxidation reaction showed first-order dependence each to [$KMnO_4$], [Ethanol] and [H^+]. The rate of increase for many reactions with the addition of surfactants has been explained based on the Menger-Portnoy model and Piszkiewicz's model (Menger & Portnoy, 1967; Piszkiewicz, 1976). The Pseudo-phase kinetic model proposed by Menger and Portnoy was used to interpret the catalytic activity of SDS and to evaluate the kinetic data. The model considered the partitioning of the substrate between the aqueous and the micellar pseudo phase as shown in Figure 1. Moreover, Piszkiewicsz's cooperativity model, which is likened to the Hill model, applied to enzyme-catalyzed reactions was also used to explain the micellar reaction as shown in Figure 3. n and K_D were determined from the slope and intercept as 2.97 and $2.41 x 10^{-5}$ respectively.

$n > 1$, indicates positive cooperativity which implies that binding of the first molecule of the substrate allows for the binding of subsequent molecules and suggests the formation of catalytic active sub- micellar aggregates (Das, Kar, & Mondal, 2001). The linear plot supports the positive cooperativity between ethanol and micelle to form reactive micelles and it implies that the ethanol molecules are incorporated into the micellar phase. It is noteworthy that for most organic reactions in which the organic substrate is largely in the micellar pseudo phase, little reaction contribution is expected from the aqueous region (Buton, 2006). The lower value of the binding constant suggests that only the substrate is distributed between the micellar and aqueous phases, and the oxidant resides at the stern layer of the micelle and the difference in polarity between water and the micellar media is the driving force for the association of the substrate to the micellar aggregates (Pandey & Upadhyay, 2005; Ghosh & Sar, 1997).

However, the reactants are distributed between the micellar and aqueous phases. Ethanol and the kinetically active oxidant species, protonated $KMnO_4$ are preferably partitioned in the stern layer of the micellar phase. Reaction in the stern layer is electrostatically favored between the protonated $KMnO_4$ and neutral ethanol which consequently leads to an increase in $k\psi$ with an increase in [SDS] as shown in Figure 5.

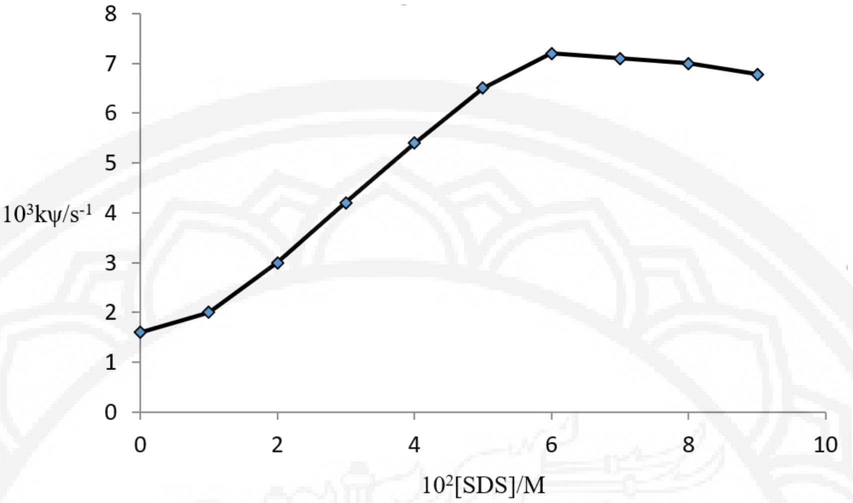

Figure 5 Plot of $k\psi$ versus [SDS]

The reaction was not restricted to the aqueous phase only, but the oxidation reaction that took place in both phases but was favored in the micellar phase where the reactants are preferably concentrated. It has been suggested that an acid-catalyzed reaction in the micellar phase takes place between the micellized alcohol and the acidified potassium permanganate across the boundary. The catalysis has been explained using a pseudo-phase ion exchange model by considering the competitive distribution of the micellar counter ions and H^+ ions with the micellar head groups as shown in equation (7).

$$H_W^+ \; + \; Na_M^+ \rightleftharpoons H_M^+ \; + \; Na_W^+ \qquad (7)$$

The active protonated oxidant accumulates on the surface of the anionic micelle of SDS and is repelled by the cationic micellar head group as shown in equation (8).

$$OX_W^+ \; + \; Na_M^+ \rightleftharpoons OX_W^+ \; + \; Na_W^+ \qquad (8)$$

The increase rate with an increase in [SDS] is attributed to the solubilization of both the substrate and oxidant in the stern layer of the micellar phase. The partitioning of the protonated $KMnO_4$ in the stern layer is due to the ion-dipole interaction and hydrogen bond interaction with the micellar head group (Das et al., 2001). Furthermore, at higher [SDS], an increase in [SDS] increases the concentration of micellar counter ions (Na+) which may displace H^+ and OX^+ ions away from the micellar surface to shift equilibrium to the left of equations 7 and 8, thereby reducing $[H_M^+]$ and $[OX_M^+]$ to inhibit the rate process in the micellar phase (Mondal, Das, Kar, & Das, 2001).

In chemical reactions in which there is an involvement of a protonation, as in this case, it is expected that an anionic surfactant should catalyze the reaction since the positively charged substrate is absorbed over the anionic surface of the surfactant which is supported by this research (Das et al., 2001; Vijapure, 2017).

Conclusion and Suggestions

The micellar effect on the oxidation of Ethanol by $KMnO_4$ in an acidic medium has been investigated. The study followed pseudo-first-order kinetics and the order of reaction was first order each to [$KMnO_4$] and [Ethanol], and fractional order to [H^+]. The oxidation reaction was catalyzed at low SDS concentrations and attained a limiting value at higher SDS concentrations. The kinetic data were analyzed based on the pseudo-phase model as proposed by Menger and Portnoy and the cooperativity model by Piczkiewicz. The two models are quite complimentary however, Piszkiewicz's model, which relates to enzyme-catalyzed reactions, does not depend on the critical micelle concentration value of the surfactant and it can be used to analyze a larger number of data. This study shows that SDS can help facilitate the oxidation of alcohols to carboxylic acids.

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