

Kinetics and Mechanism of Oxidation of Theophylline by Permanganate Ion in Aqueous Sulphuric Acid Medium

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ABSTRACT

The kinetic studies of the oxidation of theophylline by permanganate ion in aqueous acidic solution has been studied. One mole of theophylline was consumed by one mole of permanganate ion. The result showed first order dependence with respect to both theophylline and permanganate ion concentrations. The oxidation rate increased with increase in the concentration of H⁺ and ionic strength of the reaction medium. The reaction was carried out at [MnO₄⁻] = 2.0 x 10⁻⁴ mol dm⁻³, [H⁺] = 1.0 x 10⁻² mol dm⁻³, I = 0.5 mol dm⁻³ (NaSO₄), λ_{max} = 530nm and T = 24 ± 0.1 °C. This conforms to the rate law: -d [MnO₄⁻] /dt = (a + b [H⁺])[TP][MnO₄⁻], where a = 0.034 dm³mol⁻¹ s⁻¹ and b = 0.43 dm³ mol⁻¹ s⁻¹. Spectroscopic and kinetic investigation depict that there was no intermediate formation prior to electron transfer. A plausible reaction mechanism consistent with the kinetic results is proposed.

(Keywords: kinetics studies, stoichiometry, chemical oxidation, permanganate, theophylline, dimethylxanthine)

INTRODUCTION

Theophylline also known as dimethylxanthine (hereafter referred to as TP) is an alkaloid of the xanthine family, naturally found in tea and cocoa beans. It is available in a number of different salts, the most common of which are aminophylline (ethylenediamine) and choline theophyllinate (Irwin, 1997). The compound is used in the therapy of respiratory diseases such as bronchial asthma and reversible bronchospasm which acts by inhibiting cyclic nucleotide phosphodiesterase (Cheryseva, *et al.*, 2000).

Its oxidation was investigated as a tool for spectrophotometric determination of theophylline concentration in pure or pharmaceutical formulations (Singh and Sah, 2006). The compound was oxidized with sodium metaperiodate in the presence of acetic acid and a blue-colored product with a λ_{max} of 630nm was observed (Abe, *et al.*, 2003).

Permanganate is a very important oxidant which is known for its versatility (Hassan, 1991). The redox reactions of permanganate involves Mn (VII) entity which occur in the oxo-compounds like MnO₂, Mn₂O₇, MnO₃F and MnO₄⁻.

The MnO₄⁻ is the most common specie that is used for studying the oxidation of organic and inorganic substrates (Sen *et al.*, 1995). Variety of mechanisms have been proposed by different authors, depending on the nature of the reactive manganese species, the reaction environment and the nature of the substrate (Vogel, 1978). Based on the therapeutic role played by theophylline, it is therefore, expected that the result of this investigation will contribute in the better understanding of the redox reaction of theophylline with oxyanions in pharmacology.

MATERIALS AND METHODS

Standard solution of theophylline was prepared by dissolving accurately 0.435g in 100 cm³ of distilled water. Permanganate ion and sulphuric acid were prepared and standardized titrimetrically. (Idris *et al.*, 2007). Sulphuric acid was used as source of [H⁺]. Ionic strength (I) was maintained at 0.5 mol dm⁻³, using sodium sulphate.

Stoichiometry

The stoichiometry of the reactions was determined by spectrophotometric titrations using the mole ratio method (Idris, *et al.*, 2007; Ukoha and Ibrahim, 2004; Vaidya, *et al.*, 1991; Ukoha, 1999; Iyun and Ukoha, 1999). Concentration of the permanganate was kept constant at 2.0×10^{-4} mol dm⁻³, while that of the TP was varied in the range $(1.0 - 3.8) \times 10^{-2}$ mol dm⁻³, $[H^+] = 1.0 \times 10^{-2}$ mol dm⁻³, $I = 0.50$ mol dm⁻³ (Na₂SO₄) and $\lambda_{max} = 530$ nm. These reactions were allowed to go to completion. The stoichiometry indicated by the point of inflexion, was evaluated from the plot of absorbance versus mole ratio $[TP]/[MnO_4^-]$.

Kinetic Study

The rate of the reaction was monitored by measuring decrease in absorbances of the permanganate at 530nm using Corning Colorimeter 252. Kinetic measurements were made under pseudo-first order conditions with $[TP]$ at least 10-fold excess of $[MnO_4^-]$.

The pseudo first order rate constant (k_{obs}) in each case was obtained from the slope of the plots of $(\log A_t - A_\infty)$ versus time (Lohdip, *et al.*, 1998; Lohdip, 1999; Iyun and Shehu, 2004, Anweting, *et al.*, 2017). The second order rate constant (k_2) was obtained as $k_{obs}/[TP]$ at $[TP] = (1.0 - 3.8) \times 10^{-2}$ mol dm⁻³, $[H^+] = 1.0 \times 10^{-2}$ mol dm⁻³, $I = 0.50$ mol dm⁻³ (Na₂SO₄) and $\lambda_{max} = 530$ nm and $[MnO_4^-] = 2.0 \times 10^{-4}$ mol dm⁻³.

Effect of $[H^+]$ on the Rate of the Reaction

The effect of $[H^+]$ on the rate of the reaction was investigated using sulphuric acid in the range $[H^+] = 0.01 - 0.048$ mol dm⁻³ while the $[TP]$ and MnO_4^- were kept constant at $I = 0.50$ mol dm⁻³ (Na₂SO₄) and $\lambda_{max} = 530$ nm (Ukoha and Iyun, 2001, 2002; Ukoha and Ibrahim, 2004; Anweting *et al.*, 2021).

Effect of Ionic Strength (I) on the Rate of Reaction

The effect of ionic strength of the reaction medium was investigated by keeping the concentration of TP and MnO_4^- constant and varying the concentration of the inert electrolyte (Ukoha and Iyun, 2001, 2002; Ukoha and Ibrahim, 2004). (Na₂SO₄) at $I = 0.5$ mol dm⁻³ (Na₂SO₄), $[MnO_4^-] =$

2.0×10^{-4} mol dm⁻³, $[TP] = 1.0 \times 10^{-2}$ mol dm⁻³ and $[H^+] = 1.0 \times 10^{-2}$ mol dm⁻³.

Effect of Added Cations and Anions on the Rate of Reaction

The effect of added ions in the reaction medium was carried out by adding various concentrations of NO₃⁻, HCOO⁻, Mg²⁺ and Ca²⁺ to the reaction medium while all other conditions remained constants (Ukoha and Iyun, 2001, 2002; Anweting, *et al.*, 2017). (Na₂SO₄) at $I = 0.1 - 0.6$ mol dm⁻³ (Na₂SO₄), $[MnO_4^-] = 2.0 \times 10^{-4}$ mol dm⁻³, $[TP] = 1.0 \times 10^{-2}$ mol dm⁻³ and $[H^+] = 1.0 \times 10^{-2}$ mol dm⁻³.

Free Radical Test

About 5cm³ of acrylamide was added to partially oxidized reaction mixture containing various concentrations of solution of theophylline, reductant and hydrogen ion. This was followed by a large excess of methanol (Osunlaja, *et al.*, 2012; Osunlaja, 2014). The acrylamide was also added to the solution of theophylline and permanganate separately, serving as control.

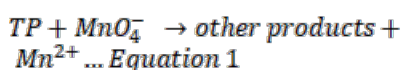
Test for Intermediate Complex Formation

Spectroscopic test was carried out by comparing the electronic spectrum of the reaction mixture ion 23 minutes after the start of the reaction with that of the permanganate ion within a wavelength of 400-700nm. Kinetic test was also investigated using Michaelis-Mentens plot of $1/k_{obs}$ versus $1/[TP]$ (Anweting, *et al.*, 2012a & b).

RESULTS AND DISCUSSION

Stoichiometry

The stoichiometric result showed that one mole of theophylline was consumed by one mole of permanganate ion. The overall reaction is as given by Equation 1.



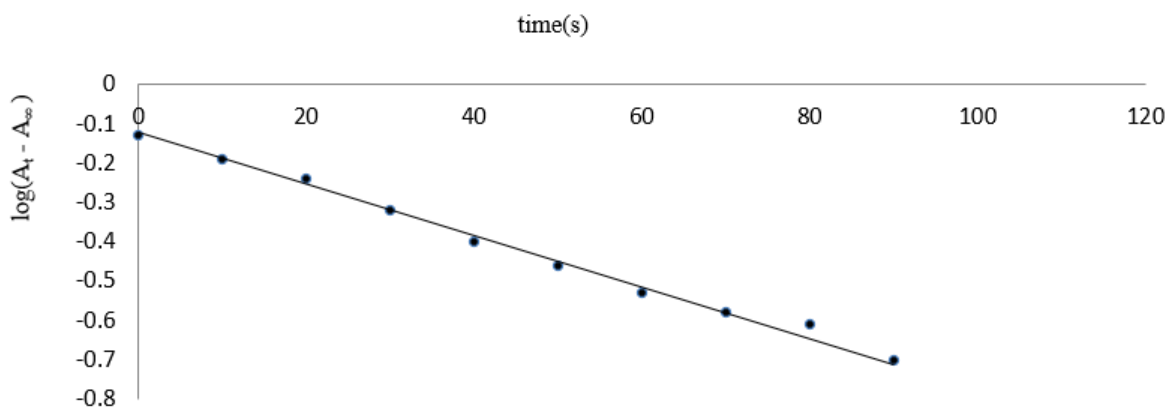


Figure 1: A typical pseudo-First order plot for the redox reaction of TP and $[MnO_4^-]$ at $[TP] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$, $[MnO_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[H^+] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$, $I = 0.5 \text{ mol dm}^{-3}$, $\lambda_{\text{max}} = 530\text{nm}$ and $T = 24.0 \pm 0.1^\circ\text{C}$.

Similar stoichiometry has been reported for the oxidation of malachite green (Mohammed, *et al.*, 2009), nicotine (Ishaq, 2010), nicotinic acid (Anweting, *et al.*, 2012a) and L- tryptophan (Anweting, *et al.*, 2012c) by permanganate ion. However, in the oxidation of L- cysteic acid and L- aspartic acid by permanganate ion, 2 moles of MnO_4^- were reduced by 5 moles each of the reductants (Anweting, *et al.*, 2012b & d). Mn^{2+} , a product of oxidation of MnO_4^- was confirmed qualitatively by reacting the product with indole (Iyun, 1989). Formation of white precipitate is an indication of the presence of Mn^{2+} .

Kinetic Measurements

The pseudo-first order plots of $\log (A_t - A_\infty)$ versus time (Figure 1) were linear to more than 80% extent of reaction, indicating that the reaction is first order in $[MnO_4^-]$ under the experimental conditions.

The pseudo-first order rate constant was determined at different initial concentrations of TP. Plot of $\log k_{\text{obs}}$ versus $\log [TP]$ at, $I = 0.5 \text{ mol dm}^{-3}$, $[H^+] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$ gave a slope of 0.92 ± 0.02 (Figure2).

This suggests a first order dependence on $[TP]$. The values of second order rate constant $k_2 = k_{\text{obs}}/[TP]$ were fairly constant (Table 1) suggesting that the reaction is also first order in $[TP]$ and that the reaction is second order overall, therefore conforming to the rate in Equation 2.

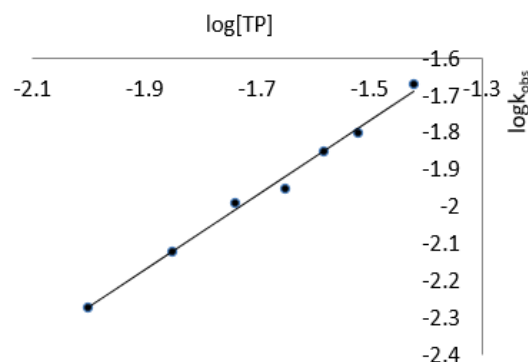
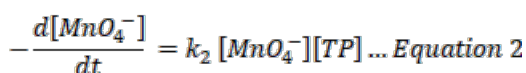


Figure 2: Plot of $\log k_{\text{obs}}$ versus $\log [TP]$ at $[MnO_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[TP] = (1.0 - 3.8) \times 10^{-2} \text{ mol dm}^{-3}$, $I = 0.5 \text{ mol dm}^{-3}$ (Na_2SO_4) and $T = 24 \pm 0.1^\circ\text{C}$.

Similar order kinetics have been reported in the reduction reactions of permanganate ion by aliphatic aldehydes (Banerji, 1973), acetaldelhydes (Ajibade and Iyun, 1992), DL- alanine (Hassan, 1991), L- aspragines (Zahedi and Bahrami, 2004) and L-tryptophan (Anweting, *et al.*, 2012a).

Table 1: Pseudo-first order and second order rate constants for the reaction of theophylline and MnO_4^- at $[\text{TP}] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$, $[\text{MnO}_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$, $T = 24.0 \pm 0.1^\circ\text{C}$, $I = 0.50 \text{ mol dm}^{-3}$ and $\lambda_{\text{max}} = 530 \text{ nm}$.

$10^2 [\text{TP}]$, mol dm^{-3}	$10^2 [\text{H}^+]$, mol dm^{-3}	$10 [\text{I}]$, mol dm^{-3}	$10^3 k_{\text{obs}}$, s^{-1}	k_2 , $\text{dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$
1.0	1.0	5.0	5.31	0.53
1.4	1.0	5.0	7.53	0.54
1.8	1.0	5.0	1.02	0.57
2.2	1.0	5.0	1.11	0.50
2.6	1.0	5.0	1.42	0.54
3.0	1.0	5.0	1.57	0.52
3.8	1.0	5.0	2.09	0.55
1.0	1.0	5.0	5.37	0.53
1.0	1.6	5.0	6.02	0.60
1.0	2.4	5.0	6.90	0.69
1.0	3.2	5.0	7.53	0.75
1.0	4.0	5.0	8.88	0.88
1.0	4.8	5.0	9.97	0.99
1.0	1.0	1.0	3.78	0.37
1.0	1.0	2.0	3.94	0.39
1.0	1.0	3.0	4.18	0.41
1.0	1.0	4.0	4.60	0.46
1.0	1.0	5.0	5.75	0.57
1.0	1.0	6.0	6.52	0.65

Effect of $[\text{H}^+]$ on the Rate

The results suggest that the rate of the reaction increased with increase in $[\text{H}^+]$ (Table 1) at $[\text{TP}] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$, $[\text{MnO}_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[\text{H}^+] = 2.0 \times 10^{-2} - 3.4 \times 10^{-1} \text{ mol dm}^{-3}$, $I = 0.5 \text{ mol dm}^{-3}$ (Na_2SO_4), $T = 24 \pm 0.1^\circ\text{C}$. A plot of k_2 versus $[\text{H}^+]$ was linear with a positive intercept and can be represented as:

$$k_2 = a + b[\text{H}^+] \dots \text{Equation 3}$$

where $a = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$ and $b = 0.5 \text{ mol dm}^{-3}$.

The rate of the reaction showed two parallel pathways of reaction; one involving the protonated and the other the unprotonated species both reacting to give a product (Babatunde and Iyun, 2009). Results analogous to this were reported in the reaction of permanganate ion with L- ascorbic acid (Babatunde, 2008), and malachite green

(Mohammed, *et al.*, 2009). Therefore, the rate equation of the reaction is as presented in Equation 4.

$$-\frac{d[\text{MnO}_4^-]}{dt} = (a + b[\text{H}^+])[\text{TP}][\text{MnO}_4^-] \dots \text{Eqn 4}$$

The Effect of Ionic Strength on the Rate of the Reaction

The increase in ionic strength of the reaction medium resulted in increase in the rate of the reaction within the concentration range studied, the positive salt effect observed for the reaction showed the interaction of like charges (Bronsted-Debye positive salt effect) in the activated complex (Wilkins, 1974). Similar results were obtained by Adetoro, *et al.* (2010) and Babatunde (2008).

Effect of Added Anions and Cations on the Rate

The added ions NO_3^- , HCOO^- , Mg^{2+} and Ca^{2+} mildly enhanced the rate of the reaction as shown on Table 2. Catalysis of the reaction by these added anions and cations is possibly due to the coulombic bridging in which the closeness of approach of the reactant ions in the activated complex is such as to allow added ions to come in between them, thereby affecting the rates of reaction. This is when the reaction proceeding through the outer-sphere pathway in which the reactant ions maintain their coordination integrity in the activated complex prior to and during electron transfer (Sharpe, 1982).

Test for Intermediate Complex Formation

Spectrophotometric measurement showed no clear shift in the λ_{max} of from 530nm characteristic of MnO_4^- when reactants were mixed together, and the electronic spectrum was recorded after 20 minutes of mixing. This suggests the absence of intermediate complex formation, the spectrum is shown in Figure 3.

Table 2: Rate constants for the effect of cations and anions in the oxidation reaction between TP and MnO_4^- at $[\text{TP}] = 1.0 \times 10^{-2} \text{ mol dm}^{-3}$, $[\text{H}^+] = 1.0$

$\times 10^{-2} \text{ mol dm}^{-3}$, $[\text{MnO}_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$ and $I = 0.5 \text{ mol dm}^{-3} (\text{Na}_2\text{SO}_4)$.

Z^{n+}	$10^3 [Z^{n+}]$, mol dm^{-3}	$10^3 k_{\text{obs}}$, s^{-1}	k_2 , $\text{dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$
Mg²⁺ ,	0.00	5.26	0.52
	5.00	5.59	0.55
	10.0	5.92	0.59
	20.0	6.52	0.65
	30.0	6.90	0.69
	40.0	7.29	0.72
Ca²⁺	0.00	5.37	0.53
	5.00	5.57	0.55
	10.0	5.83	0.58
	20.0	6.22	0.62
	30.0	6.72	0.67
	40.0	7.04	0.70
HCOO⁻	0.00	5.22	0.52
	5.00	5.40	0.54
	10.0	5.71	0.57
	20.0	6.04	0.60
	30.0	6.32	0.63
	40.0	6.64	0.66
NO₃⁻	0.00	5.37	0.53
	5.00	5.52	0.55
	10.0	5.73	0.57
	20.0	6.00	0.60
	30.0	6.20	0.62
	40.0	6.40	0.64

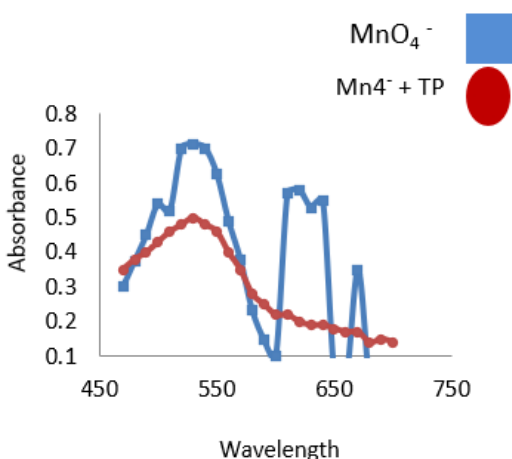


Figure 3: Spectra of MnO_4^- and $\text{MnO}_4^- + \text{TP}$

Michaelis-Mentens plot of $1/k_{\text{obs}}$ versus $1/[\text{TP}]$ was linear without intercept (Figure 4), this further ruled out the formation of intermediate complex

prior to the electron transfer (Anweting, *et al.*, 2012a, b, c & d).

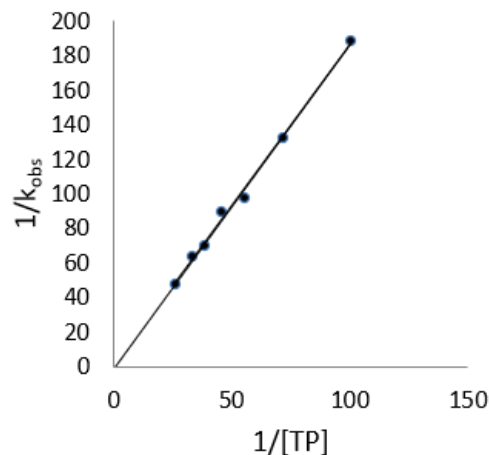
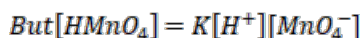
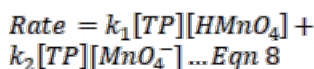
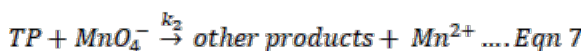
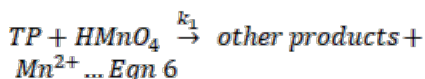
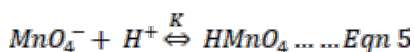


Figure 4: Michaelis-Mentens plot of $1/k_{\text{obs}}$ against $1/[\text{TP}]$ at $[\text{MnO}_4^-] = 2.0 \times 10^{-4} \text{ mol dm}^{-3}$, $[\text{TP}] = (1.0 - 3.8) \times 10^{-2} \text{ mol dm}^{-3}$, $I = 0.5 \text{ mol dm}^{-3} (\text{Na}_2\text{SO}_4)$ and $T = 24 \pm 0.1^\circ\text{C}$

Free Radical Test

There was no gel formation when 5cm^3 of acrylamide followed by excess methanol were added to partially oxidized reaction mixture, solution of theophylline and permanganate separately. The absence of gel formation (polymerization) suggests that no free radical was involved in the reactions.

The following reaction mechanism is therefore proposed to accommodate all the experimental observations in Equations 5 – 11.



$$\text{Rate} = Kk_1[TP][MnO_4^-][H^+] + k_2[TP][MnO_4^-] \dots \text{Eqn 9}$$

$$\text{Rate} = (Kk_1[H^+] + k_2)[TP][MnO_4^-] \dots \text{Eqn 10}$$

$$(Kk_1[H^+] + k_2) \approx k' \dots \text{Eqn 11}$$

Therefore

$$\text{Rate} = k'[TP][MnO_4^-] \dots \text{Eqn 11}$$

CONCLUSION

The oxidation of theophylline by permanganate ion showed a stoichiometry of 1:1, first – order dependence with respect to concentrations of both theophylline and permanganate ion and second order rate overall. The rate of the reaction increased with increase in the concentrations of hydrogen ion and ionic strength of the reaction medium. The added ions mildly enhanced the rate of the reaction. There was no evidence for the formation of an intermediate complex. From the above findings, it is evident that the reaction is operating through outer-sphere mechanism.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interest.

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