

Kinetics and Mechanism of the Oxidation of Ethylenediamine-N,N,N',N'-tetraacetatocobaltate(II) Complex by Hypochlorite ion in Aqueous Nitric Acid Medium

By

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ABSTRACT

The kinetics of the oxidation of ethylenediamine-N,N,N',N'-tetraacetatocobaltate(II) (hereafter, $[Co(II)EDTA]^{2-}$) complex by hypochlorite ion in aqueous nitric acid medium have been studied at $I = 0.5 \text{ mol dm}^{-3}$ ($NaNO_3$), $[H^+] = 4 \times 10^{-3} \text{ mol dm}^{-3}$, $T = 300 \pm 1 \text{ K}$ and $\lambda_{max} = 520 \text{ nm}$. Stoichiometry of 1:1 of the reaction is reported. The rate law derived from the kinetic study under pseudo first order condition is $\frac{d[C(II)EDTA^{2-}]}{dt} = k_2[C(II)EDTA^{2-}][ClO^-]$. The rate constant of reaction, k_2 varies inversely with acid concentration, $[H^+]$ within the investigated concentration range. Thus, the overall rate law is $\frac{d[C(II)EDTA^{2-}]}{dt} = 1.23 \times 10^{-3} [H^+]^{-1}[C(II)EDTA^{2-}][ClO^-]$. The reaction displayed positive salt effect which suggests the activated complex was made up of similar charged species. The reaction was catalysed by addition of both formate, $HCOO^-$ and lithium, Li^+ ions and the Michaelis-Menten's plot gave zero intercept indicating the absence of binuclear intermediate complex during the course of the reaction. A reaction mechanism via an outer-sphere pathway is proposed for this reaction.

Keywords: Oxidation; Electron transfer; Hypochlorite; Ionic strength; Kinetics

INTRODUCTION

Reduction-Oxidation (Redox) reaction is the transfer of electrons between two Redox species like ions, molecules, biological systems etc. The reaction usually involves rearrangement and transfer of outermost shell electrons of reacting species in order to gain stability. Thus, oxidation is the loss of electrons while reduction is the gain of electrons (Wong *et al.*, 2002). Many reactions in inorganic and biological systems involve transfer of electron, thus, electron transfer reaction plays a key role to various physical and biological systems (Onu *et al.*, 2008, 2009, 2015 and 2016; François and Jamal, 2016; Idris *et al.*, 2015; Ilbert and Bonnefoy, 2013; Xie *et al.*, 2012; Xiang-Rong and Xiang-Zhong 2010; Naik *et al.*, 2007 and 2010). Transition metal-aminocarboxylate complexes play an important role in metalloenzyme catalysed reactions (Vuckovic *et al.*, 2011) and are generally accepted as simple models for some biological systems involving transition metals (Swaroop *et al.*, 1991). Typical examples of these complexes is ethylenediamine-N,N,N',N'-tetraacetatocobalt(II) complex, $[C(II)EDTA]^{2-}$. The biological importance of cobalt is well documented (Michihiko and Sakayu, 1999; David *et al.*, 1999 and Chang *et al.*, 2010). Cobalt is an

active centre for co-enzymes called cobalamins (also known as vitamin B₁₂) which are vital in amino acid metabolism, normal functioning of the brain and nervous system via the synthesis of myelin as well as an essential factor in DNA synthesis for chromosomal replication and division (Miller *et al.*, 2005; Schoonover *et al.*, 2004; Reynolds, 2006 and Arslan *et al.*, 2013). However, deficiency of cobalt in human leads to pernicious anemia and possible lethal disorder (Michihiko and Sakayu, 1999). These can be treated by the use of synthesised cobalamins, hydroxo-cobalamin or cyano-cobalamin (Carmel, 2008 and Andres *et al.*, 2009 and 2010), hence, the need to study their reactions. Despite the applications of this important complex, there is a paucity of information on the kinetics of its oxidation by oxyanions such as hypochlorite ion, ClO⁻ though, other researchers (Onu *et al.*, 2008, 2009, 2015 and 2016; Naik *et al.*, 2007 and 2010 and Mansour, 2003) have carried out the study using different oxidants. Both inner-sphere and outer-sphere have been reported in these works. Moreover, ClO⁻ ion is the strongest oxidizing agent of the chlorine oxyanions (Mohammed *et al.*, 2010), thus its reaction with this complex will give us more insight on the mechanism of oxidation of the complex which may be used as a model in understanding the mechanism of oxygen transport and metalloenzyme reactions in biological systems (Vuckovic *et al.*, 2011). Also, the study will be used as simple models to understanding or to mimic the biochemical pathways of some metabolic processes which involve the use of co-enzymes.

The interest here is to study the kinetics of oxidation of ethylenediamine-N,N,N',N'-tetraacetatocobalt(II) complex by hypochlorite ion in aqueous nitric acid medium. It is our hope that this study will complement much needed kinetic information and highlight more on the pathway of the reaction.

EXPERIMENTAL

All chemicals and reagents used were of analar grade and distilled water was used in preparing the solutions. The complex, [Co(II)EDTA]²⁻ was prepared according to the method adopted by Onu *et al.* (2016). This gave λ_{\max} of 519 nm after characterization using Cary series 300 Uv-Vis spectrophotometre in the wavelength range of 400 – 800 nm. Stock solutions of various reagents were prepared by dissolving accurately weighed amounts of each in a known volume of distilled water.

The stoichiometry of the reaction was determined by spectrophotometric titration using the mole ratio method (Onu *et al.*, 2009, 2010, and 2015). The concentration of [Co(II)EDTA]²⁻ was kept constant at 5.0×10^{-3} mol dm⁻³ while that of ClO⁻ was varied from 4 folds below and above. The reactions were allowed to go to completion at constant [H⁺] (HNO₃ = 4.0×10^{-2} mol dm⁻³), ionic strength (NaNO₃ = 0.1 mol dm⁻³), $\lambda_{\max} = 520$ nm T = 300 ± 1 K. The stoichiometry (indicated by the point of inflexion) of the reaction was determined from the plot of absorbance against mole ratio [ClO⁻]/[Co(II)EDTA²⁻].

The rate of the reaction was monitored by recording the change in absorbance at 520 nm on a Sherwood Colorimeter 254. The kinetic study was carried out under pseudo-first order conditions with the concentration of [Co(II)EDTA]²⁻ = 5.0×10^{-3} mol dm⁻³ and that of the oxidant, ClO⁻ in at least 12-fold excess over the reductant at temperature of 300 ± 1 K, ionic strength of 0.5 mol dm⁻³ and [H⁺] = 4.0×10^{-2} mol dm⁻³. Pseudo-first order rate constants (k_{obs}) for the reactions

were obtained from slope of the plots of $\log (A_{\infty}-A_t)$ versus time. Where A_{∞} is the absorbance at infinity and A_t is the absorbance at time t . The order with respect to the oxidant concentration ($[\text{ClO}^-]$) was determined from the slope of the plots of $\log k_{\text{obs}}$ versus $\log [\text{ClO}^-]$. The second order rate constants, k_2 were obtained from k_{obs} as $k_{\text{obs}}/[\text{ClO}^-]$ and the results are presented in Table 1. The effects of change in acid concentration, ionic strength on the reaction rate were studied under their reaction conditions (Table 2 and 3). Effect of added ions is investigated and reported in Table 4 to help us assign the mechanistic pathway for the reaction. Likely presence of free radical species was tested by the addition of 5.0 cm³ of acrylamide to a partially oxidized reaction mixture.

RESULT AND DISCUSSIONS

The result of spectrophotometric titration showed that one mole of the reductant was oxidised by one mole of the oxidant. This is in conformity with stoichiometric equation presented in Equation 1:



This stoichiometry (1:1) may be attributed to the fact that, one mole of electron was transferred in the reaction of complex to form Co(III) complex. Similar results have been reported in converting Mn(III) to Mn(V) during the Jacobsen epoxidation reaction and in the conversion of Ce^{3+} to Ce^{4+} (Perumareddi *et al.*, 2003) which involved hypochlorite ion. The spectrum of the reaction product showed two maxima at 382 and 535 nm using Carey series 300 Uv-Vis spectrophotometre in the wavelength range of 400 – 800 nm. This is typical spectrum of Co(III) (Abdel-Khalek *et al.*, 1993; Mansur, 2003; Perveen *et al.*, 2013 and Onu *et al.*, 2009, 2015 and 2016). Also, qualitative analyses confirmed the presence of C_2 gas.

The pseudo – first order plots was linear to more than 70% extent of the reaction which suggests a first order dependence of rate on $[\text{C}(\text{II})\text{E}]^{2-}$. The slope obtained from the logarithmic plot of k_{obs} versus $[\text{ClO}^-]$ was 1.029 indicating first order dependence with respect to $[\text{ClO}^-]$. The observed first order dependence of the reaction rates on both $[\text{C}(\text{II})\text{E}]^{2-}$ and $[\text{ClO}^-]$ is a common feature of hypochlorite ion reactions (Jiann-Kuo, 1987; Idris *et al.*, 2015). The reaction is second order overall at constant $[\text{H}^+]$ concentrations. Therefore the rate equation for the reaction is

$$\frac{d[\text{C}(\text{II})\text{E}]^{2-}}{dt} = k_2[\text{C}(\text{II})\text{E}]^{2-}[\text{ClO}^-] \quad \dots 2$$

where $k_2 = (3.31 \pm 0.07) \times 10^{-2} \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$

Variation in hydrogen ion concentration showed that the reaction rate decreased with increase in $[\text{H}^+]$ within the concentration range investigated. This inverse relationship implies that there is deprotonation pre-equilibrium step and the rate determining step involves both the deprotonated and undeprotonated species (Wilkins, 2002 and Onu *et al.*, 2009, 2015 and 2016).

Similar inverse dependence on the acid has been reported (Onu *et al.*, 2009, 2015 and 2016) for reaction involving the complex with oxyanions. The slope obtained from the plot of $\log k_2$ vs $[\text{H}^+]$ was -0.84, indicating negative first order with respect to acid concentration. Also plot of k_2 vs $[\text{H}^+]$ was linear with zero intercept. The overall rate equation showing acid dependence effect is represented in Equation 3:

$$\frac{d[C(I)E A^-]}{dt} = (a[H^+]^{-1})[C(I)E A^{2-}][C^-] \quad \dots 3$$

where $a = 1.23 \times 10^{-3} \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$

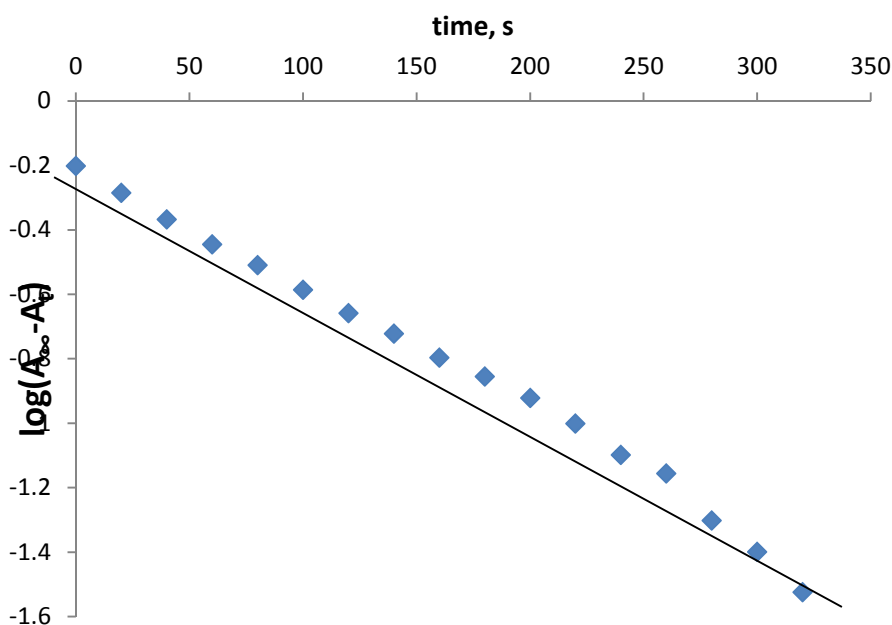


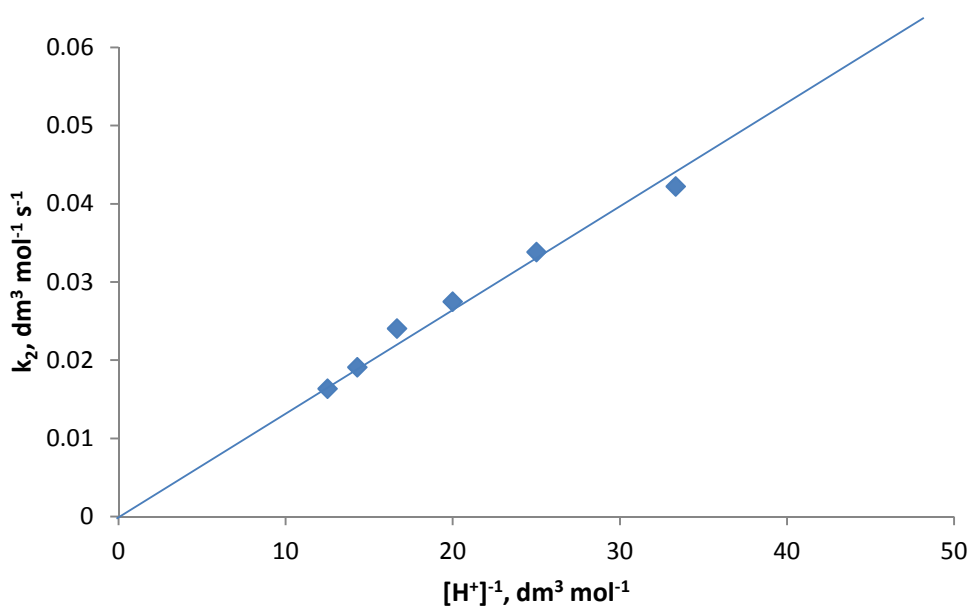
Fig. 1: Typical pseudo-first order Plot for the reaction

Table 1: The pseudo-first order and reaction rate constants for the reaction of $[Co^{II}EDTA]^{2-}$ and ClO^- at $[Co^{II}EDTA^{2-}] = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$, $T = 300 \pm 1K$ and $\lambda_{max} = 520 \text{ nm}$

$10^3[ClO^-]$, mol dm ⁻³	$10^3[H^+]$ mol dm ⁻³	$10^2 I$ mol dm ⁻³	$10^3 k_{obs}$ S ⁻¹	$10^2 k_2$ dm ³ mol ⁻¹ S ⁻¹
50.0	40.0	50.0	1.5	3.3
60.0	40.0	50.0	1.8	3.4
70.0	40.0	50.0	2.2	3.2
80.0	40.0	50.0	2.6	3.3
90.0	40.0	50.0	3.0	3.4
100.0	40.0	50.0	3.3	3.3
110.0	40.0	50.0	3.7	3.3

Table 2: Effect of Acid on the reaction rate for the reaction of $[\text{Co}^{\text{II}}\text{EDTA}]^{2-}$ and ClO^- at $[\text{Co}^{\text{II}}\text{EDTA}^{2-}] = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$, $T = 300 \pm 1\text{K}$ and $\lambda_{\text{max}} = 520 \text{ nm}$

$10^3[\text{ClO}^-]$, mol dm^{-3}	$10^3[\text{H}^+]$ mol dm^{-3}	10^2 I mol dm^{-3}	$10^3 k_{\text{obs}}$ S^{-1}	$10^2 k_2$ $\text{dm}^3 \text{mol}^{-1} \text{S}^{-1}$
90.0	30.0	50.0	3.8	4.2
90.0	40.0	50.0	3.0	3.4
90.0	50.0	50.0	2.5	2.8
90.0	60.0	50.0	2.2	2.4
90.0	70.0	50.0	1.7	1.9
90.0	80.0	50.0	1.5	1.6

**Fig. 2:** Plot of k_2 versus $[\text{H}^+]^{-1}$ for the reaction**Table 3:** Effect of Ionic Strength of the medium on the reaction rate for the reaction of $[\text{Co}^{\text{II}}\text{EDTA}]^{2-}$ and ClO^- at $[\text{Co}^{\text{II}}\text{EDTA}^{2-}] = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$, $T = 300 \pm 1\text{K}$ and $\lambda_{\text{max}} = 520 \text{ nm}$

$10^3[\text{ClO}^-]$, mol dm^{-3}	$10^3[\text{H}^+]$ mol dm^{-3}	10^2 I mol dm^{-3}	$10^3 k_{\text{obs}}$ S^{-1}	$10^2 k_2$ $\text{dm}^3 \text{mol}^{-1} \text{S}^{-1}$
90.0	40.0	20.0	2.0	2.2
90.0	40.0	30.0	2.4	2.6
90.0	40.0	40.0	2.9	3.2
90.0	40.0	50.0	3.2	3.5
90.0	40.0	60.0	4.1	4.5
90.0	40.0	70.0	4.4	4.9
90.0	40.0	80.0	4.9	5.5

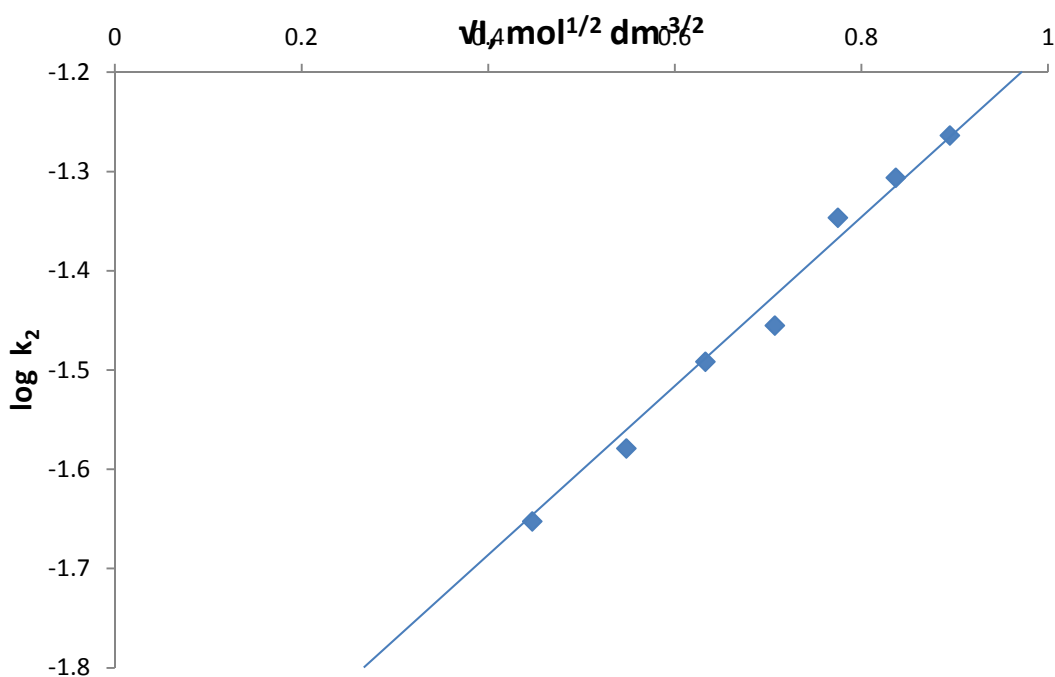


Fig. 3: Plot of $\log k_2$ versus \sqrt{I} for the reaction

Table 4: Effect of added anions on the reaction rate of $[\text{Co}^{\text{II}}\text{EDTA}]^{2-}$ and ClO^- at $[\text{Co}^{\text{II}}\text{EDTA}^{2-}] = 5.0 \times 10^{-3} \text{ mol dm}^{-3}$, $[\text{ClO}^-] = 9.0 \times 10^{-2} \text{ mol dm}^{-3}$, $I = 0.5 \text{ mol dm}^{-3}$, $T = 300 \pm 1\text{K}$ and $\lambda_{\text{max}} = 520 \text{ nm}$

X	$10^2 [X]$, mol dm^{-3}	$10^3 k_{\text{obs}}$, s^{-1}	$10^2 k_2$, $\text{dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$
HCOO^-	0.00	3.00	3.33
	1.00	4.61	5.12
	2.00	6.91	7.68
	3.00	9.21	10.24
	4.00	11.52	12.79
	5.00	13.82	15.35
Li^+	0.00	3.00	3.33
	1.00	2.85	3.17
	2.00	2.78	3.09
	3.00	2.71	3.01
	4.00	2.54	2.82
	5.00	2.20	2.44

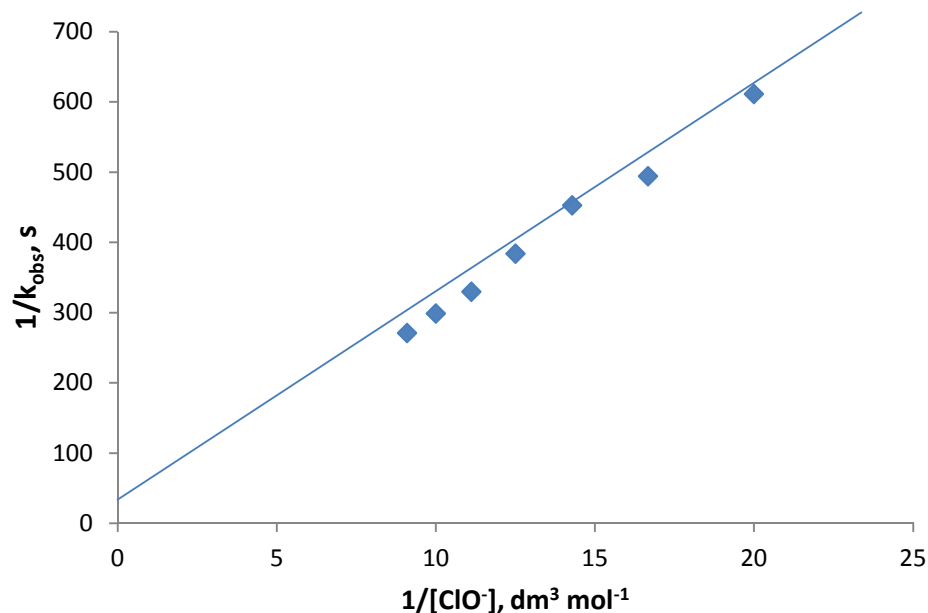
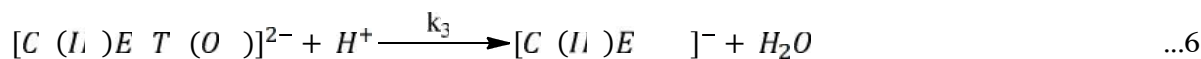
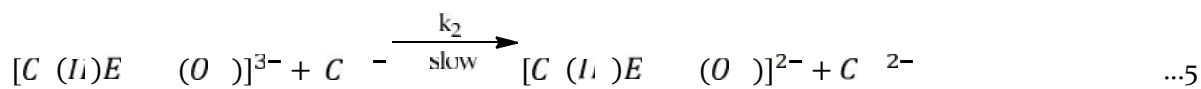
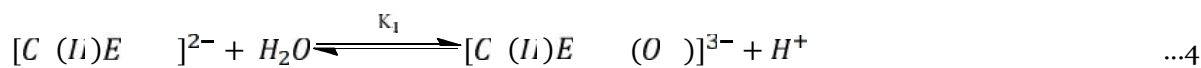


Fig. 4: Michaelis-Menten plot for the reaction

The rate of the reaction increased with increase in ionic strength of the reaction medium in the concentration range investigated (Table 3) suggesting positive Brosted-Debye salt effect (Benson, 1969). This shows that there may be the presence of similar charged species during the formation of activated complex (Atkins and de Paula, 2002) for the reaction. Positive value of the slope of plot of $\log k_2$ versus \sqrt{I} (Figure 3.0) supported the presence of two similar charged species at the rate determining step.

The rate of reaction was found to increase by the addition of formate ion and decrease with addition Li^+ ion. The result is presented in Table 4. This showed that the reaction is catalysed by the presence of both added cation and anion which implies that an outer-sphere mechanism is likely to be in operation. This may be supported by the Michaelis - Menten plot of $1/k_{\text{obs}}$ versus $1/\text{ClO}^-$ which had zero intercept suggesting the absence of an intermediate in the rate determining step, thus, an outer-sphere mechanism is likely to be in operation. The free radical species was not detected as the reaction progresses evidenced by absent of gel formation upon addition 5.0 cm^3 of acrylamide.

In view of the above results obtained and discussions, the following reaction mechanism is hereby proposed via outer-sphere pathways:



$$\frac{d[C(I)E^{2-}]}{dt} = k_2[C(I)E^{2-}][O^{3-}][C^{+}] \quad \dots 8$$

$$\text{But } [C(I)E^{2-}][O^{3-}] = \frac{K_1[C(I)E^{2-}][H_2O]}{[H^+]} \quad \dots 9$$

$$\frac{d[C(I)E^{2-}]}{dt} = \frac{K_1 k_2}{[H^+]} [C(I)E^{2-}][C^{+}][H_2O] \quad 5.38[H_2O] \approx 1$$

$$\text{Hence, } \frac{d[C(I)E^{2-}]}{dt} = \frac{K_1 k_2}{[H^+]} [C(I)E^{2-}][C^{+}] \quad \dots 10$$

This is analogous to Eq. 3, where $a = K_1 k_2$

CONCLUSION

The kinetics of the oxidation of ethylenediamine-N,N,N',N'-tetraacetatocobaltate(II) complex by hypochlorite ion in aqueous nitric acid medium was carried out. While the rate of the reaction was found to be inversely dependent on acid concentration, $[H^+]$, it increases with increase in ionic strength of the reaction medium. The experimental data showed that, the oxidation reaction proceeds through outer-sphere mechanism and a plausible mechanistic pathway which explained the experimental data was proposed for the reaction.

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