

ELECTRON TRANSFER REACTION OF GLUTAMIC ACID AND SYNTHESIZED BIS[BIS(ETHYLENEDIAMINE)SUCCINIMIDATO-COBALT(III)]DINITRATE DIHYDRATE IN AQUEOUS HYDROCHLORIC ACIDIC MEDIUM



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Abstract:	Redox reaction of the glutamic acid, (Glu) a cobalt(III)]dinitrate dehydrate, $(suc)(en)_2Co(\mu-O_2)Co(en studied in aqueous hydrochloric acidic medium at wave first order dependence each on Co(O_2)Co2+, [Glu], and shown below: Rate = k_1k_2k_3[Co(O_2)Co2+][Glu][H4]$	tamic acid, (Glu) and synthesized <i>bis</i> [<i>bis</i> (ethylenediamine)succinimidato- suc)(en) ₂ Co(μ -O ₂)Co(en) ₂ (suc)](NO ₃) _{2.2} H ₂ O hereby referred Co(O ₂)Co ²⁺ has been acidic medium at wavelength 420 nm and temperature 27 ± 1°C. The rate shows Co(O ₂)Co ²⁺ , [Glu], and [H ⁺]. The overall rate equation for the reaction can be $Co(O_2)Co^{2+}$][<i>Glu</i>][H ⁺]+ k_4k_5 [$Co(O_2)Co^{2+}$][<i>Glu</i>] Michaelis-Menten plot is			
Keywords:	linear and passes through the origin and spectroscopic maximum characteristic. Variation of the ionic streng Bronsted-Debye salt effect. The rate laws proposed is in outersphere mechanistic pathway is probably in operation Amino acid, redox reaction, <i>bis[bis</i> (ethylenediamine)suc	studies indicate no significant shift from the absorption gth in the range $1 \le I \le 1.2$ mol dm ⁻³ indicates a positive agreement with the experimental results discussed and an n in this reaction. cinimidato-cobalt(III)]dinitrate dihydrate complex			

Introduction

The understanding of the metabolisms of proteins in the biological system has made the studies of the oxidation of amino acids an utmost interest to the researcher. The amino and carboxyl functional groups present in the amino acid; RCH(NH₂)COOH are more reactive compared to the hydrocarbon (R) moiety and hence only these functional groups undergo chemical transformation. The kinetics and mechanism of glutamic acid (GCO₂H) oxidation by acid permanganate in the absence and presence of sodium dodecyl sulphate (SDS) has been reported (Yousuf and Firoz, 1989). The surfactant enhances the reaction rate without changing the reaction mechanism. The overall rate expression for the reduction of Mn(VII) ion by glutamic acid indicate first order with respect to the two reactant species and second order overall.

Literature have shown kinetics of the oxidation of Glutamic acid by bromobenzenesulphonamide in acidic medium (Puttaswamy and Nirmala, 2011), N-bromo phthalimide in aqueous perchloric acid medium (Alhaji and Sofiya, 2011) and Bi(V) phosphato complex (Avinash *et al.*, 2012). Also the kinetic of oxidation of the chromium(III) –L-glutamic acid complex by periodate in aqueous solution has been reported (Hassan *et al.*, 2001).

There are many reports on the kinetics of oxidation of amino acids with *N*-halo compounds, such as chloramine-T (Mahadevappa et al., 1981), chloramine-B (Mahadevappa et al., 1988), bromamine - B (Puttaswamy and Nirmala, 2011), *N*-chlorosaccharin (Mohamed et al., 2004), *N*bromonicotinimide (Pushphalatha and Vivekanandan, 2009), and *N*-bromophthalimide (Singh et al., 2010).

The nature of the bonding of oxygen to other atoms, and the role of the oxygen atoms as a bonding bridge have been the main problems with which investigators of the structure of chemical compounds and condensed phases have had to deal with in recent years. The mechanism of oxygen bridge formation, the nature of the oxygen bridge and the structure and properties of compounds with oxygen bonding have been studied, using different methods, for many years, Also the high level of research activity on dioxygen complexes over the past two decades has been concerned primarily with the equilibria and kinetics studies of their electronic and vibrational spectra, magnetic properties, and crystal structures (Arthur, 1982).

However, no attention has been focused on the reaction of μ peroxobridged binuclear cobalt(III) complex of succinimide with amino acid, particularly with respect to the oxidation of glutamic acid. Here we report the results of the electron transfer reaction of glutamic acid by synthesized μ peroxobridged binuclear cobalt(III) complex of succinimide in aqueous hydrochloric acidic medium.

Experimental

Peroxobis[bis(ethylenediamine)succinimidatocobalt(III)]dinitr ate (Co(O₂)Co²⁺) was synthesized using a modification of the method of Orphan, (2009). A stock solution was prepared in distilled water every week. Glutamic acid (Glu) was purchased from Sigmal Adrich and used without further purification. All chemicals used were reagent grade and double distilled water was used throughout the work. A solution of glutamic acid (Glu) was prepared by dissolving an appropriate amount of the sample in double distilled water. Its purity was checked by FTIR spectra in which all the bands are in agreement with the literature values of Orphan, (2009). *Stoichiometry*

The stoichiometry of the reaction was determined by spectrophotometric titration using the mole ratio method (Iyun, *et al.*, 1993; Mamman, and Iyun, 2007). The concentration of Co(O₂)Co²⁺ was kept constant at 1.4 x 10⁻³ mol dm⁻³; [Co(O₂)Co²⁺]:[Glu] = 1.00 – 3.00 mole ratio, [H⁺] = 1.0 x 10⁻³ mol dm⁻³; I = 0.5 mol dm⁻³. The reactions were allowed to go to completion and the absorbance of the solutions was taken at 420 nm the maximum absorption of the synthesized complex. The stoichiometry was determined from the plot of absorbance versus mole ratio [Co(O₂)Co²⁺]:[Glu]. *Kinetic measurement*

The inetic runs were carried out in aqueous hydrochloric acid medium. The ionic strength of the reaction medium was maintained by the addition of NaCl. The kinetics were followed spectrophotometrically in a manner as described by Iyun *et al.*, (1995) under pseudo first order conditions. The concentration of glutamic acid (Glu) was at least 50-fold in excess over the μ -peroxobridged binuclear cobalt(III) complex concentration. The [H⁺], ionic strength and the temperature (27 ± 1°C) were kept constant (Table 1). Pseudo-



first order rate constants for the reactions were obtained from the plots of the logarithm of absorbance difference (i.e. log (A_t - A_x) against time, where A_t is the absorbance at time t, while A_x is the absorbance at infinity.

Table 1: Pseudo-first order and second order rate constant

for the redox reaction of Glutamic acid by Co(O₂)Co²⁺ in

Table 2: Dependence of rate constant by cations and anions for the redox reaction of the $Co(O_2)Co^{2+}$. Glutamic acid system at I = 0.5 mol dm⁻³ (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O_2)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻²

aqueous HCl medium, $\lambda_{max} = 420$ nm, T = 27 ± 1 °C, I							
	<u>0.50 mol di</u> 10 ³ [Glu] (mol dm ⁻³)	<u>m⁻³ (NaCl), [</u> 10 ² [H ⁺] (mol dm ⁻³)	$\frac{Co(O_2)Co^{2+}}{10 [I]}$	$\frac{ =1.4 \text{ x} }{10^3 \text{ k}_1}$	$\frac{10^{-4} \text{ mol dm}^{-3}}{\text{k}_2}$		
•				(8)			
	4.2	1.0	5.0	3.57	0.85		
	7.0	1.0	5.0	6.09	0.87		
	9.8	1.0	5.0	8.62	0.88		
	12.6	1.0	5.0	10.83	0.86		
	15.4	1.0	5.0	14.01	0.91		
	18.2	1.0	5.0	16.19	0.89		
	21.0	1.0	5.0	18.06	0.86		
	15.4	0.8	5.0	12.94	0.83		
	15.4	1.0	5.0	14.01	0.91		
	15.4	1.2	5.0	17.40	1.13		
	15.4	1.4	5.0	21.71	1.41		
	15.4	1.6	5.0	24.33	1.58		
	15.4	1.8	5.0	27.10	1.76		
	15.4	2.0	5.0	27.87	1.81		
	15.4	1.0	1.0	3.69	0.24		
	15.4	1.0	3.0	5.08	0.33		
	15.4	1.0	5.0	14.01	0.91		
	15.4	1.0	7.0	15.70	1.02		
	15.4	1.0	9.0	17.40	1.13		

Hydrogen ion concentration

1.0

1.0

154

15.4

The effect of changes in the hydrogen ion concentration on the reaction rate was investigated by keeping the concentration of the reactants constant while varying the hydrogen ion concentration in the range $(1.0 - 2.0) \times 10^{-2}$ mol dm⁻³ (below this range, the reaction was too slow while above this range, the reaction was too fast to be monitored and µperoxobridged binuclear cobalt(III) complex was unstable). Ionic strength was maintained at 0.50 mol dm⁻³ (NaCl). Order of the reaction with respect to acid concentration was obtained as the slope of the plot of logk₁ against log[H⁺]. Variation of acid dependent rate constant with [H⁺] was obtained by plotting k₂ against [H⁺].

1.1

1.2

1971

20.63

1 28

1.34

Effect of ionic strength and dielectric constant

The effect of the ionic strength on the rate of the reaction was studied over a range of $(1.0 - 1.2) \times 10^{-1}$ mol dm⁻³ using NaCl while other reaction conditions were kept constant. The effect of dielectric constant on the rate of the reactions was investigated by varying the acetone-water mixture, while all other conditions were kept constant. A plot of logk₂ against 1/D gives the relationship on the rate of reaction of the dielectric constant D.

Added ions

The effect of added ions on the reaction rate was observed by the addition of $(40.0 - 140.0) \times 10^{-3}$ mol dm⁻³ of ions (Mg²⁺, Ca²⁺, HCOO⁻, CH₃COO⁻) (Table 2) while keeping concentration of the μ -peroxobridged binuclear cobalt(III) complex, glutamic acid (Glu) and hydrogen ion constant as found in Idris (2005).

$10^{3}[C_{2}^{2+1}]$ (mol dm ⁻³)	10 ³ k ₁	k ₂			
10° [Ca ⁻⁺] (mol dm ⁻)	(s ⁻¹)	(dm ³ mol ⁻¹ s ⁻¹)			
00.0	14.01	0.91			
40.0	19.25	1.25			
60.0	24.17	1.57			
80.0	28.33	1.84			
100.0	29.56	1.92			
120.0	36.34	2.36			
140.0	37.57	2.44			
10 ³ [Mg ²⁺] (mol dm ⁻³)					
00.0	14.01	0.91			
40.0	15.70	1.02			
60.0	18.94	1.23			
80.0	24.17	1.57			
100.0	26.95	1.75			
120.0	28.02	1.82			
140.0	32.80	2.13			
10 ³ [HCOO ⁻] (mol dm ⁻³)					
00.0	14.01	0.91			
40.0	15.55	1.01			
60.0	23.71	1.54			
80.0	26.18	1.70			
100.0	32.18	2.09			
120.0	34.03	2.21			
140.0	35.11	2.28			
10 ³ [CH ₃ COO ⁻] (mol dm ⁻³)					
00.0	14.01	0.91			
40.0	17.71	1.15			
60.0	19.09	1.24			
80.0	23.40	1.52			
100.0	28.33	1.84			
120.0	29.56	1.92			
140.0	30.49	1.98			

Free radical test

A partially oxidizing reaction mixture containing various concentrations of the cobalt(III) complex of succinimide, glutamic acid (Glu) and hydrogen ion, about 2 g of acrylamide was added followed by the addition of excess methanol. The control experiment was also carried out by adding no acrylamide to the solution of the reaction mixture to know if there will be formation of any gel in the reaction mixture.

Results and Discussion

Stoichiometric studies indicated that one mole of $C_O(O_2)Co^{2+}$ reacted completely with one mole of [Glu] (Fig. 1). This gives the overall equation of the reaction to be as represented by Equation 1

 $NH_2RCHCOOH + Co(O_2)Co^{2+} + 2H^+$ $RCOCOOH + 2Co^{2+} + NH_3 + H_2O$ 1

Where: $R = C_3H_5$ for Glu

The plot of log(A_t - A_∞) versus time for the C₀(O₂)Co²⁺ - Glu reaction was linear to about 90% of the reaction (Fig. 2). The linearity of the plots indicate that the reaction is first order



with respect to $Co(O_2)Co^{2+}$. This is also supported by the constancy of k_2 values (Table 1), meaning that the reaction is first order with the concentration of the succinimide complex. A plot of logk₁ versus log[Glu] (Fig. 3) was linear with a slope of 1.02 correlation, r = 0.99) showing that the reaction is also first order with respect to [Glu]. Therefore the reaction is second order overall at constant [H⁺]. Thus, the rate equation for the reaction can be written as shown in Equation 2

$$-\frac{d[Co(O_2)Co^{2+}]}{dt} = k_2[Co(O_2)Co^{2+}][Glu] = 2$$

The order of one in both reactants in the reaction conforms to some already reported redox reaction of glutamic acid (Glu) by N-bromobenzenesulphonamide (Puttaswamy and Nirmala, 2011), N-bromophthalimide (Alhaji and Sofiya, 2011), and Bi(V) phosphate (Avinash *et al.*, 2012).



Figure 1: Plot of absorbance versus mole ratio for $Co(O_2)Co^{2+}$. Glu system at $[Co(O_2)Co^{2+}] = 1.4 \times 10^{-4} \text{ mol } dm^{-3}$, $\lambda_{max} = 420 \text{ nm}$, $[H^+] = 1.0 \times 10^{-2} \text{ mol } dm^{-3}$ I = 0.5 mol dm⁻³, (NaCl), T = 27 ± 1°C, [Glutamic acid] = (7.0 - 35.0) x 10^{-5} \text{ mol } dm^{-3}



Fig. 2: Typical pseudo-first order plot for the redox reaction of $Co(O_2)Co^{2+}$. Glu at λ_{max} = 420 nm, T = 27 ± 1 °C, I = 0.50 mol dm⁻³ (NaCl), [Co(O_2)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³



Fig. 3: Plot of log k_1 versus log [Glu] at $\lambda_{max} = 420$ nm, T = 27 ± 1°C, I = 0.50 mol dm⁻³ (NaCl), [Glu] = (4.2 - 21.0) x 10⁻⁴ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³

The rate constant of the reaction increases with increase in $[H^+]$ and the plot of $\log_{k_1} \operatorname{versus} \log[H^+]$ (Fig. 4) gave a slope of 0.93 indicating that the reaction is first order with respect to $[H^+]$. Also the plot of k_2 versus $[H^+]$ (Fig. 5) was linear with positive intercept in the acid concentration range investigated and the overall rate equation can be represented by Equation 3 $= d[\Omega_0(\Omega), \Omega_0^{2+1}]$

$$\frac{-a[\text{Co}(\text{O})_2\text{Co}^{-1}]}{dt} = (a + b[H^+])[\text{Co}(\text{O})_2\text{Co}^{2+}][Glu]$$
3

This Observation suggests that the oxidation-reduction reaction occur via acid-dependent and acid-independent pathways. Changes in the ionic strength in the range $(1.0 - 1.2) \times 10^{-1}$ mol dm⁻³ of the reaction medium also increased the reaction rate (Table 1, Fig. 6). Logk1 vs \sqrt{I} showed a positive brownsted-Debye salt effect suggests that like charges are reacting at the rate determining step (Iyun *et al.*, 1993).



Fig. 4: Plot of log k_1 versus log $[H^+]$ at $\lambda_{max} = 420$ nm, $T = 27 \pm 1^{\circ}C$, I = 0.50 mol dm⁻³ (NaCl), $[H^+] = (0.8 - 2.0) \times 10^{-3}$ mol dm⁻³, $[Glu] = 15.4 \times 10^{-3}$ mol dm⁻³, $[Co(O_2)Co^{2+}] = 1.4 \times 10^{-4}$ mol dm⁻³









Fig. 6: Plot of log k_1 versus \sqrt{I} at $\lambda_{max} = 420$ nm, $T = 27 \pm 1$ °C, $I = (1.0 - 1.3) \times 10^{-1}$ mol dm⁻³ (NaCl), $[H^+] = (0.8 - 2.0) \times 10^{-3}$ mol dm⁻³, [Glu] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³



Fig. 7: Plot of k_2 (dm³mol⁻¹s⁻¹) versus [Ca²⁺] (mol dm⁻³) at I = 0.5 mol dm⁻³ (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻²



Fig. 8: Plot of k_2 (dm³mol⁻¹s⁻¹) versus [Mg²⁺] (mol dm⁻³) at I = 0.5 mol dm⁻³ (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻²



Fig. 9: Plot of k_2 (dm³mol⁻¹s⁻¹) versus [HCOO⁻] (mol dm⁻³) at I = 0.5 mol dm⁻³ (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻²



[CH₃COO⁻] (mol dm⁻³)

Fig. 10: Plot of k_2 (dm³mol⁻¹s⁻¹) versus [CH₃COO⁻] (mol dm⁻³) at I = 0.5 mol dm⁻³ (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻²





Fig. 11: Michaelis-menten plot: Plot of $1/k_1$ (s⁻¹) versus $1/[Glutamic acid] dm^3 mol^{-1}$ at I = 0.5 mol dm⁻³, (NaCl), T = 27 ± 1°C, [Glutamic acid] = 15.4 x 10⁻³ mol dm⁻³, [Co(O₂)Co²⁺] = 1.4 x 10⁻⁴ mol dm⁻³, $\lambda_{max} = 420$ nm, [H⁺] = 1.0 x 10⁻² mol dm⁻³

Added Ca^{2+} , Mg^{2+} , CH_3COO^- and $HCOO^-$ on the rate of the reaction (Table 2, Fig. 7 - 10) were studied. There was an enhancement of the reaction due to the presence of added anions which also indicate positive charge species are

involving in the rate determining step. Also, the enhancement of the anions suggests that outer-sphere mechanism is probably in operation in this reaction (Huck and Wieghardt, 1980). When acrylamide solution was added to initiate polymerization for the presence of free radicals, the acrylamide did not polymerize either the reactant alone or the reaction mixture. The lack of polymerization even after adding excess methanol indicates the probable absence of free radicals in the reaction mixture. This suggests that the free radicals are not involved in this reaction and this indicate an outer-sphere mechanism through the origin. Also, the result of the spectroscopic studies indicate no significant shift from the absorption maxima 420 nm characteristic of Co(O₂)Co²⁺. This suggests that formation of an intermediate complex during the course of this reaction is very unlikely. This further support the possibility of the outer-sphere mechanism in operation in this reaction. Based on the results obtained in this investigation, a plausible mechanism that is in consistent with the kinetic data gathered for the $Co(O_2)Co^{2+}$ - Glu reaction is proposed in Equation 4-8 The acid-dependent pathway involves the protonation of the amino acid in Equation 4 and the protonated species forms an adduct with the complex in Equation 5. The adduct decays in Equation 6 to give products. While the acid-independent pathway involves the formation of an adduct of the amino acid and the complex in Equation 7, which also decays in Equation 8 to give products.

$$\begin{array}{c} H_{2}NRCHCOOH + H^{+} & \stackrel{K_{1}}{\longrightarrow} H_{3}^{+}NRCHCOOH & 4 \\ Co(O_{2})Co^{2+} + H_{3}NRCHCOOH & \stackrel{k_{2}}{\longrightarrow} [Co(O_{2})Co^{2+}, H_{3}NRCHCOOH] & 5 \\ [Co(O_{2})Co^{2+}, H_{3}NRCHCOOH] & \stackrel{k_{3}}{\longrightarrow} 2Co^{2+} + NH_{3} + RCOCOOH & 6 \\ Co(O_{2})Co^{2+}, H_{2}NRCHCOOH] & \stackrel{K_{5}}{\longrightarrow} 2Co^{2+} + NH_{3} + RCOCHOOH] & 7 \\ [Co(O_{2})Co^{2+}, H_{2}NRCHCOOH] & \stackrel{k_{5}}{\longrightarrow} 2Co^{2+} + NH_{3} + RCOCHOO^{-} \\ Equation 5 and 9 are proposed as the rate determining steps \\ From equations 5 and 8 \\ Rate = k_{2}[Co(O_{2})Co^{2+}][H_{3}^{+}NRCHCOOH] + k_{5}[Co(O_{2})Co^{2+}, H_{2}NRCHCOOH] & 9 \\ From equations 4 and 7 \\ [H_{3}^{+}NRCHCOOH] = K_{4}[Co(O_{2})Co^{2+}][H_{2}NRCHCOOH] & 11 \\ Substituting equations 10 and 11 into 9 \\ Rate = K_{1}k_{2}[Co(O_{2})Co^{2+}][H_{2}NRCHCOOH][H^{+}] + K_{4}k_{5}[Co(O_{2})Co^{2+}][H_{2}NRCHCOOH] & 12 \\ (K_{4}k_{5} + K_{1}k_{2}[H^{+}])[Co(O_{2})Co^{2+}][H_{2}NRCHCOOH] & 13 \end{array}$$

Where: $R = C_3H_5$ for Glu, equation 13 is similar to equation 3 with $K_4k_5 = a = 14.01 \times 10^{-3} s^{-1}$ and $K_1k_2 = b = 0.91 dm^3 mol^{-1} s^{-1}$



Conclusion

It can be concluded that the $Co(O_2)Co^{2+}$ - Glu reaction shows a first order with respect to all the reactancts and two pathways: protonated and unprotonated acid dependent. The outer-sphere mechanism in which the succinimide complex itself is the oxidizing species and a plausible mechanism satisfying the various kinetic parameters are proposed

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