https://chemclassjournal.com/
ChemClass Journal Vol. 9 Issue 1 (2025); 348-359
e-ISSN:0198-2734 p-ISSN:0198-5680
doi.org/10.33003/chemclas_2025_0901/028

Kinetics and Mechanism of the Reduction of Potassium Trisoxalatoferrate (III) by Gallic Acid in Aqueous Hydrochloric Acid Medium

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Abstract

Some of the industrial or chemical importance of the reaction between gallic acid (GH) and potassium trisoxalatoferrate (III), ($K_3[Fe(C_2O_4)_3]$ is that the product(s) of the redox reaction is used to extend the shelf life of food products, corrosion prevention and pollutant degradation. The redox kinetic and mechanistic studies of the reaction between gallic acid (GH) and potassium trisoxalatoferrate (III), ($K_3[Fe(C_2O_4)_3]$ has been investigated in aqueous hydrochloric acid medium. The stoichiometry of the reaction determined by the mole ratio method was observed to be 1:1. The observed rate of oxidation showed first order dependence with respect to both the complex and gallic acid concentrations. The experimental data for the redox system are consistent with the following rate law:

$$\frac{-d[GH]}{dt} = (a + b[H^+])\ [GH][Fe(C_2O)_3{}^{3-}]$$

where a = 5.7×10^{-3} $mol^{-1}\,dm^3\,s^{-1}$, b = 1.81×10^{-2} $mol^{-2}\,dm^6\,s^{-1}$ at 420 nm and 29.0 ± 1.0 °C.
The rate of reaction was found to increase with increase in ionic strength and hydrogen ion concentration in the salt and acid concentration range used. The rate of reaction was also observed to increase with decreasing dielectric constant of the reaction medium and the addition of CH_3COO^-, $HCOO^-$, $Li^+$and K^+ catalyzed the rate of the reaction. The Michaelis-Menten plot of $1/k_{obs}$ versus 1/[GH] gave a straight line with an insignificant positive intercept. The addition of acrylamide to the partially reacted mixture produced a gel in the presence of excess methanol. Based on the strength of the experimental data, a plausible mechanism involving outer sphere electron transfer pathway has been proposed for the redox reaction between gallic acid and potassium trisoxalatoferrate (III). The findings in this study would give more insights into the redox behaviors of the redox species and its application as a pharmaceutical and personal care product among others.

Keywords: acrylamide, gallic acid, ionic strength, kinetics, mechanism, outer-sphere, potassium trisoxalatoferrate (III).

Introduction

Potassium trisoxalatoferrate (III), $K_3[Fe(C_2O_4)_3]$ has been useful in many areas of co-ordination chemistry, mainly because of its spectroscopic and kinetic behaviors in solution and in certain cases its resolution into enantiomers [1]. The complex, for example, has been used in photography, analytical chemistry, medicine and actinometry. The potential use of the complex as an antibacterial agent has been reported elsewhere [2]. The electron-transfer reaction of the complex with many substrates have been receiving attention. For example, the redox reaction between the complex and $S_2O_3^{2-}$, MnO_4^- and SO_4^{2-}, have been reported [1,2,3].

Gallic acid (3, 4, 5-trihydroxybenzoic acid) is one of the most abundant phytochemical in nature, the gallate moiety being a key component of many functional phytochemicals [4]. This acid is used as antioxidant in cosmetics, pharmaceutical and food industry [5,6]. It is also extensively used in tanning, ink dyes and the manufacturing of paper [4]. Several studies have revealed that gallic acid has various pharmacological and biological activities, including antibacterial, antitumor and antiviral properties [7].

A large amount of wastewater containing gallic acid is discharged into the aquatic environment during the production processes of drugs, foods and cosmetics. Related researches have demonstrated that gallic acid in water leads to the death of aquatic organisms and deteriorates water quality [8]. Therefore, it is necessary to seek effective methods for removing gallic acid from wastewater.

Details of the kinetics and mechanism of the redox reaction between gallic acid and potassium trisoxalatoferrate (III) has not appeared in literature to the best of our knowledge and it is in this view that we seek to study the title reaction as our contribution to understand the redox behavior of the redox partners, establish the kinetic data between them and to propose a plausible mechanism for the reaction.

Experimental

Materials and Reagents

All the reagents used were of analytical grade and were used without further purification unless otherwise stated. Solutions of all the reagents used were made in distilled water. All the kinetic runs and other runs were carried out using a UK Jenway 6300 UV-visible spectrophotometer. Reagent grade HCl and NaCl were used to adjust the acidity and ionic strength of the reaction media respectively.

Stoichiometric Studies

Spectrophotometric titration using the mole ratio method was used to determine the stoichiometry of the reaction. Solutions containing a fixed concentration of $[Fe(C_2O_4)_3^{3-}] = (3.5 \times 10^{-3}$ mol dm^{-3}) and varying concentration GH $(3.5 – 12.3) \times 10^{-2}$ mol dm^{-3} were reacted at $[H^+] = 0.15$ mol dm^{-3} and I = 0.5 mol dm^{-3} (NaCl) and allowed to stand for 24 hours for the reaction to go to completion. The stoichiometry was determined from the plot of absorbance against mole ratio [1,3].

Kinetic Measurements

Rate data for the titled reaction were obtained by monitoring absorbance changes at $\lambda_{max} = 420$ nm under pseudo-first conditions with the [reductant] in at least 10-fold excess over the [oxidant] using a UK Jenway 3600 UV-visible spectrophotometer under the stated experimental conditions.

Results and Discussion

Stoichiometry

Spectrophotometric titration for the redox system revealed that one mole of the oxidant was consumed by one mole of the reductant and the observation is represented in equation (1) and Figure 1.

$$Fe^{3+} + GH \longrightarrow Fe^{2+} + H^{+} + G \ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where G = quinones

However, 1:3 and 2:3 stoichiometries have been reported for the redox reaction between GH/MnO_4^- [9] and GH/ BrO_3^- [5].

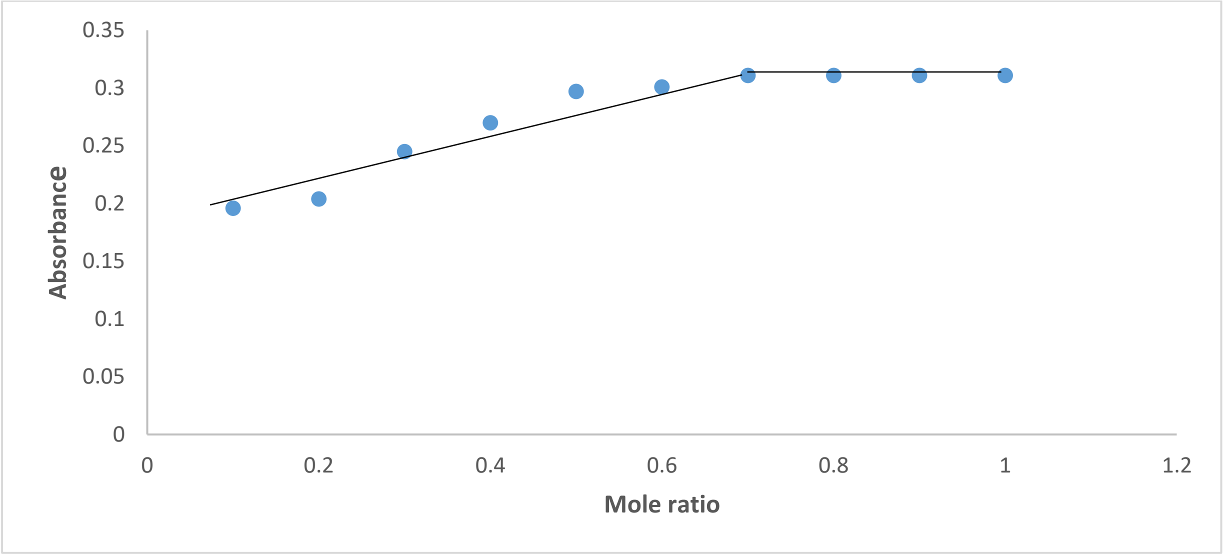

Figure 1: Plot of absorbance versus mole ratio for the reduction of [$Fe(C_2O_4)_3^{3-}$] by gallic acid at [$Fe(C_2O_4)_3^{3-}$] = 3.5 × 10^{-3} mol dm^{-3}, I = 0.5 mol dm^{-3} (NaCl), [H^+] = 0.15 mol dm^{-3} λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Product Analysis

Fe^{2+}, identified as one of the products by the addition of freshly prepared $K_3[Fe(CN)_6]$ to the completely reacted mixture which gave a deep blue-green solution indicating the presence of iron(II) [3]. Hydrogen peroxide, semiquinones and quinones have been reported as products of the oxidation of gallic acid [4,10]. Qualitative analysis was carried out to test for the presence of quinones. The formation of red colour to the reaction mixture on addition of concentrated H_2SO_4 indicate the presence of quinones [11].

Determination of Order of the Reaction

Pseudo-first order plots of log(A_o-A_t) against time were linear to more than 98 % of the extent of the reaction (Figure 2), indicating first order with respect to the complex, suggesting that there is no product inhibition [12]. A plot of log k_{obs} against log [GH] was linear with a slope of 0.97 (Figure 3) indicating first order in GH and second order overall. Values for k_{obs} and k_2 are presented in Table

1. Second order rate constants, k_2 was calculated from $k_2 = k_{obs}/[GH]$ and were observed to be fairly constant (Table 1). This further indicates that the reaction is indeed first order, with respect to [GH]. Second order kinetics was reported for the oxidation of gallic acid by chromium (VI) [13]. Order of reaction less than unity has been reported for GH/ BrO_3^- system [5].

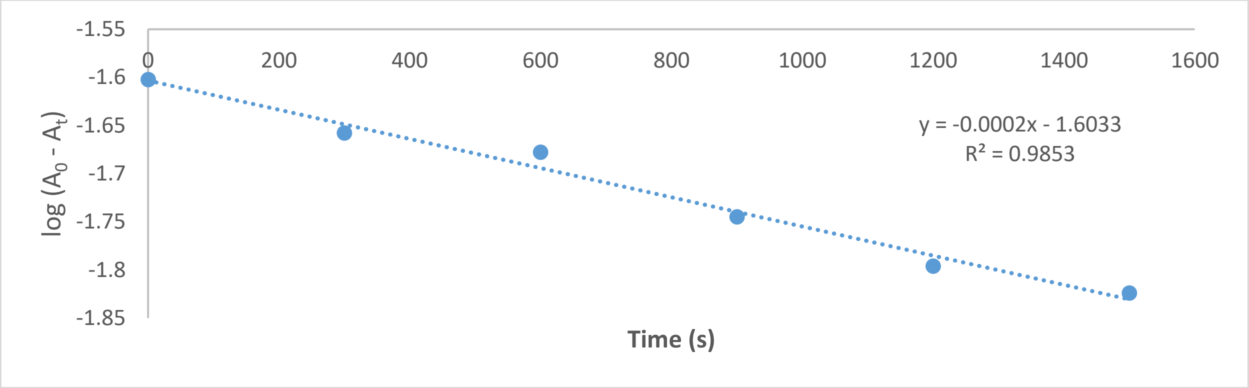

Figure 2: Typical pseudo-first order plot of log (A_o-A_t) versus time for the reduction of [$Fe(C_2O_4)_3^{3-}$] by gallic acid at [$Fe(C_2O_4)_3^{3-}$] = 3.5 × 10^{-3} mol dm^{-3}, I = 0.5 mol dm^{-3} (NaCl), [H^+] = 0.15 mol dm^{-3} λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Therefore, rate law is given by equation:

$$\frac{-d[GH]}{dt} = k_2[Fe(C_2O_4)_3^{3-}]\,[GH] \qquad \ldots\ldots 2$$

$k_2 = (8.84 \pm 0.06) \times 10^{-2}\ dm^3\ mol^{-1}\ s^{-1}$.

Table 1: Pseudo-first and second order rate constants for the oxidation of gallic acid by $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}] = 3.5 \times 10^{-3}$ mol dm^{-3}, I = 0.5 mol dm^{-3}(NaCl), [H] = 0.15 mol dm^{-3}, λ_{max} = 420 nm and T = 29 ± 0.1 °C.

10^2 [GH] mol dm^{-3}	10^1 [H$^+$] mol dm^{-3}	I, 10^1 mol dm^{-3} (NaCl)	10^3 k_{obs} s^{-1}	10^2 k_2 dm^3 mol^{-1} s^{-1}
3.5	1.5	5	3.1	8.86
5.3	1.5	5	4.5	8.85
7.0	1.5	5	6.2	8.86
8.8	1.5	5	7.7	8.80
10.5	1.5	5	8.9	8.50
12.3	1.5	5	10.3	8.40
12.3	0.7	5	0.5	0.71
12.3	1.4	5	1.2	0.86
12.3	2.1	5	2.2	1.11
12.3	2.8	5	2.9	1.04
12.3	3.5	5	4.4	1.25
12.3	1.5	1.4	0.4	0.29
12.3	1.5	2.8	1.3	0.46
12.3	1.5	4.2	2.3	0.56
12.3	1.5	5.6	2.9	0.63
12.3	1.5	7.0	4.9	0.70
12.3	1.5	8.4	6.3	0.75

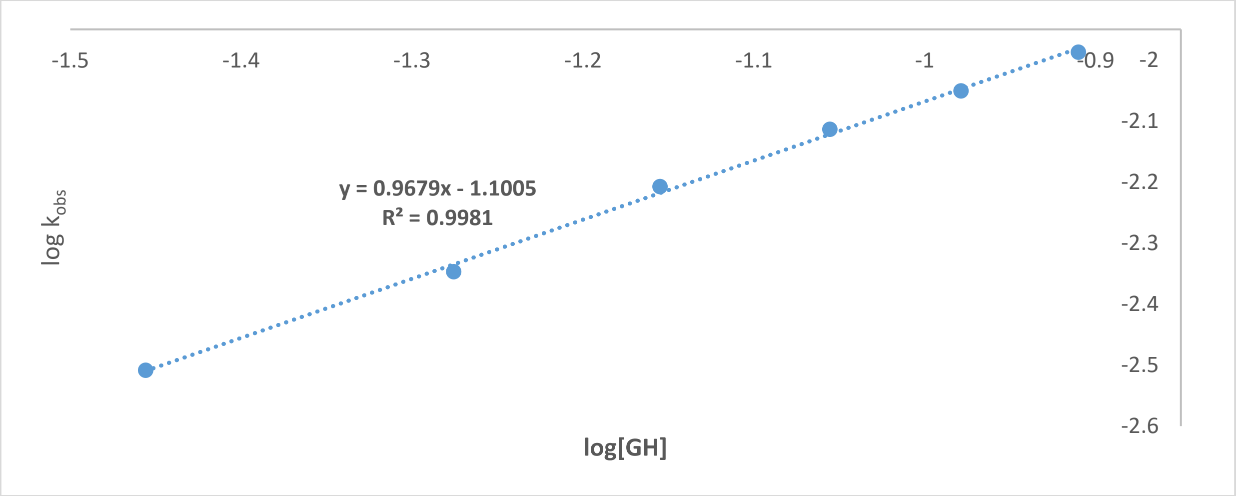

Figure 3: Plot of log k_{obs} versus log [GH] at for the reduction of $[Fe(C_2O_4)_3^{3-}]$ by gallic Acid at $[Fe(C_2O_4)_3^{3-}] = 3.5 \times 10^{-3}$ mol dm^{-3}, I = 0.5 mol dm^{-3} (NaCl), [H$^+$] = 0.15 mol dm^{-3} λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Acid Dependence

The effect of $[H^+]$ on the reaction rate was monitored by varying the concentration of HCl within the range of $(0.7 – 3.5) \times 10^{-1}$ mol dm^{-3}, $[Fe(C_2O_4)_3^{3-}]$ = 3.5×10^{-3} mol dm^{-3}, [GH] = 0.035 mol dm^{-3}, I = 0.5 mol dm^{-3}(NaCl), λ_{max} = 420 nm and T = 29.0 ± 0.1°C. Under these conditions, the rate of the reaction increased with increase in concentration of hydrogen ion. This acid catalysis has been reported to be arising from a pre-equilibrium or pre-protonation step before the electron transfer reaction [3,15]. Acid dependent rate constants are shown in Table 1 and the acid dependent rate equation is given as:

$$k_2 = a + b\,[H^+] \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad 3$$

The rate equation for the reaction can then be represented by equation (4):

$$\frac{-d[GH]}{dt} = \{a + b[H^+]\}[Fe(C_2O_4)_3^{3-}]\,[GH] \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \quad (4)$$

Where a = 5.7×10^{-3} dm^3 mol^{-1} s^{-1} and b = 1.81×10^{-2} dm^6 mol^{-2} s^{-1} and R^2 = 0.86, obtained from the plot of k_2 against $[H^+]$ as represented in Figure 4. This implies that the reaction proceeded through acid dependent and acid independent pathways.

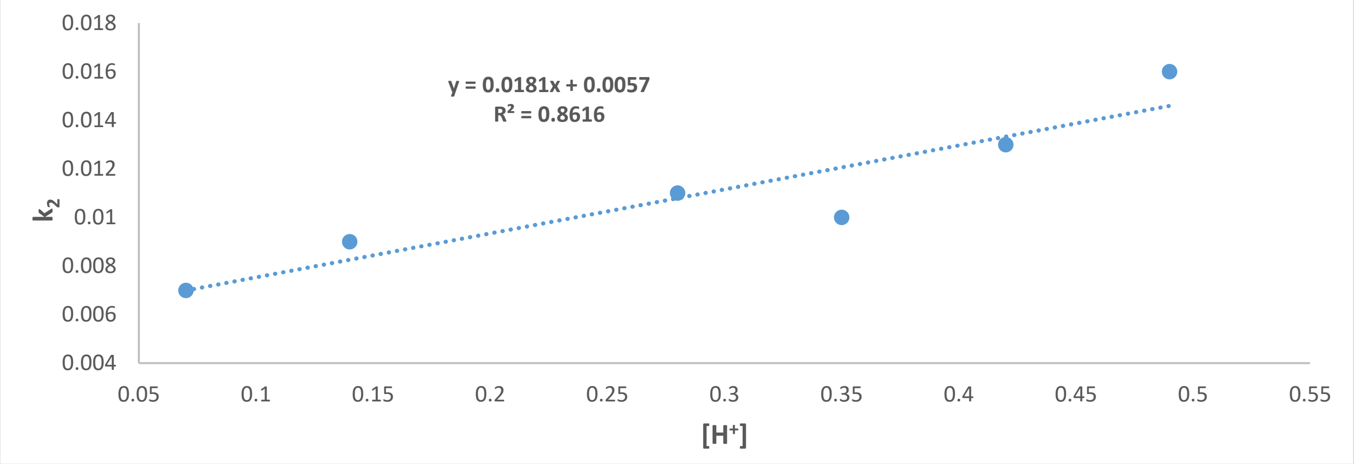

Figure 4: Plot of k_2 against $[H^+]$ for the reduction of gallic acid by $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}]$ = 3.5×10^{-3} mol dm^{-3}, [GH] = 0.035 mol dm^{-3}, I = 0.5 mol dm^{-3} (NaCl), λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Effect of Ionic Strength

The effect of ionic strength on the reaction rate was studied between $(1.4 - 8.4) \times 10^{-1}$ mol dm^{-3} using various amounts of NaCl at [GH] = 0.035 mol dm^{-3}, $[Fe(C_2O_4)_3^{3-}]$ = 3.5×10^{-3} mol dm^{-3} $[H^+]$ = 0.15 mol dm^{-3}, λ_{max} = 420 nm and T = 29.0 ± 0.1°C. The results as shown in Table 1 indicated that the rate of the reaction increased with increase in ionic strength which is attributed to the fact that the redox species are of same charge [16]. A plot of log k_2

against √I was linear with a positive slope showing a positive Brønted-Debye salt effect [17] and that species carrying same charged sign in the activated complex are interacting [12,18,19]. The independence of ionic strength on reaction rate has been reported for the oxidation of gallic acid by potassium bromate [5]

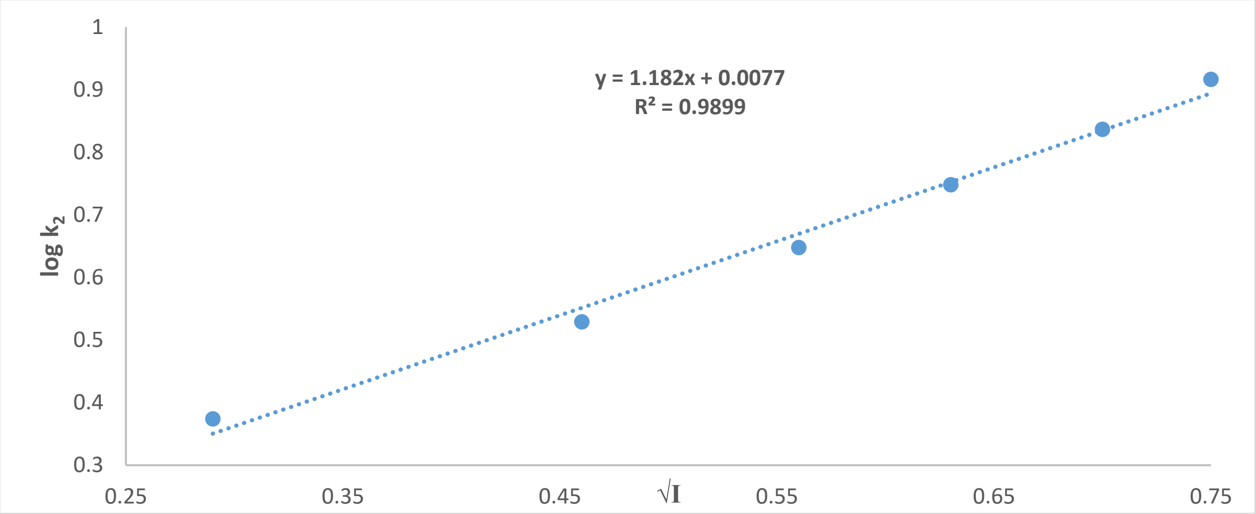

Figure 5: Plot of log k_2 versus √I for the reduction of $[Fe(C_2O_4)_3^{3-}]$ by gallic acid at $[Fe(C_2O_4)_3^{3-}] = 3.5 \times 10^{-3}$ mol dm^{-3}, [GH] = 0.035 mol dm^{-3}, $[H^+] = 0.15$ mol dm^{-3}, $\lambda_{max} = 420$ nm and T = 29 ± 0.1 °C.

Effect of Dielectric Constant (D)

Keeping other parameters constant, the effect of relative permitivity (D) was studied by adding varying portions of acetone-water mixture. The results showed increase in reaction rate as dielectric constant decreased (Table 2). Plot of log k_{obs} against 1/D, gave a positive slope implying ion-dipolar interaction in the rate determining step [2,19,20].

Table 2: Effect of dielectric constant on the oxidation of gallic acid by $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}] = 3.5 \times 10^{-3}$ mol dm^{-3}, [GH] = 0.035 mol dm^{-3}, I = 0.5 mol dm^{-3}(NaCl), [H] = 0.15 mol dm^{-3}, $\lambda_{max} = 420$ nm and T = 29 ± 0.1 °C.

D	$10^2 1/D$	$10^3 k_{obs}$, s^{-1}	$10k_2$, dm^3 mol^{-1} s^{-1}
72	1.39	0.7	5.0
63	1.59	2.2	7.8
54	1.85	3.8	9.0
48	2.08	5.5	9.9
39	2.56	7.6	10.9
30	3.33	9.5	11.3

Effect of Added Ions

Addition of varying concentrations of CH_3COO^-, $HCOO^-$, K^+ and Li^+ increased the reaction rate (Table 3). The positive catalysis of the reaction rate on addition of these ions is a characteristics of outer-sphere reaction pathway [3,21,22,23,24]. Positive catalysis of the reaction rates by added cations and anions was observed in the redox reactions between $Fe(C_2O_4)_3^{3-}$/ $S_2O_3^{2-}$ system [3] while negative catalysis on rate is consistent with the report for $Fe(C_2O_4)_3^{3-}/SO_4^{2-}$ and MnO_4^- systems [1,2].

Table 3: Effect of added ions on the oxidation of gallic acid by [$Fe(C_2O_4)_3^{3-}$] in aqueous hydrochloric acid at [$Fe(C_2O_4)_3^{3-}$] = 3.5 × 10^{-3} mol dm^{-3}, [GH] = 0.054 mol dm^{-3}, I = 0.5 mol dm^{-3}(NaCl), [H] = 0.15 mol dm^{-3}, λ_{max} = 420 nm and T = 29 ± 0.1 °C.

X	10^1[X], mol dm^{-3}	10^3 k_{obs}, s^{-1}	10^2 k_2, dm^3 mol^{-1} s^{-1}
CH_3COO^-	1.4	0.5	0.36
	2.8	1.5	0.53
	4.2	3.5	0.81
	5.6	4.8	0.86
	7.0	6.5	0.93
$HCOO^-$	1.4	0.9	0.63
	2.8	2.4	0.86
	4.2	3.8	0.90
	5.6	5.8	0.95
	7.0	7.6	1.09
K^+	1.4	0.5	0.36
	2.8	2.2	0.78
	4.2	3.5	0.83
	5.6	4.8	0.86
	7.0	6.7	0.94
Li^+	1.4	0.7	0.49
	2.8	1.9	0.68
	4.2	3.1	0.74
	5.6	4.5	0.80
	7.0	6.2	0.89

Michaelis-Menten Plot

Michaelis-Menten type plot of $1/k_{obs}$ versus 1/[GH] was linear with an insignificant intercept as shown in Figure 6. This is in accordance with reactions occurring through the outer-sphere pathway. This suggests the absence of intermediate or precursor complex formation [12,19]. Similar reaction pathway was reported for the redox reaction between $Fe(C_2O_4)_3^{3-}$/ $S_2O_3^{2-}$ and $Fe(C_2O_4)_3^{3-}/SO_4^{2-}$ systems [2,3].

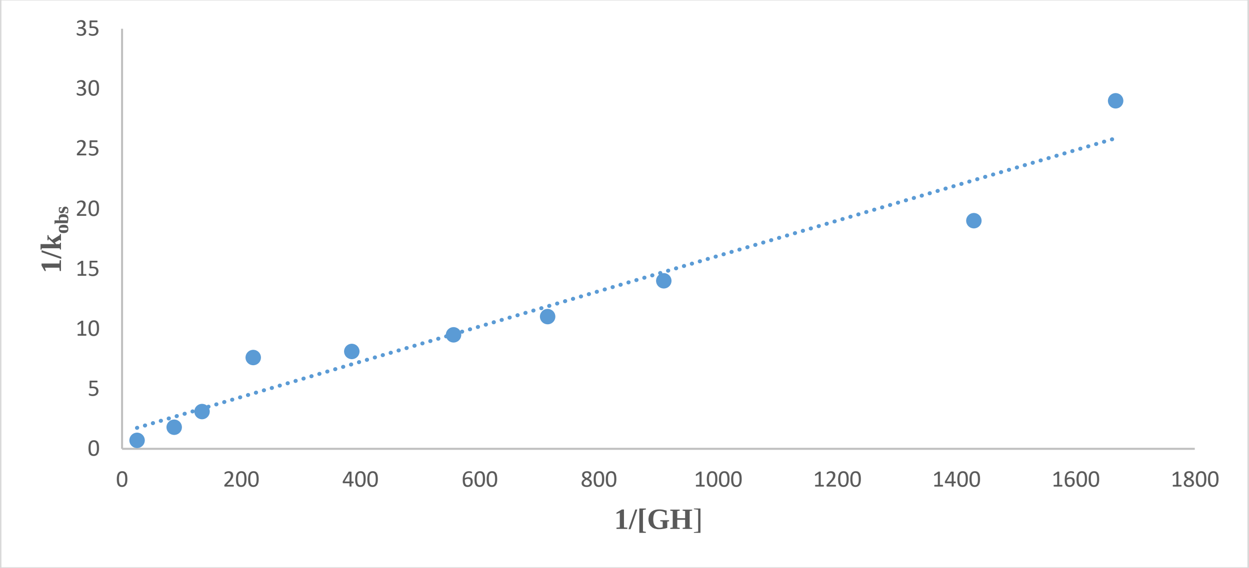

Figure 6: Plot of 1/k_{obs} versus 1/[GH] at $[Fe(C_2O_4)_3^{3-}]$ = 3.5 × 10^{-3} mol dm^{-3}, I = 0.5 mol dm^{-3} (NaCl), $[H^+]$ = 0.15 mol dm^{-3} λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Test for Free Radicals

On addition of acrylamide to partially reacted mixture of the reactants formed a gel. Excess methanol was added to the mixture and precipitate was formed. The methanol was responsible for trapping the polymer as an insoluble precipitate. This suggested that free radicals participated in this reaction as indicated by the positive polymerization test. which suggests an inner-sphere reaction pathway. [17,25]. Polymerization of acrylamide by potassium trisoxalatoferrate (III) had earlier been reported [2,19]. Absence of free radical has been reported for the oxidation of gallic acid by potassium bromate [5].

Mechanism of the Reaction

Based on the stoichiometry, observed positive acid and ionic strength dependence, anion and cation catalysis, a plausible reaction scheme has been proposed to account for the kinetic data generated for gallic acid - $[Fe(C_2O_4)_3^{3-}]$ redox system as follows:

$$[Fe(C_2O_4)_3^{3-}] + H^+ \underset{k_{-1}}{\overset{k_1}{\leftrightharpoons}} [HFe(C_2O_4)_3^{2-}] \quad \text{(fast)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (5)$$

$$[HFe(C_2O_4)_3^{2-}] + GH \overset{k_2}{\rightarrow} [HFe(C_2O_4)_3^{2-}, GH] \quad \text{(slow)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (6)$$

$$[HFe(C_2O_4)_3^{2-}, GH] \overset{k_3}{\rightarrow} Fe(C_2O_4)_3^{2-} + \text{products} \quad \text{(slow)} \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (7)$$

From equations (6) and (7), the rate equation can be written as:

$$\text{Rate} = k_2[Fe(C_2O_4)_3^{3-}] [GH] + k_3[GH][Fe(C_2O_4)_3^{2-}] \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (8)$$

From equation (5),

$$[HFe(C_2O_4)_3^{2-}] = k_1[Fe(C_2O_4)_3^{3-}][H^+] \ldots\ldots\ldots\ldots (9)$$

Substituting equation (9) into (8), we obtain:

$$\text{Rate} = k_1k_2[Fe(C_2O_4)_3^{3-}] [GH][H^+] + k_3[GH] [Fe(C_2O_4)_3^{3-}] \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (10)$$

$$\text{Rate} = (k_3 + k_1k_2[H^+]) [Fe(C_2O_4)_3^{3-}] [GH] \ldots\ldots (11)$$

Equation (11) is analogous to equation (4), where:

$a = k_3 = 5.7 \times 10^{-3}\ mol^{-1}\ dm^3\ s^{-1}$

$b = k_1k_2 = 1.81 \times 10^{-2}\ mol^{-2}\ dm^6\ s^{-1}$

Conclusion

The plausible mechanism through which the reduction of potassium trisoxalatoferrate (III) by gallic acid occurred, was arrived at by considering the following points:

(a) Table 1 and Figure 5 showed that reaction rates increased with increase in ionic strength, implicating the occurrence of outer sphere reaction pathway [12].

(b) The dependence of the reaction on dielectric constant, D, supports the formation of ion-pairs of outer-sphere characteristics [15].

(c) The Michaelis-Menten plot was linear without an appreciable intercept (Figure 6). This is in support of an outer sphere reaction mechanism.

(d) The addition of added ions (Table 3) increased the reaction rate. Ions catalysis or inhibition has been proposed to support outer sphere reaction pathway [19].

Based on these kinetic evidences (a) - (d), plausible outer-sphere mechanism has been proposed for the redox reaction between potassium trisoxalatoferrate (III) and gallic acid in accord with the acid catalysis of the reaction

Acknowledgement

We are grateful to the Department of Chemistry University of Jos, for the provision of facilities for this work and the following Technologists for their assistance in the course of the laboratory work: Mr. Yusuf Chakyin and Mr. Atsen Samuel.

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