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Kinetics and Mechanism of the Reduction of Potassium Trisoxalatoferrate (III) by 5-Sulfosalicylic Acid in Aqueous Hydrochloric Acid Medium

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Abstract

The reaction between iron (III) complexes and sulfosalicylic acid (SSH) is very important in waste water treatment, detecting the presence of phenols in pharmaceuticals and the pulp and paper industry. In this study, the kinetics and mechanism of the oxidation-reduction reaction between sulfosalicylic (SSH) acid and potassium trisoxalatoferrate(III), $K_3[Fe(C_2O_4)_3]$ has been carried out in aqueous acidic medium spectrophotometically under pseudo first-order conditions. The stoichiometric studies obtained by the mole ratio method, showed that 1 mole of SSH was oxidized for every 2 moles of $Fe(C_2O_4)_3^{3-}$ reduced. Under pseudo-first order conditions of SSH >> $Fe(C_2O_4)_3^{3-}$, the reaction was found to have first order dependence on each of the reactants, giving second order overall.

The experimental data are consistent with the rate law:

$$\frac{-d[SSH]}{dt} = (a + b[H^+])[SSH][Fe(C_2O_4)_3^{3-}]$$

with $a = 9.0 \times 10^{-3} \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$ and $b = 4.0 \times 10^{-2} \text{ dm}^6 \text{ mol}^{-2} \text{ s}^{-1}$ at 420 nm and 29.0 ± 1.0 °C.

The reaction rate was found to be directly dependent on acid concentration and decreased with increase in ionic strength of reaction system. It was also evident that decrease in dielectric constant of the reaction medium (D) enhanced the reaction rate and addition of added ions showed negative catalysis on the rate of the reaction. The reduction of potassium trisoxalatoferrate (III) is by sulfosalicylic acid is strongly rationalized in terms of absence of ion pair complex formation, free radical participation with outer-sphere reaction pathway characteristics.

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

Introduction

Potassium trisoxalatoferrate (III), $K_3[Fe(C_2O_4)_3]$ has demonstrated key roles in many areas of co-

ordination chemistry, particularly 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 [1,3,4].

5-sulfosalicylic acid, a member of pharmaceutical and personal care products, is widely and extensively used as a medical intermediate, dye, surfactant and fine chemical material. It finds applications in many industrial processes and it is continuously released into the environment via the improper discharge of the wastewater [5,6]. Because of the poor chemical oxidability and biodegradability of 5-sulfosalicylic acid, the industrial wastewater containing it is difficult to be treated by the conventional biological system [7], and this has posed threats to aquatic life [8]. Therefore, the effective removal of sulfosalicylic acid from wastewater has a significant impact on the environment.

However, not much attention has been given to the kinetic study of the redox reaction of 5-sulfosalicylic acid except that reported by [6]. In this paper, we report the kinetics and mechanism of the redox reaction of 5-sulfosalicylic acid and potassium trisoxalatoferrate (III) in aqueous hydrochloric acid medium as our contribution of providing some valuable information on the redox

pattern of both the oxidant and reductant in the industry.

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 with 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 $[Fe(C_2O_4)_3^{3-}] = (5.4 \times 10^{-3} \text{ mol dm}^{-3})$ and varying concentration SSH $(5.4 - 37.8) \times 10^{-2}$ mol dm⁻³ were reacted at $[H^+] = 0.2$ mol dm⁻³ and I = 0.3 mol dm⁻³ (NaCl) and allowed to stand for 24 hours for the reaction to go to completion. The stoichiometry was evaluated from the plot of absorbance against mole ratio [9].

Kinetic Measurements

All kinetic runs were made under pseudo-first order conditions with [SSH] in at least 10-fold excess of the [Fe(C₂O₄)₃³⁻]. The rate of the reaction was monitored by measuring the change in absorbance of the reaction mixture using a Jenway 6300 UVvisible spectrophotometer at [H⁺] = 0.2 mol dm⁻³ and I = 0.3 mol dm⁻³ (NaCl), $\lambda_{max} = 420$ nm and T = 29.0 ± 0.1°C unless otherwise stated.

Results and Discussion

Stoichiometry

The results of the spectrophotometric titration revealed a 2:1 stoichiometry and the overall reaction is given in Figure 1 and equation 1: Similar stoichiometric ratio was reported for the redox reaction between sulfosalicylic acid and diterlluratocuprate (III) [6].

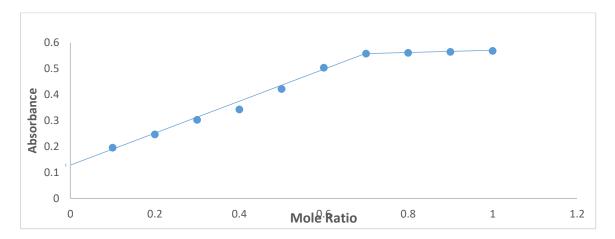


Figure 1: Plot of absorbance against mole ratio at for the reduction of $[Fe(C_2O_4)_3^{3-}]$ by sulfosalicylic acid at $[Fe(C_2O_4)_3^{3-}] = 5.4 \times 10^{-3} \text{ mol dm}^{-3}$, I = 0.3 mol dm⁻³ (NaCl), [H⁺] =0.2 mol dm⁻³ $\lambda_{max} = 420 \text{ nm}$ and T = 29 ± 0.1 °C.

Product Analysis

Fe²⁺ was identified as one of the products by the addition of freshly prepared $K_3[Fe(CN)_6]$ with the completely reacted mixture which gave a deep bluegreen solution indicating the presence of iron(II) [3]. CO₂ was also identified as another product of the oxidation of sulfosalicylic acid, which was done by its Ba(OH)₂ water test in which a white precipitate of BaCO₃ was formed. The formation of CO₂ as a product of the oxidation of SSH has been reported elsewhere [6].

Determination of Order of Reaction

Pseudo-first order plots of $log(A_o-A_t)$ versus time (Figure 2) were linear to about 95 % of the extent of the reaction indicating first order with respect to the complex. A plot of log k_{obs} versus log [SSH] (Figure 3) was also linear with a slope of approximately 1, indicating first order in [SSH] and second order overall. Values for k_{obs} and k₂ are presented in Table 1. Second order rate constants, k₂ were calculated from k₂ = k_{obs}/[SSH] were found to be fairly constant (Table 1), showing that the

zreaction is first order in [SSH]. First order dependence on rate has been reported for the oxidation of sulfosalicylic acid by dihydroxydiperiodatonickelate (IV) [10]. However, fractional order reaction has also been reported for the redox reaction between sulfosalicylic acid and ditelluratocuprate (III) [6].

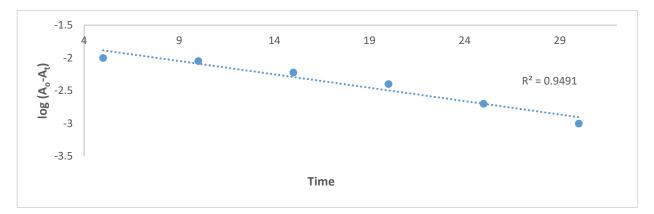


Figure 2: Typical pseudo-first order plot of log (A₀-A_t) versus time for the reduction of [Fe(C₂O₄)₃³⁻] by sulfosalicylic acid at [Fe(C₂O₄)₃³⁻] = 5.4×10^{-3} mol dm⁻³, I = 0.3 mol dm⁻³ (NaCl), [H⁺] = 0.2 mol dm⁻³ $\lambda_{max} = 420$ nm and T = 29 ± 0.1 °C.

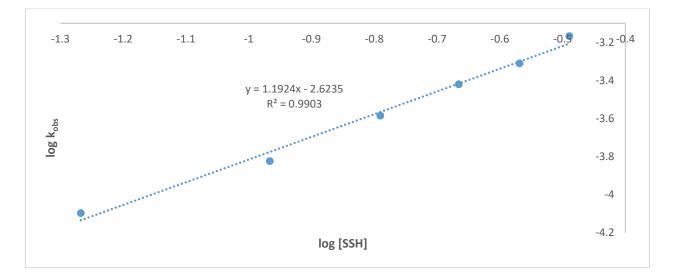


Figure 3: Plot of log k_{obs} versus log [SSH] for the reduction of [Fe(C₂O₄)₃³⁻] by sulfosalicylic acid at [Fe(C₂O₄)₃³⁻] = 5.4×10^{-3} mol dm⁻³, I = 0.3 mol dm⁻³ (NaCl), [H⁺] = 0.2 mol dm⁻³ λ_{max} = 420 nm and T = 29 ± 0.1 °C



 $k_2 = (1.6 \pm 0.2) \times 10^{-3} \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$.

Acid Dependence

The acid dependence on the rate of the reaction was monitored at constant [Fe(C₂O₄)₃³⁻], [SSH] and [I] while varying the concentration of [H⁺] (0.54 – 1.62) × 10⁻¹ mol dm³ at 29.0 ± 1.0 °C. The rate was found to increase with increase in [H⁺] (Table 1). Plot of k₂ versus [H⁺] was found to be linear with a positive slope and intercept and R² = 0.97 (Fig. 1) which indicates first order in [H⁺] and the relationship is represented by equation (3):

$$k_2 = a + b [H^+] \dots (3)$$

 $a = 0.9 \times 10^{-4} \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$ and $b = 0.4 \times 10^{-3} \text{ dm}^6 \text{ mol}^2 \text{ s}^{-1}$. The rate equation for the reaction can therefore be represented by equation (4):

$$\frac{-d[SSH]}{dt} = (a + b[H^+])[SSH][Fe(C_2O_4)_3^{3-}]$$
.....(4)

This suggests that the reaction occurred through acid dependent and acid independent pathways [11]. The acid dependence of this nature shows that there is a rapid pre-equilibrium between the protonated and non-protonated and in which both are reactive [12]. Reaction rate have been reported to increase with increase in [OH⁻] [6,10].

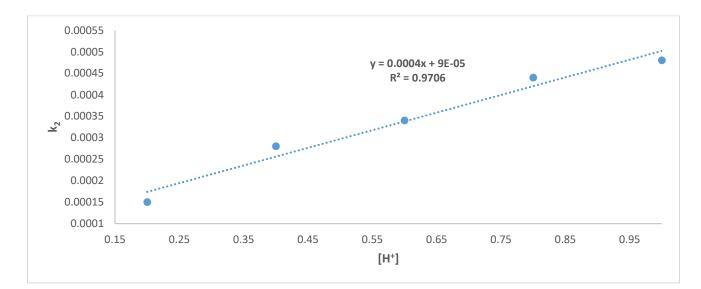


Fig. 1: Plot of k_2 versus [H⁺] for the reduction of potassium trisoxalatoferrate (III) by sulfosalicylic acid at [Fe(C₂O₄)₃³⁻] = 5.4 × 10⁻³ mol dm⁻³, [SSH] = 0.054 mol dm⁻³, I = 0.3 mol dm⁻³ (NaCl), λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Ionic Strength Dependence

The effect of ionic strength was studied in the range $(0.27 - 1.35) \times 10^{-1}$ mol dm⁻³ using NaCl and the results obtained is presented in Table 1. A close examination of the results indicates that the rate of

the reaction decrease with increase in ionic strength in the concentration range stated (Table 1). Similar observation has been reported elsewhere [6,10]. A plot of log k₂ versus \sqrt{I} (Figure 5) was linear with a negative slope, showing a negative Brønted-Debye

salt effect and suggesting the absence of intermediate complex formation and that the product of the charges at the rate determining step is negative [11,12,13,14,15]. This also indicates that the species operating at the rate determining

step are of different or unlike charges [16], the negative charge would tend to reduce the ability of ion to encounter the other reactant, therefore suppressing the reaction [12,17,18].

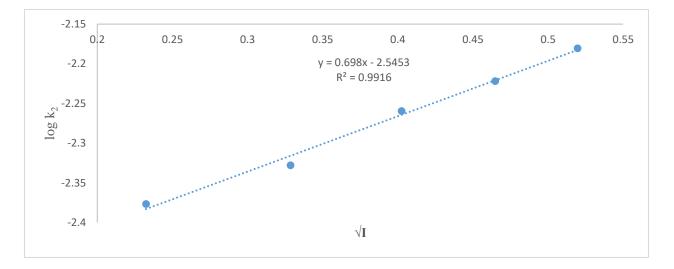


Figure 5: Plot of log k₂ versus \sqrt{I} for the reduction of [Fe(C₂O₄)₃³⁻] by sulfosalicylic acid at [Fe(C₂O₄)₃³⁻] = 5.4 × 10⁻³ mol dm⁻³, [SSH] = 0.054 mol dm⁻³, [H⁺] = 0.2 mol dm⁻³, $\lambda_{max} = 420$ nm and T = 29 ± 0.1 °C.

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10 ² [SSH] mol dm ⁻³	10 ¹ [H ⁺] Mol dm ⁻³	I, 10 ¹ Mol dm ⁻³ (NaCl)	10 ⁴ k _{obs} s ⁻¹	10 ² k ₂ dm ³ mol ⁻¹ s ⁻¹
5.4	2	3	0.8	0.15
10.8	2	3	1.5	0.14
16.2	2	3	2.6	0.16
21.6	2	3	3.8	0.18
27.0	2	3	4.9	0.18
32.4	2	3	6.8	0.20
5.4	0.54	3	1.5	0.28
5.4	0.81	3	2.8	0.35
5.4	1.08	3	4.5	0.42
5.4	1.35	3	6.9	0.51
5.4	1.62	3	9.5	0.59
5.4	2	0.27	19.2	7.11
5.4	2	0.54	15.9	2.84
5.4	2	0.81	12.4	1.53
5.4	2	1.08	9.2	0.85
5.4	2	1.35	6.6	0.48

Table 1: Pseudo-first and second order rate constants for the reduction of potassium trisoxalatoferrate (III) by sulfosalicylic acid at $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}] = 5.4 \times 10^{-3}$ mol dm⁻³, I = 0.3 mol dm⁻³(NaCl), [H] = 0.2 mol dm⁻³, $\lambda_{max} = 420$ nm and T = 29 ± 0.1 °C

Dielectric Constant Dependence

The effect of relative permittivity, D, was studied in solution containing varying volume of acetone-water mixture while keeping other conditions (ionic strength, $[H^+]$, $[Fe(C_2O_4)_3^{3-}]$ and [SSH]) constant and the result obtained is as presented in Table 2. It was observed that the rate of the reaction was

enhanced as dielectric constant, D decrease. This implies that species of opposite charges are operating at the rate determining step [12,18,25]. Similar enhancement of rate has been reported for $Fe(C_2O_4)_3^{3-7}$ S₂O₃²⁻, MnO⁴⁻ and SO₄²⁻ systems [1,3,4].

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Table 2: Effect of dielectric constant on the reduction of potassium trisoxolatoferrate (III) by sulfosalicylic acid at $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}] = 5.4 \times 10^{-3} \text{ mol dm}^{-3}$, $[SSH] = 0.054 \text{ mol dm}^{-3}$, $I = 0.3 \text{ mol dm}^{-3}$ (NaCl), $[H] = 0.2 \text{ mol dm}^{-3}$, $\lambda_{max} = 420 \text{ nm and } T = 29 \pm 0.1 ^{\circ}C$.

D	10 ² 1/D	$10^4 k_{obs}, s^{-1}$	10^4 k ₂ , dm ³ mol ⁻¹ s ⁻¹
66	1.52	2.6	5.2
57	1.75	2.8	3.5
48	2.08	3.4	3.1
39	2.56	4.0	2.8
30	3.33	4.4	2.5
21	4.76	4.8	2.4

Effect of Added Ions

Results of the effect of added ions as shown in Table 3 indicated that the addition of varying concentrations of CH_3COO^- , $HCOO^-$, Ca^{2+} and Mg^{2+} gradually decreased the reaction rate. The

inhibitive effect by these ions shows the characteristics of outer-sphere reaction pathway [1,12,15,19,20,21]. This is consistent with the report of [21,22].

Table 3: Effect of added ions on the oxidation of sulfosalicylic acid by $[Fe(C_2O_4)_3^{3-}]$ in aqueous hydrochloric acid at $[Fe(C_2O_4)_3^{3-}] = 5.4 \times 10^{-3} \text{ mol dm}^{-3}$, $[SSH] = 0.054 \text{ mol dm}^{-3}$, $I = 0.3 \text{ mol dm}^{-3}$, $I = 0.3 \text{ mol dm}^{-3}$, $I = 0.2 \text{ mol dm}^{-3}$, $\lambda_{max} = 420 \text{ nm}$ and $T = 29 \pm 0.1 \text{ °C}$.

	1 2	4	2 2 1 1
Х	10^{1} [X], mol dm ⁻³	$10^4 k_{obs}, s^{-1}$	$10^2 \text{ k}_2, \text{ dm}^3 \text{ mol}^{-1} \text{ s}^{-1}$
CH ₃ COO ⁻	0.81	5.7	7.00
	1.62	5.4	3.30
	2.43	5.0	2.10
	3.24	4.8	1.10
	4.05	4.4	0.80
	4.86	4.0	0.60
HCOO ⁻	0.81	6.0	7.40
	1.62	5.1	3.20
	2.43	4.4	1.80
	3.24	3.7	1.20
	4.05	3.0	0.70
	4.86	2.6	0.20
Ca^{2+}	0.81	1.8	2.00
	1.62	2.4	1.50
	2.43	3.2	1.30
	3.24	3.8	1.10
	4.05	4.4	1.00
	4.86	4.8	0.90
Mg^{2+}	0.81	1.0	1.20
	1.62	1.3	0.80
	2.43	1.5	0.62
	3.24	1.8	0.60
	4.05	2.2	0.54
	4.86	2.4	0.49

Michaelis-Menten Plot

Least square analysis of the plots of $1/k_{obs}$ versus 1/[SSH] (Figure 6) gave a straight line with an insignificant negative intercept which is in favor of outer-sphere reaction pathway [1,3]. This also suggests the absence of intermediate complex

formation [4,23,24]. Similar reaction pathway was reported for the $Fe(C_2O_4)_3^{3-7}/S_2O_3^{2-7}$ and $Fe(C_2O_4)_3^{3-7}/SO_4^{2-7}$ systems [1,4]. However, inner-sphere reaction pathway has been reported for the redox reaction of $Fe(C_2O_4)_3^{3-7}/MnO_4^{-7}$ [3].

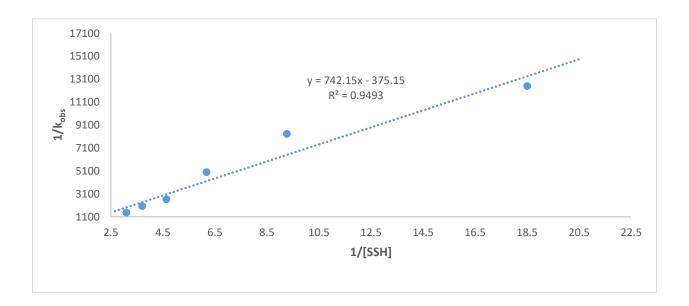


Figure 6: Plot of 1/k_{obs} versus 1/[SSH] for the reduction of potassium trisoxalatoferrate (III) by sulfosalicylic acid at [Fe(C₂O₄)₃³⁻] = 5.4 × 10⁻³ mol dm⁻³, I = 0.3 mol dm⁻³ (NaCl), [H⁺] = 0.2 mol dm⁻³ λ_{max} = 420 nm and T = 29 ± 0.1 °C.

Test for Free Radicals

The involvement of free radical in the redox process was studied by the addition of acrylamide to partially reacted mixture of oxidant and reductant. There was precipitate when excess methanol was added to the reacted mixture. This is an evidence of the possible occurrence of inner-sphere electron transfer mechanism [12]. This suggests that free radicals are important in this reaction [18,24,27]. A similar result for the polymerization of acrylamide by potassium trisoxalatoferrate (III) has been reported other researchers [3,4]. Free radical was not detected elsewhere [10].

Mechanism of the Reaction

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

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$[Fe(C_2O_4)_3]^{3-} + H^+ \qquad \stackrel{k_1}{\xleftarrow}_{k_{-1}} [HFe(C_2O_4)_3]^{2-} (fast)$
$HFe(C_2O_4)_3^{2-} + SSH \xrightarrow{k_2} [HFe(C_2O_4)_3]^{2-}, SSH]$ (slow)
$[HFe(C_2O_4)_3]^{2-}, SSH] \xrightarrow{k_3} Fe(C_2O_4)_3]^{4-} + H + 2S^+ $ (slow)(7)
$\operatorname{Fe}(\operatorname{C}_{2}\operatorname{O}_{4})_{3}^{3-} + \operatorname{SH} \stackrel{k_{4}}{\rightleftharpoons} [\operatorname{Fe}(\operatorname{C}_{2}\operatorname{O}_{4})_{3}]^{4-} + \operatorname{H} + \operatorname{S}^{+} \dots \dots \dots (8)$
From equations (6) and (7),
Rate = k_2 [HFe(C ₂ O ₄) ₃ ²⁻][SSH] + k_3 [Fe(C ₂ O ₄) ₃ ³⁻][SSH](9)
From equation (5):
$[HFe(C_2O_4)_3^{2-}] = k_1[Fe(C_2O_4)_3^{3-}][H^+]$ (10)
Substituting equation (10) into (9),
Rate = $k_1k_2[Fe(C_2O_4)_3^{3-}][H^+][SSH] + k_3[Fe(C_2O_4)_3^{3-}][SSH]$ (11)
Rate = $(k_3 + k_1k_2 [H^+])[Fe(C_2O_4)_3^{3-}][SSH](12)$
Equation (12) agrees with equation (4) where

$$\label{eq:k3} \begin{split} k_3 &= a = 0.9 \times 10^{\text{-4}} \ mol^{\text{-1}} \ dm^3 \ s^{\text{-1}} \ and \ k_1 k_2 &= b = 0.4 \\ &\times 10^{\text{-3}} \ mol^2 \ dm^6 \ s^{\text{-1}}. \end{split}$$

Conclusion

A major point of interest as to whether or not the reaction proceeded via outer sphere, inner sphere mechanism or a combination of both was resolved as follows:

 (a) Negative ions catalysis is suggestive that the reaction likely proceeded via an outersphere electron transfer mechanism [24]. Ion catalysis and inhibition has been reported to be a characteristics of an outer sphere reaction mechanism [12,21,27].

- (b) Positive result for free radical test is pointing to an inner sphere electron transfer mechanism [12].
- (c) Insignificant negative intercept for Michaelis-Menten plots of 1/k_{obs} versus 1/[SSH] from (Figure 6) is in support of an outer-sphere reaction pathway [1].
- (d) Negative ionic strength dependence and positive slope obtained from plots of log k₂ versus √I (Table 1) correspond reasonably with an outer-sphere mechanism [11,22].

Based on the results obtained, the evidences (a -d) are more in favor of an outer-sphere reaction pathway and the outer sphere mechanism is hereby proposed for the redox reaction of Fe³⁺/SSH system.

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