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# Kinetics and Mechanism of the Oxidation of Orange II by Heptaoxodichromate(VI) Ion in Aqueous Acid

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## Abstract

The kinetics of the oxidation of orange II (here and thereafter referred to as $OR^-$) by heptaoxodichromate(VI) ion has been studied spectrophotometrically at constant ionic strength, I = 0.50 $C^2$mol $dm^{-3}$ (NaCl), $[H^+] = 5.0 \times 10^{-2}$mol $dm^{-3}$ (HCl) and T = 24± 1°C. The redox reaction displayed a stoichiometry of 2:1 andrate equation for the reaction is:$-d[OR^-]/dt = (a + b[H^+])[OR^-][Cr_2O_7^{2-}]$. The rate of the reaction increases with increase in acid concentration. Varying the ionic strength of the medium from 0.2 – 0.9 $C^2$mol $dm^{-3}$ (NaCl) showed that the rate constant increases with increase in ionic strength, suggesting a positive BrØnsted - Debye salt effect. The reaction shows a first order dependence on [oxidant] and [reductant]. Added ions ($Ca^{2+}$, $Mg^{2+}$, $CH_3COO^-$ and $SO_4^{2-}$) on the reaction rate increased the rates of the reactions. Michaelis–Menten's plot of $1/k_1$ versus $1/Cr_2O_7^{2-}$and spectrophotometric test suggest absence of an intermediate in the rate determining step. Polymerisation was not induced by the addition of acrylonitrile to reaction mixture followedby excess methanol, as evidenced by the formation of a gelatinous precipitate. The results obtained suggest the absence of the formation of intermediate complex thereby supporting the outersphere mechanism.

**Keywords:**Polymerisation; outersphere; kinetics; intermediate; stoichiometry; product

## Introduction

Azo compounds have a lot of applications in photodynamic therapy as well as photosensitive species in photographic or electrophotographic systems and are dominant organic photoconductive materials. The dyes are important structures in the medicinal and pharmaceutical fields [1]. Due to the complex structure of azo dyes, they are highly persistent in natural environments, which may lead to acute toxicity of ecosystem. Textile factories discharge large quantity of effluents into water streams and soils. The treatment of textile wastewater is of great concern because of their toxicity being created. The typical techniques used to treat dye-containing wastewater are physical, biological and chemicalprocesses [2].

The commonly applied physical or physicochemical methods such as adsorption, sedimentation, flotation, and flocculation effectively remove certain dyes from water. However, these techniques are non-destructive and merely transfer the dye from one form of waste to another, thus generating secondary pollutants requiring further treatment. In biological treatment methods degradation of dyes occurs by means of living microorganisms. Usually, these techniques comprise combined anaerobic and aerobic treatment to ensure efficient dye removal. Biological processes are generally cheap and capable of removing dyes from large volumes of wastewater. However, most textile wastewaters cannot be readily degraded by biological treatment because many dyes are no biodegradable and toxic to the organisms used in the processes [3]

Chemical oxidation methodsare traditionally accepted as the most promising alternative for the degradation of persistent pollutants with limited biodegradability and tendency to accumulate in the environment. Chemical oxidation is the only degradation method that has the potential to completely transform harmful organic contaminants in water into biodegradable substrates, harmless end products [4].

Heptaoxodichromate(VI) ion, an excellent oxidising agent has been observed to oxidised naphthol green B [5] and indigo carmine [6].Kinetics of pyrite oxidation in potassium dichromate solution was investigated. The effect of stirring rate, temperature and particle size, have been examined [7]. The rate of pyrite oxidation was found to be independent of the stirring speed. The degree of dichromate ion reduction in the final solutions showed that at all temperatures sulphide was oxidized to sulphate according to the following reaction as shown in equation 1

$$2FeS_2 + 5Cr_2O_7^{2-} + 38H^+ \longrightarrow 2Fe^{3+} + 4SO_4^{2-} + 10Cr^{3+} + 19H_2O \qquad 1$$

A linear relationship between the rate constant k, and the inverse of the initial particle diameter was established. Also, the value of the apparent activation energy of 43.5 ± 1.5kJ $mol^{-1}$, supports that the rate of dissolution is limited by the chemical reaction on the surface of particles. Despite the redox reactions of heptaoxodichromate(VI) ion with several compounds, its reaction with $OR^-$ has not been given much attention. The desire to further study on the reaction of $OR^-$ with heptaoxodichromate(VI) ion prompted this research. The aim of this research is to carry out kinetic study and propose mechanism of the redox reactions of orange II with heptaoxodichromate(VI) ion in aqueous acidic solution. This will objectively lead to the determination of the stoichiometry, order of the reactions, effect of changes in acid concentration, changes in ionic strength on reaction rates and effect of added cations and anions on the reaction rates. Also, the formation of intermediate complex and free radical in the course of the reactions and products analysis.

## Materials and Methods

### *Materials and Reagents*

All chemicals and reagents used in this work were analar grade and were used without further purification. $K_2Cr_2O_7$ was used as the oxidant, HCl was used to furnish $H^+$ to the reaction and NaCl was used to maintain a constant ionic strength. Acetone was used to vary the dielectric constant of the reaction medium. $OR^-$, the oxidant and the other solutions were prepared with distilled water. The $\lambda_{max}$ (484nm) used for this work was determined by scanning the dye solution using UV-Vis spectrophotometer 6405 in the wavelength range 400 – 600nm.

### *Kinetics measurements*

The stoichiometry of the reaction was determined spectrophotometrically using the mole ratio method by keeping the concentration of the dye constant at $5.0\times10^{-5}$moldm$^{-3}$,$[H^+] = 5.0 \times 10^{-2}$mol dm$^{-3}$, I = 0.50 $C^2$mol dm$^{-3}$, $\lambda_{max}$ = 484 nm, T=24±1°C, and $[Cr_2O_7^{2-}]$ = $4.8\times10^{-3}$-$9.0\times 10^{-3}$M. The stoichiometry was determined from the plot of absorbance versus [reductant]/[oxidant] after the reaction had reached completion by the observation of a steady zero absorbance value over a period of two days. A UV-Vis spectrophotometer 6405was used to follow the decrease in absorbance of the dye at 484nm, =24±1°C, I = 0.5 $C^2$moldm$^{-3}$ (NaCl), and $[H^+]$= $5.0\times10^{-2}$moldm$^{-3}$ (HCl). The kinetic runs were conducted under pseudo-first-order conditions with$[Cr_2O_7^{2-}]$ in atleast 90-foldexcess over$[OR^-]$.The pseudo-first-order plotsoflog ($A_t$ - $A_\infty$) versus time were made,(where $A_\infty$ and $A_t$ are the absorbance at the end of the reaction and at time, t respectively).From the slope of the plots, the pseudo-first order rate constant ($k_1$) was determined [8].

### *Effect of hydrogen ion concentration and ionic strength on the reaction rate*

The effect of hydrogen ion on the rate of reaction was investigated by varying $[H^+]$ in the range (2.0 – 15.0) × $10^{-2}$mol dm$^{-3}$while $[OR^-]$and$[Cr_2O_7^{2-}]$ were kept constant at 24 ± 1°C and I = 0.5 $C^2M$ (NaCl).This range was chosen because acid was stable at such range. The effect of ionic strength on the rate of the reaction was studied in the range of 0.2– 0.9 $C^2$mol dm$^{-3}$ (NaCl) while the concentrations of other reagents were kept constant at 24±1°C.

### *Effect of added ions on the reaction rate*

The effect of added ions on the reaction rate was observed by addition of various amount of ions ($Ca^{2+}$, $Mg^{2+}$, $SO_4^{2-}$ and $CH_3COO^-$), while the concentrations of $OR^-$, the oxidant, hydrogen ion, temperature and ionic strength of reaction medium were kept constant [9].

### *Test for free radicals*

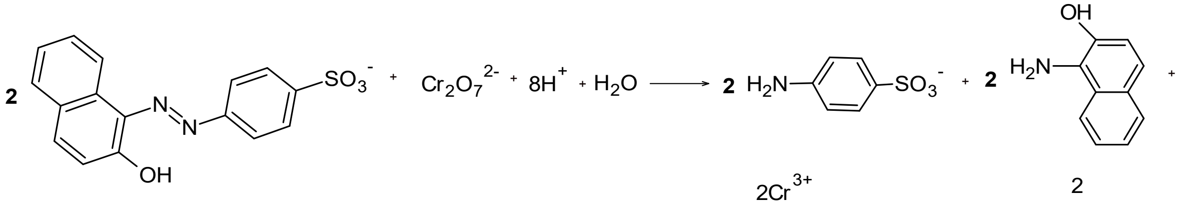
About 5cm$^3$ of acrylonitrile was added to a partially oxidised reaction mixture containing various concentrations of oxidant, reductant and hydrogen ion. This was followed by addition of an excess of methanol. The same treatment was applied to the solution of oxidant and reductant separately as a control.

### *Products analysis*

At the completion of the reaction, the reaction mixture was analysed for possible formation of organic and inorganic products in the reaction. The products were confirmed by chemical test as follows: To test for the presence of sulphanilic acid as one of the expected products; conc. HCl (4 cm$^3$) was added to the reaction mixture in a 50 cm$^3$ beaker and cooled in an ice-bath to 4 $^0$C. Few drops of sodium nitrite solution were added to the mixture while keeping the contents on beaker in the ice-bath. A diazonium salt was formed. A solution of 2-naphthol prepared in 0.1 mol dm$^{-3}$NaOH was added to the diazonium salt and stirred for 30 minutes. To test for the presence of 2-naphthol, a few drops of $FeCl_3$ solution was added to the reaction mixture and a faint green colour was observed [10].Inorganic product was confirmed by qualitative inorganic analysis [11].

## Results and Discussion

The stoichiometric study showed that two moles of $OR^-$ was oxidised by one mole of $Cr_2O_7^{2-}$. This stoichiometry is represented by Equation 1.



A stoichiometery of 1:1 was however, reported for the oxidation of indigo carmine by dichromate [6] and oxidation of malachite green by dichromate [12]. The inorganic product of the reaction involving $OR^-$ and $Cr_2O_7^{2-}$ was qualitatively confirmed to be $Cr^{3+}$similar result has been reported on redox reaction of indigo carmine with heptaoxodichromate(VI) ion in acidic medium[6]. Sulphanilic acid and 2-naphthol were confirmed as the organic products as determined by chemical test [11].

The pseudo - first order plots of log($A_t$ - $A_\infty$) versus time for this reaction was linear; the linearity of these plots indicates that these reactions are first order with respect to

[OR$^-$]. A plot of logk$_1$ versus log [$Cr_2O_7^{2-}$] (Fig. 1) was linear with a slope of 1.05 showing that the reaction is also first order with respect to [$Cr_2O_7^{2-}$]. Similar results have been reported for the oxidation of indigo carmine by heptaoxodichromate(VI) ion in aqueous acidic medium [6] and oxidation of propane-1,2,3-triol by Cr(VI) in aqueous acidic medium [13]. This is also supported by the constancy of k$_2$values shown in Table 1.

The result of the acid dependence shows that the rate constant increases with increase in [H$^+$]. Plot of logk$_1$ versus log[H$^+$] (Figure 2) gave a slope of about one indicating that the reaction is first order with respect to [H$^+$]. Plot of k$_2$ versus [H$^+$]was linear with an intercept in the acid concentration range investigated ($R^2$ = 0.99). This indicates that a pre-equilibrium between protonated and deprotonated forms of a reactant is rapid, that the protonation equilibrium constant is small, that both the forms are reactive, that the protonated form is more reactive [14]. The rate equation for the reaction is shown in equation 2.

$$-\frac{d[OR^-]}{dt} = (a + b[H^+])[OR^-][Cr_2O_7^{2-}] \qquad 2$$

Varying the ionic strength of the medium from 0.2 – 0.9 C$^2$moldm$^{-3}$ (NaCl) showed that the rate constant increases with increase in ionic strength (Table 1), suggesting a positive BrØnsted - Debye salt effect [15-16]. Plot of logk$_2$ against I$^{1/2}$(Figure 3) gave a slope of 0.63 ($R^2$ = 0.97). This implies that activated complex was formed from reactants with similar charges in the rate determining step and the product of their charges was 0.63. This is an indication that the non-integral value for the products of the species at the transition state was as a result of some other interactions that might have taking place [13].

**Table 1: Pseudo – first order and second order rate constants for the reaction of OR$^-$and $Cr_2O_7^{2-}$ at [OR$^-$] = 5.0 × 10$^{-5}$ mol dm$^{-3}$, λ = 484 nm and T = 24 ± 1°C**

| $10^3$[$Cr_2O_7^{2-}$], mol dm$^{-3}$ | $10^2$[H$^+$], mol dm$^{-3}$ | I, C$^2$mol dm$^{-3}$ | $10^3$k$_1$, s$^{-1}$ | k$_2$, dm$^3$ mol$^{-1}$ s$^{-1}$ |
|---|---|---|---|---|
| 4.8 | 5.0 | 0.5 | 1.84 | 0.384 |
| 6.0 | 5.0 | 0.5 | 2.30 | 0.383 |
| 7.0 | 5.0 | 0.5 | 2.71 | 0.387 |
| 7.5 | 5.0 | 0.5 | 2.88 | 0.384 |
| 8.0 | 5.0 | 0.5 | 3.07 | 0.384 |
| 9.0 | 5.0 | 0.5 | 3.41 | 0.379 |
| 7.5 | 2.0 | 0.5 | 1.49 | 0.199 |
| 7.5 | 3.0 | 0.5 | 1.85 | 0.247 |
| 7.5 | 5.0 | 0.5 | 2.87 | 0.383 |
| 7.5 | 7.0 | 0.5 | 3.16 | 0.421 |
| 7.5 | 8.0 | 0.5 | 3.58 | 0.477 |
| 7.5 | 10.0 | 0.5 | 4.07 | 0.543 |
| 7.5 | 15.0 | 0.5 | 5.74 | 0.765 |
| 7.5 | 5.0 | 0.2 | 1.76 | 0.235 |
| 7.5 | 5.0 | 0.3 | 2.11 | 0.281 |
| 7.5 | 5.0 | 0.4 | 2.55 | 0.340 |
| 7.5 | 5.0 | 0.5 | 2.88 | 0.384 |
| 7.5 | 5.0 | 0.7 | 3.14 | 0.418 |
| 7.5 | 5.0 | 0.8 | 3.44 | 0.459 |
| 7.5 | 5.0 | 0.9 | 3.77 | 0.503 |

Added ions ($Ca^{2+}$, $Mg^{2+}$, $CH_3COO^-$ and $SO_4^{2-}$) on the reaction rate increased the rates of the reactions (Tables 2 and 3). The effects exerted by these ions suggest the occurrence of theoutersphere mechanism. Polymerisation was not induced by the addition of acrylonitrile to reaction mixture followedby excess methanol, as evidenced by the formation of a gelatinous precipitate. Thissuggests that free radicals were not formed in the course of the reaction. The result from the spectroscopic study indicated no significant shift from the absorption maximum of 484nm characteristic of OR$^-$. This suggests that the formation of an intermediate complex might be unlikely. Plot of 1/k$_1$ versus 1/ [$Cr_2O_7^{2-}$] (Figure 4) gave a straight line that passed through the origin. This further suggests the absence of the formation of intermediate complex thereby supporting the outersphere mechanism.

**Table 2: Rate data for the effect of added cations ($Ca^{2+}$ and $Mg^{2+}$) on the rate of reaction of $OR^-$ and $Cr_2O_7^{2-}$ at $[OR^-] = 5 \times 10^{-5}$moldm$^{-3}$, $[Cr_2O_7^{2-}] = 7.5 \times 10^{-3}$mol dm$^{-3}$, $[H^+] = 5.0 \times 10^{-2}$ moldm$^{-3}$, I = 0.50 C$^2$mol dm$^{-3}$, λ = 484 nm and T = 24 ± 1 °C**

| Ion | $10^3$ [ion], mol dm$^{-3}$ | $10^3$ $k_1$, s$^{-1}$ | $k_2$, dm$^3$ mol$^{-1}$ s$^{-1}$ |
|---|---|---|---|
| $Ca^{2+}$ | 0.0 | 2.89 | 0.385 |
| | 10.0 | 3.27 | 0.436 |
| | 20.0 | 3.92 | 0.523 |
| | 40.0 | 4.41 | 0.588 |
| | 60.0 | 4.82 | 0.643 |
| | 80.0 | 5.31 | 0.708 |
| | 100.0 | 6.12 | 0.816 |
| | 150.0 | 6.97 | 0.929 |
| $Mg^{2+}$ | 0.0 | 2.87 | 0.384 |
| | 10.0 | 3.55 | 0.473 |
| | 20.0 | 3.98 | 0.531 |
| | 40.0 | 4.62 | 0.616 |
| | 60.0 | 5.27 | 0.703 |
| | 80.0 | 5.88 | 0.784 |
| | 100.0 | 6.74 | 0.899 |
| | 150.0 | 7.29 | 0.972 |

**Table 3: Rate data for the effect of added anions ($CH_3COO^-$ and $SO_4^{2-}$) on the rate of reaction of $OR^-$ and $Cr_2O_7^{2-}$at $[OR^-] = 5.0 \times 10^{-5}$moldm$^{-3}$, $[Cr_2O_7^{2-}] = 7.5 \times 10^{-3}$moldm$^{-3}$, $[H^+] = 5.0 \times 10^{-2}$ moldm$^{-3}$, I = 0.50 C$^2$mol dm$^{-3}$, λ = 484 nm and T = 24 ± 1 °C**

| Ion | $10^3$ [ion], mol dm$^{-3}$ | $10^3$ $k_1$, s$^{-1}$ | $k_2$, dm$^3$ mol$^{-1}$ s$^{-1}$ |
|---|---|---|---|
| $CH_3COO^-$ | 0.0 | 2.88 | 0.384 |
| | 10.0 | 3.01 | 0.401 |
| | 20.0 | 3.63 | 0.484 |
| | 40.0 | 3.81 | 0.508 |
| | 60.0 | 4.74 | 0.632 |
| | 80.0 | 5.35 | 0.713 |
| | 100.0 | 5.35 | 0.767 |
| | 150.0 | 6.71 | 0.895 |
| $SO_4^{2-}$ | 0.0 | 2.87 | 0.383 |
| | 10.0 | 3.14 | 0.419 |
| | 20.0 | 3.69 | 0.492 |
| | 40.0 | 3.83 | 0.511 |
| | 60.0 | 4.49 | 0.599 |
| | 80.0 | 4.82 | 0.643 |
| | 100.0 | 5.26 | 0.701 |
| | 150.0 | 5.97 | 0.796 |

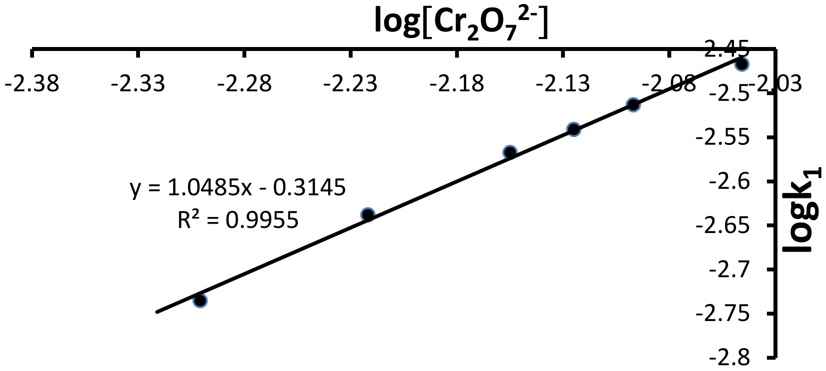


**Fig 1: Plot of log k$_1$ Versus log[$Cr_2O_7^{2-}$] for the Redox Reaction of $OR^-$ with $Cr_2O_7^{2-}$ at [$OR^-$] = 5.0 × $10^{-5}$moldm$^{-3}$, [$Cr_2O_7^{2-}$] = (4.8 – 9.0) × $10^{-3}$moldm$^{-3}$, [$H^+$] = 5.0 × $10^{-2}$ moldm$^{-3}$, I = 0.50 $C^2$ moldm$^{-3}$, λ = 484 nm and T = 24 ± 1°C**

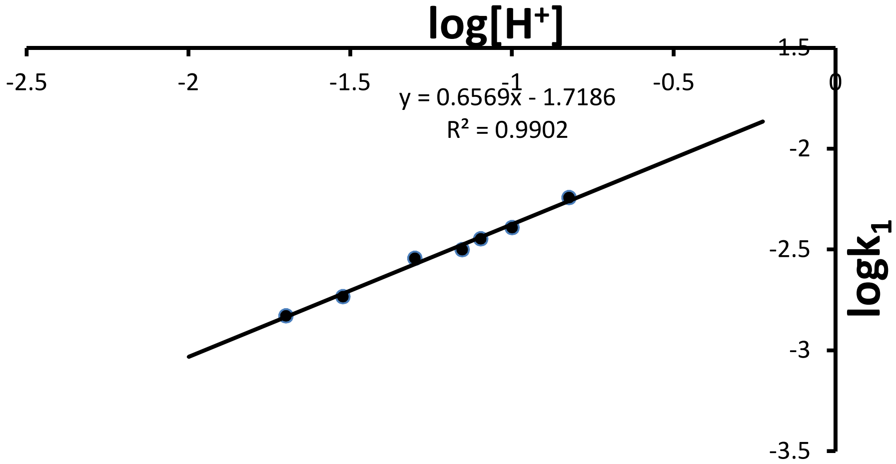


**Fig 2: Plot of log k$_1$ Versus log [$H^+$] for the Redox Reaction Between $OR^-$ and $Cr_2O_7^{2-}$ at [$OR^-$] = 5.0 × $10^{-5}$moldm$^{-3}$, [$Cr_2O_7^{2-}$ ] = 7.5 × $10^{-3}$moldm$^{-3}$, [$H^+$] = (2.0 – 15.0) × $10^{-2}$moldm$^{-3}$, I = 0.50 $C^2$mol dm$^{-3}$, λ = 484 nm and T = 24 ± 1 °C**

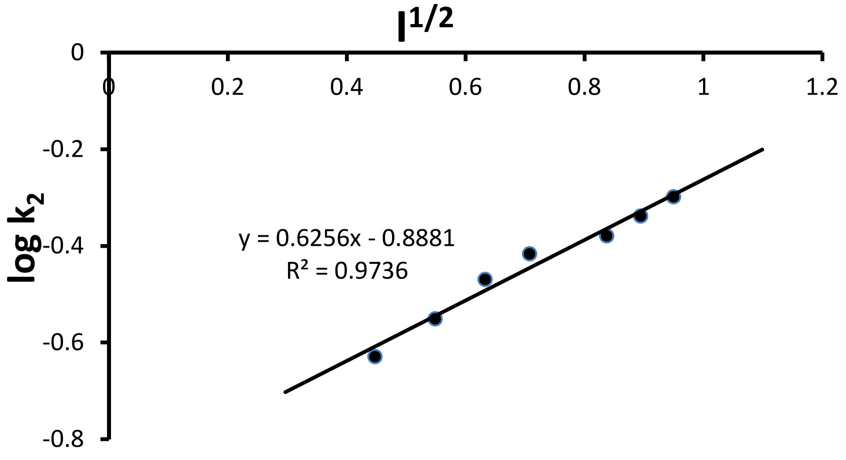


Fig 3: Plot of log$k_2$ Versus $I^{1/2}$ for the Redox Reaction Between $OR^-$ and $Cr_2O_7^{2-}$at $[OR^-]$ = 5.0 × $10^{-5}$moldm$^{-3}$, $[Cr_2O_7^{2-}]$ = 7.5 × $10^{-3}$ moldm$^{-3}$, $[H^+]$ =5.0 × $10^{-2}$ moldm$^{-3}$, I = 0.50 $C^2$ moldm$^{-3}$, λ = 484 nm and T= 24 ± 1°C

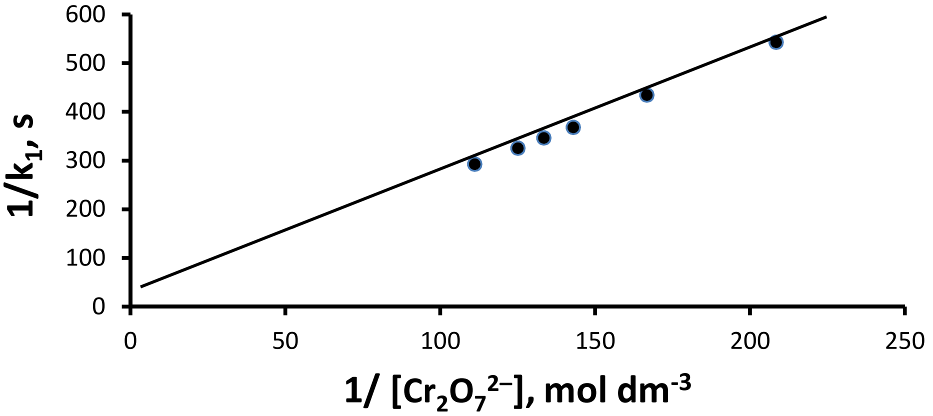
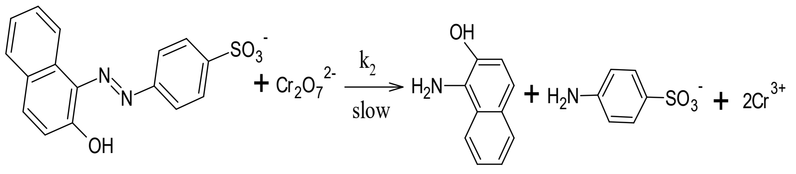
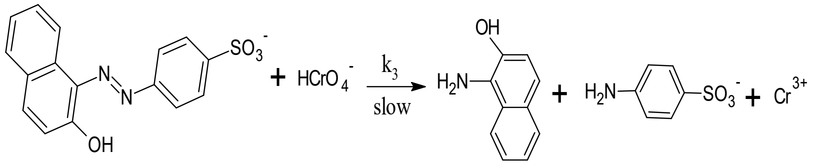


Fig 4: Michaelis - Menten plot for the Redox Reaction Between $OR^-$ and $Cr_2O_7^{2-}$at $[OR^-]$ = 5.0 × $10^{-5}$moldm$^{-3}$, $[Cr_2O_7^{2-}]$ = (4.8 – 9.0) × $10^{-3}$moldm$^{-3}$ $[H^+]$ = 5.0 × $10^{-2}$ moldm$^{-3}$, I = 0.50 $C^2$mol dm$^{-3}$, λ = 484 nm and T= 24 ± 1 °C

On the basis of the above findings, the following mechanisms are proposed for this reaction (equations 3 – 6).

$$CrO_4^{2-} + H^+ \underset{}{\overset{K_1}{\rightleftharpoons}} HCrO_4^- \qquad (3)$$


(4)


(5)

$$Rate = k_2[OR^-][Cr_2O_7^{2-}] + k_3K_1[OR^-][CrO_4^{2-}][H^+] \qquad [6]$$

## Conclusion

The redox reaction of orange II and heptaoxodichromate (VI) ionin aqueous acid medium showed a stoichiometry of 2:1; a first order was observed for $OR^-$ and $Cr_2O_7^{2-}$ions. The rate constant increases with increase in hydrogen ion and ionic strength of the reaction medium. Spectroscopic evidence and the Michaelis-Menten'splot of $1/k_1$ versus 1/ $Cr_2O_7^{2-}$suggests that intermediates maybe unimportant in the rate determining step. Polymerizationtest suggests the absence of free radicals in the reactionmedium. Based on the above results, it is proposed that thereaction most probably operates through the outerspheremechanism.

### Competing Interests

Authors have declared that no competing interests exist

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