



Spectrophotometric Study of Redox Reaction between Synthesized Amino Heterocyclic Azo Dye and Iodate Ion in Aqueous Acidic Media

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Abstract: Spectrophotometric study between synthesized amino heterocyclic azo dye (hereafter referred to as DYE) and iodate ion (IO_3^-) in acid medium has been carried out in aqueous solution with a 1:1 stoichiometry as the reaction goes to total completion. Pseudo-first order reaction condition was employed with first-order in both [DYE] and [IO_3^-] obtained. Enhancement of the reaction rate was obtained as the ionic strength of the reaction medium increases within the concentration studied. The rate of the reaction showed dependence on acid (in the range studied). Spectroscopic investigation and even kinetic study suggest no complex formation. No radicals are implicated in this reaction. The system is proposed to be an outersphere mechanism.

Keywords: Spectrophotometric, Kinetics; Redox Reaction; Outer-sphere; Dye

1. Introduction

Chemical kinetics is an aspect of chemistry that involved the rates and their impact by physical parameters such as light, temperature, pressure, and concentration of solvent on the rate of reaction. Calculation of the reaction rates in different modes provides useful guide in the elucidation of the reaction mechanism (El Seoud *et al.*, 2017; Taylor *et al.*, 2023). Chemical kinetics attempts to give adequate answer to all that transpires in the reactants and the products of the reaction. Does it involve many steps or through complexes? This subject is of immense significance in biochemical processes especially in dealing with organic synthesis, in industry and industry (Chigwada, 2005). In chemical kinetic scientist propose in line with natural occurrences, understanding how fast the reactions follows from reactants to products. Sustainability in manufacturing economic realities depend on the rate of occurrence of the reactions. Besides, catalytic enzymes have been found controlling many complex systems in living organisms at a carefully controlled rate. (Muhammad, 2003). The operations in living

systems and chemical industries are basically redox process that involved transfer of electron between any two chemical entities.

Kinetics and mechanisms of redox reaction of dyes have received considerable attentions over the past decades (Ibrahim *et al.*, 2023; Ouhammou *et al.*, 2019; N'daye *et al.*, 2022). Compounds having azo groups (-N=N-) joined with one or polycyclic ring systems are called Azo dyes. They are manufacture during diazotization of amine, linked with one or more electron-rich nucleophiles of amino group (Gürses *et al.*, 2016). The report of other researchers such as (Zhao *et al.*, 2011; Leriche *et al.*, 2010) showed other methods of synthesizing azo dye such as redox reaction of nitroso molecules by AlLiH_4 , reduction of nitroaromatic derivatives in alkaline medium, reaction quinones and hydrazines, primary amines in the presence of permanganate or lead tetraacetate etc. This group of compounds have their bond joined to ring systems, naphthalenes, heterocycles, to ionizable aliphatic groups (Benkhaya, *et al.*, 2020). Examples includes methyl orange, methyl yellow, methyl red etc. They are importantly used as coloring component in the textile industries and leather technology, paper manufacturing and printing, etc. (Benkhaya, and El Harfi, 2017; N'daye *et al.*, 2020; Ali *et al.*, 2022; Soko *et al.*, 2023). Their importance is due to their rare complexing behavior, and significance in the photometric with extractive detection of different metal ions and sensitivity as chromogenic reagents. In complexometric titration, it is highly helpful as indicators. Due to their reactivity with d-block ions it produces stable chelates, it is also use in generating beautiful colors similar to other organic reagents which are useful in separation techniques (Eltaboni *et al.*, 2022). Removal of these dyes from environments after application is very importance to scientists (Elsherif, *et al.*, 2022; El Badraoui, *et al.*, 2024).

Iodate is one of several oxyanions of iodine, with oxidation number of +5. In recent, oxyanions have participated in several redox reactions with dyes, such as: oxidation of pyrocatechol violet by chlorite and iodate ion (Adetoro *et al.*, 2010, 2011), kinetic of nitrite ions and toluidine dye (Hamza *et al.*, 2012), oxidation of malachite green by hypochlorite ion (Idris *et al.*, 2015), permanganate ion reduction by indigo carmine (Falodun *et al.*, 2015), mechanism reaction of nitrite ion and bromate ion with neutral red in aqueous (Ibrahim *et al.*, 2016; Ibrahim and Hamza, 2016), sulphite ion and malachite green by Imam *et al.*, 2018, oxidation of iodide ion by dichromate ion (Nyong *et al.*, 2020) and report of Myek *et al.* (2020) on orange II with thiosulphate ion. The importance of this present work is to elucidate and present plausible mechanism in the reaction of a synthesized azo dye with iodate ion and propose the mechanism of the reaction. The structure of the synthesized dye is given in **Figure 1** as:

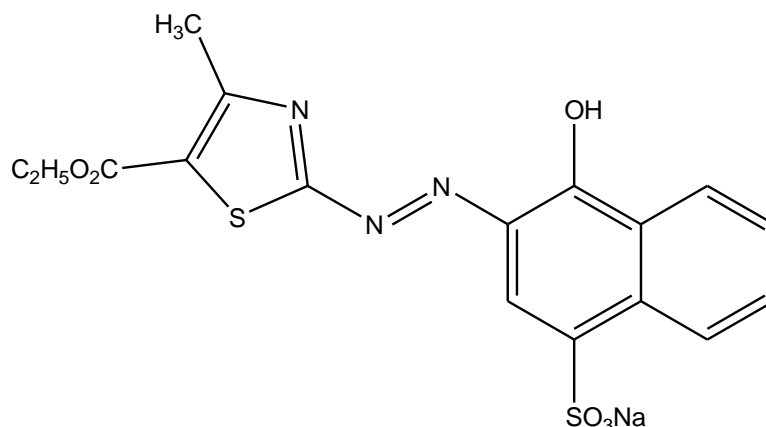


Figure 1: Structure of the synthesized heterocyclic azo dye

2. Methodology

2.1 Materials

All chemical reagents and solvents used were of analytical grade (BDH, Kermel, Sigma-Aldrich) and were used without further purification. Singly distilled water was used throughout for all solutions preparation. Standard aqueous solution of the synthesized amino heterocyclic dye was prepared by dissolving accurately weighed quantity in distilled water. Stock solutions of 2.00 mol dm^{-3} (HCl) was made by diluting 8.5 cm^3 of 36% HCl (specific gravity 1.18) in 50 cm^3 standard flask then made up to mark with distilled water. The solution was standardized titrimetrically with standard solution of previously dried Na_2CO_3 using methyl red as indicator and it was used as a source of H^+ . Stock solutions of NaIO_3 , Na_2SO_4 , NaCl , MgCl_2 , CaCl_2 and NaCO_3 prepared with a known solute dissolving in accurately measured volume of distilled water. Apparatus used were weighing bottles, beaker of various sizes, conical and volumetric flask of various sizes, burette, pipettes of various sizes, glass rod, thermometer, photoelectric colorimeter and analytical balance.

2.2 Stoichiometry

Mole ratio method in line with some researchers (Idris *et al.*, 2007; Ukoha and Ibrahim, 2004; Anweting *et al.*, 2021; Jones *et al.*, 2023a) using photometric titration method was used to determine stoichiometry of the reactions. Concentration of the IO_3^- was varied between $(1 - 12) \times 10^{-4} \text{ mol/dm}^3$ while the DYE was kept constant at $5 \times 10^{-4} \text{ mol/dm}^3$, that of $[\text{H}^+] = 1 \times 10^{-3} \text{ mol/dm}^3$, $\lambda_{\text{max}} = 530 \text{ nm}$ and $U = 0.30 \text{ mol/dm}^3$ (NaCl). After allowing the reaction to go to completion, the stoichiometry was determined as shown by marking the point from the curve of absorbance against mole ratio $[\text{IO}_3^-]/[\text{DYE}]$.

2.3 Kinetic study

Kinetic studied was followed by monitoring how absorbance of the DYE (530nm) using photoelectric spectrometer SM202 digital LED, systonic band and 50 Hz frequency decreases. The measurements made under pseudo-first order conditions of $[\text{IO}_3^-]$ at 40-folds of $[\text{DYE}]$. The pseudo first order rate constant (k_{obs}) in each case was obtained from the slope of the plots of $(\log A_t - A_\infty)$ versus time (Iyun and Shehu, 2004, Anweting *et al.*, 2017; Oladunni *et al.*, 2021; Anweting *et al.*, 2023). The second order rate constant (k_2) shows as $k_{\text{obs}}/[\text{IO}_3^-]$ at $[\text{IO}_3^-] = (4.0 - 11) \times 10^{-2} \text{ mol/dm}^3$, $U = 0.30 \text{ mol/dm}^3$ (NaCl) $[\text{H}^+] = 1 \times 10^{-3} \text{ mol/dm}^3$, $\lambda_{\text{max}} = 530 \text{ nm}$ and $[\text{DYE}] = 1 \times 10^{-3} \text{ mol/dm}^3$.

2.4 Dependence of $[\text{H}^+]$ on the rate of the reaction

The $[\text{H}^+]$ dependence was carried out in the presence of hydrochloric acid between $[\text{H}^+] = (2.0 - 14.0) \times 10^{-4} \text{ mol/dm}^3$ during the $[\text{IO}_3^-]$ and $[\text{DYE}]$ keeping constant $U = 0.30 \text{ mol/dm}^3$ (NaCl) and $\lambda_{\text{max}} = 530 \text{ nm}$ (Ukoha and Iyun, 2002; Ukoha and Ibrahim, 2004; Anweting *et al.*, 2021; Akartasse *et al.*, 2022).

2.5 Ionic strength dependence

At $[\text{IO}_3^-]$, $[\text{DYE}]$ and $[\text{H}^+]$ constant and concentration of the inert electrolyte varied, between $(1 - 7) \times 10^{-1} \text{ mol/dm}^3$ (NaCl), ionic strength dependence on the rate of reaction was monitored (Ukoha and Iyun, 2001, 2002; Chibowski *et al.*, 2009 Adetoro *et al.*, 2011; Myek *et al.*, 2020). The $[\text{IO}_3^-]$, $[\text{DYE}]$ and $[\text{H}^+]$ were kept constant at $1 \times 10^{-1} \text{ mol/dm}^3$ and $1 \times 10^{-3} \text{ mol/dm}^3$ and $[\text{H}^+] = 1 \times 10^{-3} \text{ mol/dm}^3$ respectively.

2.6 Anions and cations effects

Ions in this reaction medium was carried out by varying Mg^{2+} , Ca^{2+} , SO_4^{2-} and CO_3^{2-} concentration in the medium while all other conditions remained constants (Ibrahim *et al.*, 2016; Anweting *et al.*, 2017; Imam *et al.*, 2018) at $[\text{IO}_3^-] = 1 \times 10^{-1} \text{ mol/dm}^3$, $U = 0.30 \text{ mol/dm}^3$ (NaCl), $[\text{H}^+] = 1 \times 10^{-3} \text{ mol/dm}^3$ and $[\text{DYE}] = 1 \times 10^{-3} \text{ mol/dm}^3$

2.7 Analysis of Intermediate complex formation

Analyzing formation of intermediate complex carried out using spectroscopic test using spectrum of the reaction obtaining 120 s after the beginning of the reaction with the DYE in a wavelength of 400 – 700 nm. Plot of $1/k_{\text{obs}}$ versus $1/[\text{IO}_3^-]$ for the kinetic test was also used to verified the results obtained according to Anweting *et al.* (2012a & b) and Imam *et al.* (2018).

2.8 Presence of radicals

5cm^3 acrylamide solution mixed to an oxidized mixtures of a known concentrations of iodate ion, DYE and hydrogen ion. After, excess methanol was added (Osunlaja, *et al.*, 2012; Osunlaja, 2014). The acrylamide was also added to the separate mixture of iodate ion and DYE to serve as control.

3. Results and Discussion

3.1 Stoichiometry

The result obtained from photometric titration curve (Figure 2), when absorbance was plotted against mole ratio ($[\text{IO}_3^-]/[\text{DYE}]$) shows that one mole of DYE reacted with one mole of IO_3^- and the reaction can be simplified as given by equation 1.

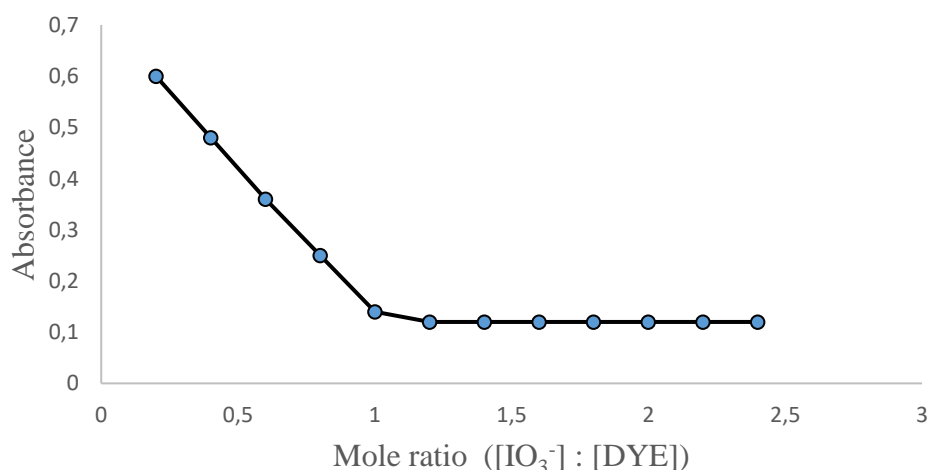


Figure 2: Plot of absorbance versus mole ratio for determination of stoichiometry of redox reaction between DYE and IO_3^- .

Similar results has been found in the report of pyrocatechol violet and iodate ion (Adetoro *et al.*, 2011), the oxidation of malachite green with permanganate ion (Mohammed *et al.*, 2009), redox reaction of neutral red with an azo dye with bromate ion and nitrite ion (Ibrahim *et al.*, 2016; Ibrahim and Hamza, 2016), electron transfer reaction of malachite green and iodate ion (Imam *et al.*, 2018).

However, in toluidine blue and nitrite ions, 3 moles of the dye reacted with 1 mole of nitrite ion (Hamza *et al.*, 2012). Myek *et al.* (2020) reported that one mole of orange II was oxidized by four moles of thiosulphate ion.

3.2 Kinetic Study

Linear plot up to more than 70 % extent of reaction of $\text{Log}(A_t - A_\infty)$ versus time was obtained, meaning that the reaction under the experimental conditions is first order in [DYE]. The pseudo-first order rate constant was determined at different initial concentrations of IO_3^- . Plot of $\text{Log } k_{\text{obs}}$ against $\text{Log} [\text{IO}_3^-]$ at $[\text{H}^+] = 1 \times 10^{-3} \text{ mol/dm}^3$ $U = 0.3 \text{ mol/dm}^3$, yield a gradient of 0.999 (Figure 3). This make $[\text{IO}_3^-]$ first order. The calculation of $k_2 = k_{\text{obs}}/[\text{IO}_3^-]$ value (Table 1) was fairly consistence meaning that the reaction is also in unity order with respect to $[\text{IO}_3^-]$ and that the overall is second order rate of reaction, therefore conforming to the equation 2 below:



Similar order kinetics have been reported between nitrite ions and toluidine blue (Hamza *et al.*, 2012), neutral red and bromate ion (Ibrahim *et al.*, 2016; Ibrahim and Hamza, 2016), similarly orange II and thiosulphate (Myek *et al.*, 2020), and redox reaction of permanganate and theobromine (Jones *et al.*, 2023b).

3.3 Dependence of $[\text{H}^+]$ on the rate of reaction

The results suggest an increase in hydrogen ions as the rate of reaction increases (Figure 4) at $[\text{IO}_3^-] = 1 \times 10^{-1} \text{ mol/dm}^3$, $[\text{DYE}] = 1 \times 10^{-3} \text{ mol/dm}^3$, $[\text{H}^+] = (2 - 14) \times 10^{-4} \text{ mol/dm}^3$, $T = 24 + 0.1^\circ\text{C}$, $U = 0.3 \text{ mol/dm}^3$ (NaCl). The plotting of k_2 against $[\text{H}^+]$ gave a linearity graph with a positive intercept represented as shown in equation 3:

$$k_2 = a + b[\text{H}^+] \quad \text{Eqn. 3}$$

Given that $a = 0.0199 \text{ mol/dm}^3$ and $b = 3.78 \times 10^2 \text{ mol/dm}^3$.

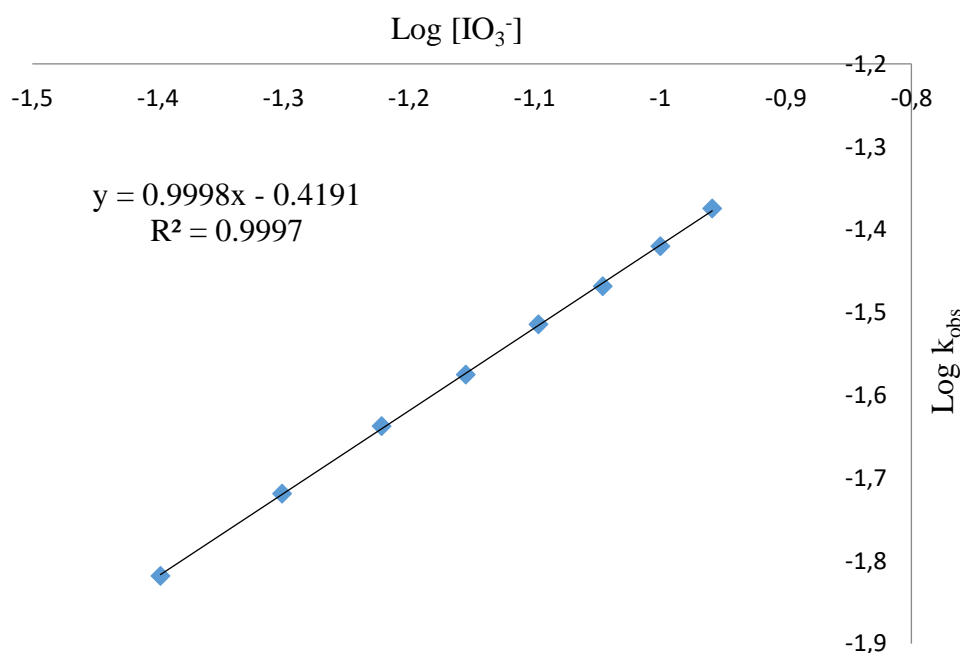


Figure 3: Plot of $\text{Log } k_{\text{obs}}$ versus $\text{Log} [\text{IO}_3^-]$ for the redox reaction of DYE and IO_3^-

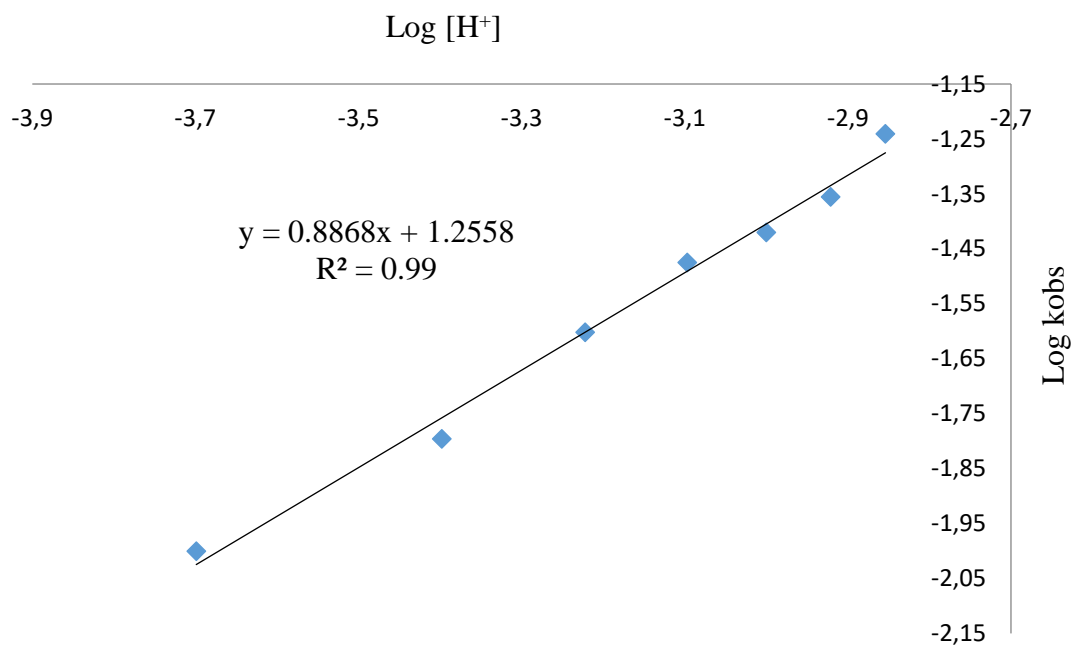


Figure 4: Plot of $\text{Log}k_{\text{obs}}$ versus $\text{Log}[\text{H}^+]$ for the redox reaction of DYE and IO_3^-

Table 1: Pseudo-first order and second order rate constant for the redox reaction of the synthesized Amino heterocyclic dye by IO_3^- in HCl aqueous medium, $\lambda_{\text{max}} = 530\text{nm}$, $T = 27 \pm 1^\circ\text{C}$, $U = 0.30\text{ mol/dm}^3$ (NaCl), $[\text{DYE}] = 1 \times 10^{-3}\text{ mol/dm}^3$

$10 [\text{IO}_3^-]$ (mol/dm^3)	$10^3 [\text{H}^+]$ (mol/dm^3)	$10 [U]$ (mol/dm^3)	$10^3 k_{\text{obs}}$ (s^{-1})	$10k_2$ ($\text{dm}^3/\text{mol/s}$)
0.4	1.0	3.0	15.20	3.80
0.5	1.0	3.0	19.10	3.82
0.6	1.0	3.0	23.05	3.84
0.7	1.0	3.0	26.60	3.81
0.8	1.0	3.0	30.60	3.82
0.9	1.0	3.0	34.00	3.80
1.0	1.0	3.0	38.00	3.80
1.1	1.0	3.0	42.20	3.84
1.0	0.2	3.0	10.00	1.00
1.0	0.4	3.0	16.00	1.60
1.0	0.6	3.0	25.00	2.50
1.0	0.8	3.0	33.50	3.35
1.0	1.0	3.0	38.00	3.80
1.0	1.2	3.0	44.10	4.41
1.0	1.4	3.0	57.50	5.75
1.0	1.0	1.0	32.20	3.22
1.0	1.0	2.0	35.50	3.55
1.0	1.0	3.0	38.00	3.80
1.0	1.0	4.0	40.50	4.05
1.0	1.0	5.0	43.00	4.30
1.0	1.0	6.0	44.40	4.44
1.0	1.0	7.0	48.00	4.80

The rate of the reaction showed two parallel pathways of reaction; one involving the protonated and the other the unprotonated species both reacting to give a product (Jones *et al.*, 2023a & b.). Similar results have been seen in the report of permanganate ion with malachite green and theophylline (Babatunde, 2008; Mohammed *et al.*, 2009; Anweting *et al.*, 2023). Therefore equation 4 can best described the reaction rate in acidic medium:

$$- \frac{d[DYE]}{dt} = (a + b[H^+])[IO_3^-][DYE] \quad \text{Eqn. 4}$$

3.4 Ionic strength dependence

In this experiment, as the concentration of the ionic strength increases by using NaCl, from 0.1 - 0.7 mol/dm³, the rate of the reaction also increases. This indicates a like charges interaction and a phenomenon of Bronsted- Debye salt effect in the activated complex. Results analogous to this have been reported by earlier researchers (Adetoro *et al.* 2010; Babatunde, 2008; Hamza *et al.*, 2012; Myek *et al.*, 2020). Linear plot was obtained for log k₂ versus I^{1/2} (Figure 5).

3.5 Anions and cations effects

Ions Mg²⁺ and Ca²⁺, SO₄²⁻ and CO₃²⁻ retarded the rate of the reaction, a characteristic of reaction that occurred by the outer-sphere mechanistic pathway (Hamza *et al.*, 2012; Myek *et al.*, 2020).

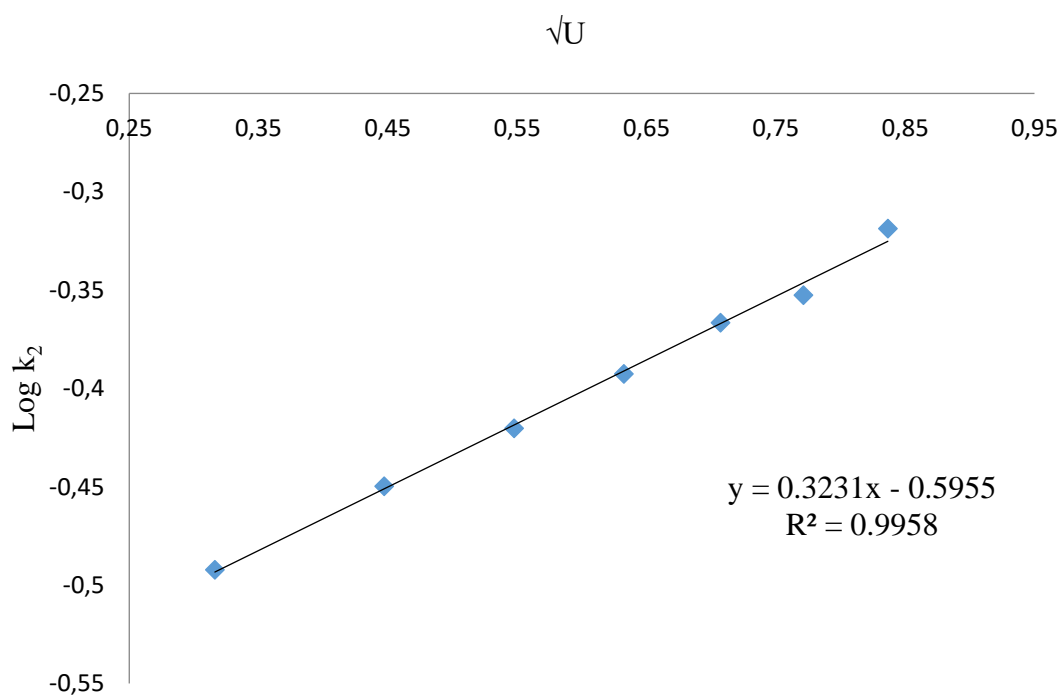


Figure 5: Graph of $\text{Log } k_2$ against \sqrt{I} between redox reaction of DYE and IO_3^-

3.6 Analysis of Intermediate complex formation

Spectrophotometric measurement showed no observable λ_{max} shift from 530nm characteristic of DYE during the reaction with IO_3^- and the electronic spectrum was recorded after 20 minutes of the reaction's mixture. This means intermediate complex formation is absent during the reactions (Babatunde and Ajayi, 2013; Anweting *et al.*, 2017; Jones *et al.*, 2023a, b). Michaelis-Mentens plot of $1/k_{\text{obs}}$ versus $1/[IO_3^-]$ also gave straight line plot with no intercept (Figure 6) further ruled out any

means of intermediate complex prior to the electron transfer (Anweting *et al.*, 2012c, d; Adetoro *et al.*, 2010, 2011, 2021; Oladunni *et al.*, 2021a, b; Anweting *et al.*, 2023).

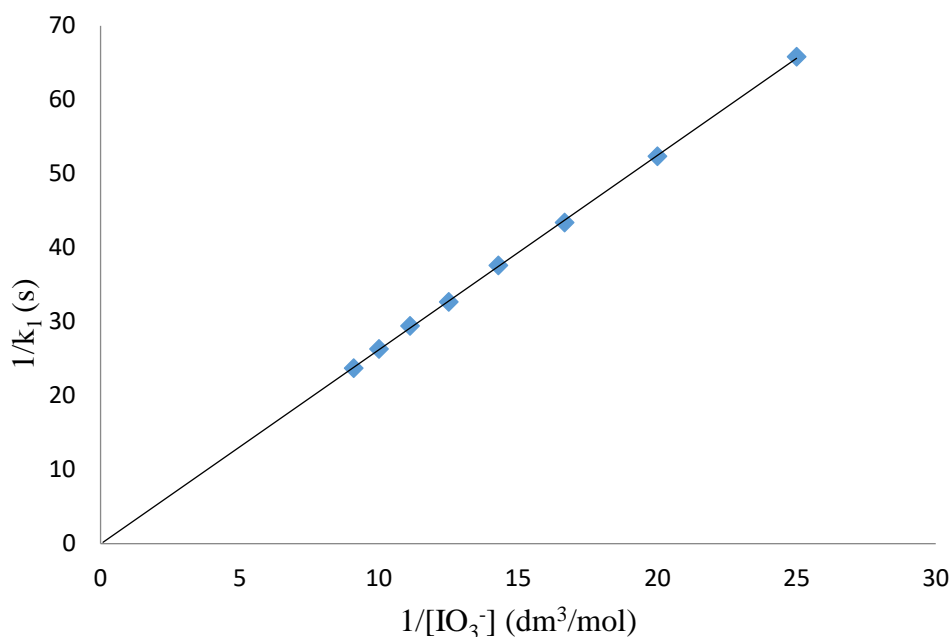


Figure 6: Michaelis-menten graph: Plot of $1/k_1$ (s) versus $1/[\text{IO}_3^-]$ dm³/mol

3.7 Presence of radicals

Absence of gel occurred when 5cm³ of acrylamide followed by excess methanol was introduced to partially oxidized reaction system, solution of DYE and IO₃⁻ separately. The absence of gel formation (polymerization) suggests that no free radical was involved in the reactions (Adetoro *et al.*, 2010, 2011; Anweting *et al.*, 2012a; 2021; Johnson *et al.*, 2023).

3.8 Mechanism of the reaction

The following reaction mechanism is therefore proposed to accommodate all the experimental observations.



$$\text{Rate} = k_1[\text{IO}_3^-][\text{DYEH}^+] + k_2[\text{DYE}][\text{IO}_3^-] \quad \text{Eqn. 8}$$

Consider Eqn 5:

$$[\text{DYEH}^+] = K[\text{H}^+][\text{DYE}] \quad \text{Eqn. 9}$$

Putting Eqn 9 into 8:

$$\text{Rate} = k_1K[\text{IO}_3^-][\text{H}^+][\text{DYE}] + k_2[\text{DYE}][\text{IO}_3^-] \quad \text{Eqn. 10}$$

$$\text{Rate} = k_1K([\text{H}^+] + k_2)[\text{DYE}][\text{IO}_3^-] \quad \text{Eqn. 11}$$

$$k_1K([\text{H}^+] + k_2) \approx k_3$$

Therefore:

$$\text{Rate} = k_3[\text{DYE}][\text{IO}_3^-] \quad \text{Eqn. 12}$$

3.9 Explanation of Mechanism of the reaction

In equation 5, there is a protonation of amino- heterocyclic dye to produce DYEH⁺, which is the protonated form of the dye, similar protonated species have been reported by (Muhammed *et al.*, 2009; Anweting *et al.*, 2012a; 2023; Jones *et al.*, 2023a &b). The rate determining steps (slow steps) of the reactions are equations 6 and 7, and these involve acid dependent and independent pathways respectively, mechanisms that occurred by two pathways were proposed by (Ibrahim *et al.*, 2016, 2011; Anweting *et al.*, 2023; Jones *et al.*, 2023a &b). Equation 11 is similar to equation 4, attesting to the fact that the proposed mechanism is consistent with the kinetic data.

Conclusion

The redox reaction of DYE and IO₃⁻ showed a stoichiometry of 1:1, first – order dependence with respect to concentrations of both DYE and IO₃⁻ and second order rate overall. The rate of the reaction increased with increase in the concentrations of hydrogen ion and ionic strength of the reaction medium. The added ions inhibited the rate of the reaction. There was no evidence for the formation of an intermediate complex. The reaction was rationalized in favour of outer-sphere mechanistic scheme.

Disclosure statement: *Conflict of Interest:* The authors declare that there are no conflicts of interest.

Compliance with Ethical Standards: This article does not contain any studies involving human or animal subjects.

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