Reaction of \( N, N^1 \)-phenylenebis(salicylideneiminato)cobalt(III) and l-cysteine in mixed aqueous medium: kinetics and mechanism

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**ABSTRACT**

The redox kinetics involving the reaction of \( N, N^1 \)-phenylenebis(salicylideneiminato)cobalt(III) ([CoSalophen]\(^+\)) and l-cysteine (LSH) was studied using pseudo-first order approach under the following conditions, \([H^+] = 1.0 \times 10^{-3} \text{ mol/dm}^3\), \( \mu = 0.1 \text{ C}^2 \text{ mol/dm}^3 \) (NaCl), \( \lambda_{\text{max}} = 470 \text{ nm} \) and \( T = 27 \pm 1 \text{ °C} \) in DMSO: H\(_2\)O; 1:4 v: v medium. The redox reaction was 1st order in both [CoSalophen\(^+\)] and [LSH], with the overall 2nd order. Hydrogen ion concentration effect revealed the activeness of both the protonated and deprotonated form of the reactant, positive Bronsted-Debye salt effect and was also ion catalyzed. There was no evidence suggesting an intermediate complex of significant stability in the reaction. Free radical was detected to take part and as such the reasonable mechanistic pathway for the reaction is suggested to be outer-sphere, hence proposed.

1. Introduction

Schiff base complexes of cobalt(III) are utilized as impetuses for the oxygenation reactions of organic molecules (Niederhofer et al., 1984), eminently in the preparative oxygenation of phenols (Matsuura, 1977), indols (Nishinaga et al., 1993) and amines (Nishinaga et al., 1988), a reversible process similar to Co(III)/Co(II) redox potential (Hirotsu et al., 1996). It likewise discovers applications in oxidative coupling catalytic reactions of phenols and oxidation of olefins (Zhang et al., 2003). The catalytic activity of Co(III) salen (b(\( \text{bis(salicylidene)ethylenediamine} \)) complexes demonstrates the catalytically dynamic species to be cobalt in the +3 oxidation state (Eichhorn et al., 1992, 1993; 1997; Eichhorn and Speiser, 1994; Nishinaga et al., 1983; Speiser and Stahl, 1995).

On the other hand, these complexes have been utilized extensively to imitate cobalamin (B\(_{12}\)) coenzymes (Aly, 1998; Amirnasr et al., 2001; Cini et al., 1998; Polson et al., 1997), classified as an oxygen carrier (Niederhofer et al., 1984) and oxygen activators (Bianchini and Zollner, 1996; Henson et al., 1999; Yamada, 1999). Metal complexes of salophen (b(\( \text{bis(salicylidene)phenylenediamine} \))) and salen-type ligands also find applications in the intercalation of DNA base pairs (Kocak et al., 2016; Yilmaz et al., 2017) and for potentiometric discoveries of basic anions existing in biological and environmental systems (Kleij et al., 2006). Salophen-type ligands have additionally been utilized as nitrite sensors (Ganjali et al., 2004). In addition, Co(III) Schiff base complexes have been utilized as antimicrobial agents (Böttcher et al., 1997).

Furthermore, Co(III) salen complexes have shown an outstanding selectivity to poly(propylene carbonate) development with 99% of carbonate linkages. This led to significant efforts been devoted to the synthesis of these complexes as well as mechanistic studies for a better understanding of their reaction mechanism (Cohen and Coates, 2006; Hostélek et al., 2015; Lu and Wang, 2004; Luijstra et al., 2005). Recently, Co(III) salen metal complexes have been used in the electro-reduction of CO\(_2\) to CO, acting as a multi-electron reducing agent (Solomon et al., 2018).

L-cysteine plays structural roles in proteins and can likewise partake in electron transfer reactions and its sulphydryl group, thiolate (RS\(^-\)) is a relatively more potent nucleophilic agent than its protonated form. As such, reactive l-cysteine is in its anionic structure mostly in proteins. In addition, l-cysteine is significant for numerous protein functions; the molecule is toxic to cells in both prokaryotes and eukaryotes even at low concentrations (El-Hag and Dahab, 2016; Özkaz et al., 2003). Thiol-containing compounds are easily susceptible to oxidation and hence a reason for most of their biological functions (Friedman, 1973; Jocelyn, 1972) and numerous kinetic investigations have been stated on the redox reactions with a view to understand the role of enzymes containing l-cysteine group in electron transfer reactions. Oxidation of thiols by metal ions (Amjad et al., 1977; Ayoko et al., 1993; Ayoko and Olatunji, 1983; Gangopadhay et al., 1994; Ghosh et al., 1987; Grases et al., 1986; McAuley and Olatunji, 1977; Olatunji and Okechukwu, 1987;...
Ozawa et al., 1992; Sisley and Jordan, 1995) and non-metallic oxidants (Abedinzadeh et al., 1989; Elskens et al., 1988; Hupe and Wu, 1980; Konidari et al., 1992; Shaked et al., 1986; Weaver and Rabenstein, 1995; Zhao et al., 1994) have been stated to proceed through different mechanisms. An example is where dichromate was used, the overall reaction mechanism involved the formation of a thioester as an intermediate, then intra- and inter-molecular redox reactions (Bose et al., 1992; Braun and Wetterhahn, 1991). However, with Cr(VI) and l-cysteine, redox reaction occurs through an inner-sphere mechanism (Adari et al., 2006), but with Mo(VI) and 12-tungstocobaltate(III), the reaction occurs through an outer-sphere mechanistic pathway (Ayoko and Olutunj, 1983; Martin and Spence, 1970; Sami et al., 2009).

Reports have also been previously published involving oxidation of l-cysteine by Corey’s reagents (Adari et al., 2006), trinuclear Mn(IV) species (Chakraborty et al., 2012) [Mn4O6]4– core (Chakraborty et al., 2013), potassium ferrate (Read et al., 2000) and 11-tungstophosphovanadate(V) (Sami et al., 2009).

[C. Filtration was used to collect the precipitate, then washed and dried with ethanol and desiccator, respectively. Yield: 2.37 g (91%).

Similarlly, this research studies the kinetics of redox reaction of [CoSalophen]⁺, (Salophen = Bis(salicylidene)phenylenediamine) by l-cysteine (LSH) and reported herein for the mechanism of the reaction of this complex to be further understood.

2. Methodology

Chemicals used for this research work were of analytical quality and were used without further purification. The rates of reactions were studied by monitoring the reaction mixture as the absorbance decreases with increasing reaction time at 470 nm on a SHERWOOD colorimeter. The effect changing the concentration of H⁺ was examined by changing the H⁺ concentration within the range of 1.0–18.0 M, while keeping [CoSalophen]⁺ and [LSH] constant at 0.1 M. The second-order acid-dependent variation with [H⁺] was achieved from the plot of k₂ against [H⁺] (Adetoro et al., 2011).

The effect of reaction rate as ionic strength of the medium of reaction was examined for [Z] = 0.3–18.0 M at constant concentrations of the reactants at 27 ± 1 °C. A plot log k₂ against 1/μ was also made.

The effect of added ions were examined for [Z] = 3.0–18.0 M at constant [CoSalophen]⁺, [LSH] and μ. The effect of temperature on the reaction rates was studied within 300–325 K. The data were then considered using Eyring plot of ln k₂/θ.

Conductivity measurements were carried out at room temperature utilizing a HACH Sension5 conductivity meter. Molar conductivity of synthesized complex with the concentration of 1.0 × 10⁻⁴ mol/dm³ at 14 °C. v of DMSO: H₂O was obtained as 122 cm²·Ohm/mol with 12.2 × 10⁻⁶ Ohm/cm as the specific conductance, indicating an electrolytic ratio of 1:1.

Fourier transform infrared (FT-IR) spectra were recorded on shimadzu varian 8400 spectrophotometer in KBr pellets. The FT-IR spectrum of the complex showed the complexation of the ligand with the complex by a slight shift of peak due to the imine (ν(C=N)) bond from 1620 cm⁻¹ to 1612 cm⁻¹ (Salavati-niasari & Shaterian, 2007). Peaks around 547 cm⁻¹ and 462 cm⁻¹ was assigned to ν(C=O) and ν(C=O) respectively (Figures 1 and 3). Magnetic susceptibility (μeff, BM) measurement shown that the Cobalt(III) complex synthesized is of low spin inner orbital hybridized with a magnetic moment μeff of 0.2 BM. This value indicates the oxidation to Co(III) during the preparation of the complex(Abu-Surrah et al., 2008).

Spectrophotometric titration utilized for this study was the mole proportion method of stoichiometric determination. the stoichiometry of the reaction using the method of mole proportion. The concentration of [CoSalophen]⁺ was kept consistent while the proportion of [LSH]: [CoSalophen]⁺ was differed from (0.25–4.00) μ at μ = 0.1 C² mol/dm³ (NaCl) and T = 27 ± 1 °C. A plot of absorbance against mole proportion gave the reaction stoichiometry. A point of inflexion on the graph indicated the mole proportion (Hamza et al., 2012).

Every kinetic measurement was done under condition of pseudo-first order with [LSH] in excess over [CoSalophen]⁺ by at least 20 fold at 0.1 C² mol/dm³ (NaCl), [H⁺] = 1.0 × 10⁻³ mol/dm³ (HCl) and T = 27 ± 1 °C. Pseudo-first order plots of log (Aₜ-A∞) versus time were made (where A₀ and Aₜ are the absorbance at time t and at the end of the reaction, respectively). Pseudo-first order rate constants, k₁ values were obtained from the slopes of the plots. The second order constants (k₂) were calculated as shown in Eq. (1):

$$k_2 = k_1/[\text{LSH}]$$

The effect changing the concentration of H⁺ on the reaction rate was examined by changing the H⁺ concentration within the range of (1.0–18.0) × 10⁻³ mol/dm³, while keeping [CoSalophen]⁺ and [LSH] of 2.0 × 10⁻⁴ mol/dm³ and 12.0 × 10⁻³ mol/dm³, respectively constant at T = 27 ± 1 °C and μ = 0.1 C² mol/dm³. The second-order acid-dependent variation with [H⁺] was achieved from the plot of k₂ against [H⁺] (Adetoro et al., 2011).

The effect of reaction rate as ionic strength of the medium of reaction is varied was examined within range of 0.04–0.18 C² mol/dm³, with constant concentrations of the reactants at 27 ± 1 °C. A plot log k₂ against 1/μ was also made.

The effect of added ions were examined for [Z] = 3.0–18.0 × 10⁻³ mol/dm³ ([Z] = Ca²⁺/SO₄²⁻) at constant [CoSalophen]⁺, [LSH] and μ. The effect of temperature on the reaction rates was studied within 300–325 K. The data were then considered using Eyring plot of ln k₂/θ.
versus $1/T$ and thermodynamic parameters also were determined at constant $[\text{CoSalophen}^+]$, $[\text{LSH}]$ and ionic strength. Free radical participation as the reaction is occurring was investigated by addition of 5 cm$^3$ of 0.02 mol/dm$^3$ solution of acrylamide to the mixture of reactions that has oxidized partially then addition of excess methanol.

During the reaction progress, comparison was made between the spectra of the mixture of reaction with that of the complex within the range of wavelength 400–700 nm. A graph of $1/k_1$ against $1/[\text{LSH}]$ of Michaelis-Menten type was also made.

3. Results and discussion

Stoichiometric results obtained for this study revealed a mole proportion to be 1 to 1 base on the reaction as depicted in Figure 3. The stoichiometric equation for the reaction is shown below:

$$2[\text{CoSalophen}]^+ + 2\text{LSH} \rightarrow 2[\text{CoSalophen}] + \text{LSSL} + 2\text{H}^+$$  \hspace{1cm} (2)

A result of stoichiometry analogous to this research was reported previously in the reduction of heteropoly 11-tungstophosphovanadate(V) by l-cysteine (Komiyama et al., 1999).

The plots of pseudo-first order, log $\left( \frac{A_t - A_\infty}{A_0 - A_\infty} \right)$ against time were linear to 80% completion of the reaction (Figure 4) suggesting a first order dependence in $[\text{CoSalophen}^+]$. The order with respect to [LSH] was determined from a plot log $k_1$ against log [LSH]. The slope of the plot was 0.97 (Figure 5). The values of the second order rate constants remained

![Figure 1. FT-IR spectrum of the Salophen ligand.](image1)

![Figure 2. FT-IR spectrum of the Co(III)Salophen complex.](image2)

![Figure 3. Absorbance versus mole ratio plot for the reaction of $[\text{CoSalophen}]^+$ and LSH at $[\text{CoSalophen}^+] = 2.0 \times 10^{-3}$ mol/dm$^3$, $[\text{H}^+] = 1.0 \times 10^{-3}$ mol/dm$^3$, $\mu = 0.1 \text{dS/m}$, $[\text{LSH}] = (0.5–7.0) \times 10^{-4}$ mol/dm$^3$, $T = 27 \pm 1$ $^\circ$C and $\lambda_{\text{max}} = 470$ nm.](image3)
The rate law for the reaction is represented by Eq. (3).

\[
\frac{d[\text{CoSalophen}^+]_{138}}{dt} = k_2 \left[\text{CoSalophen}^+\right]_{138} [LSH]_{138}
\]

where \( k_2 = (6.9 \pm 0.18) \times 10^{-1} \text{ dm}^3/\text{mol}./\text{s}. \)

A second order dependence in [LSH] was previously reported in the reaction of LSH by trans-dichlorotetraycynoplutinate(IV) (Shi et al., 1996), heteropoly 11-tungstophosphovanadate (V) (Sami et al., 2009) and 12-tungstocobaltate(III), while a fractional order dependence in [LSH] in the oxidation of L-cysteine by Corey’s reagent (Adari et al., 2006).

The reaction displayed acid independent and dependent pathways as obtained from a plot of \( k_2 \) versus \([H^+]\) presented in Figure 6. Acid dependence effect of this kind is represented by Eq. (4)

\[
k_{H^+} = a + b[H^+]
\]

\[a = 0.16 \text{ dm}^3/\text{mol}/\text{s} \text{ and } b = 464.16 \text{ dm}^6/\text{mol}^2/\text{s}.\]

The final rate scheme for the reaction is presented in Eq. (5)

\[
\frac{-d[\text{CoSalophen}^+]}{dt} = (a + b[H^+]) [\text{CoSalophen}^+][\text{LSH}]
\]

This nature of acid dependence is signifying that the un-protonated.
and protonated species of the reactant are reactive (Gupta and Gupta, 1984) as shown in Eq. (6).

\[
\text{LSH} + \text{H}^+ \rightleftharpoons \text{HLSH}^+ \tag{6}
\]

Similar results of acid dependence were reported by earlier researchers in the reaction of l-cysteine (Ayoko et al., 1993; Ayoko and Olatunji, 1983; Chakraborty et al., 2012).

From Table 1, the results indicated the reaction rate to be enhanced as the strength of ions in the medium of the reaction is increased. A straight line graph was obtained from a graph of log \( k_2 \) against \( \sqrt{\mu} \) with positive slope (+1.1) (Figure 7), indicating a positive effect of salt based Brunsted – Debye model. This indicates the participation of the same charged species with a magnitude of 1 at the slow step (Onu et al., 2009).

The reaction rate was found to be inhibited in the presence of added cations while anions catalyzed the reaction rate (Ca\(^{2+}\) and SO\(_4^{2-}\), Table 2). Catalysis of added ion suggests that the mechanism of the reaction is outer-sphere (Ibrahim et al., 2019). The formation of a gel was observed on adding acrylamide, which is a radical scavenger, to partially reacted mixture in excess methanol suggesting the presence of free radicals in the reaction (Alioke et al., 2012).

No shift in \( \lambda_{\text{max}} \) (470 nm) was observed when the spectrum of the reaction mixture was matched with that of [CoSalophen]\(^+\) (Figure 8). The quantitative test of Co\(^{2+}\) was established by adding a few drops of potassium thiocyanate in excess acetone to the reaction product. The blue solution formed in the reaction mixture showed the presence of Co\(^{2+}\) (Osunlaja et al., 2013). The presence of disulphide, LSSL was ascertained by comparing the reaction mixture’s spectrum after the completion of the reaction with the spectrum obtained when LSH was treated the H\(_2\)O\(_2\) which is known to oxidize thiols to disulphide (Amjad et al., 1977). A similarity in the spectra suggests oxidation of LSH to LSSL (Figure 10).

From the results obtained, Eqs. (7), (8), (9), and (10) is suggested to be the elementary steps involved for the reaction:

\[
\text{LSH} + \text{H}^+ \rightleftharpoons \text{HLSH}^+ \tag{7}
\]

\[
[\text{CoSalophen}]^+ + \text{HLSH}^+ \rightleftharpoons [\text{CoSalophen}] + \text{LS} + 2\text{H}^+ \tag{8}
\]

![Figure 7. Plot of log\( k_2 \) versus \( \sqrt{\mu} \) for the reaction of [CoSalophen]\(^+\) and LSH at [CoSalophen]\(^+\) = 2.0 \times 10^{-4} \text{ mol/dm}^3, [LSH] = 12.0 \times 10^{-3} \text{ mol/dm}^3, [\text{H}^+] = 1.0 \times 10^{-3} \text{ mol/dm}^3, \mu = (0.04–0.18) \text{ C}^2 \text{ mol/dm}^3, T = 27 \pm 1^\circ\text{C}, \text{ and } \lambda_{\text{max}} = 470 \text{ nm.}]

![Figure 8. Plot for spectrophotometric test for the redox reaction of [CoSalophen]\(^+\) and LSH at [CoSalophen]\(^+\) = 2.0 \times 10^{-4} \text{ mol/dm}^3, [LSH] = 12.0 \times 10^{-3} \text{ mol/dm}^3, [\text{H}^+] = 1.0 \times 10^{-3} \text{ mol/dm}^3, \mu = 0.1 \text{ C}^2 \text{ mol/dm}^3, T = 27 \pm 1^\circ\text{C}, \text{ and } \lambda_{\text{max}} = 470 \text{ nm.}]

### Table 2. The Effect of added anion and cation on the rate of reaction of [CoSalophen]\(^+\) and LSH at [CoSalophen]\(^+\) = 2.0 \times 10^{-4} \text{ mol/dm}^3, [LSH] = 12.0 \times 10^{-3} \text{ mol/dm}^3, [\text{H}^+] = 1.0 \times 10^{-3} \text{ mol/dm}^3, \mu = 0.1 \text{ C}^2 \text{ mol/dm}^3, T = 27 \pm 1^\circ\text{C}, \text{ and } \lambda_{\text{max}} = 470 \text{ nm.}

| [2] \text{(mol/dm}^3\text{)} | 10^3 [2], (mol/dm}^3\text{)} | 10^3 k_1, (s}^{-1}\text{)} | 10^3 k_2, (dm}^3\text{mol/s)} |
|-----------------|-----------------|-----------------|-----------------|
| \text{SO}_4^{2-} | 0.0             | 8.20            | 6.83            |
|                 | 3.0             | 8.39            | 6.99            |
|                 | 6.0             | 8.59            | 7.16            |
|                 | 9.0             | 8.67            | 7.22            |
|                 | 12.0            | 8.78            | 7.31            |
|                 | 15.0            | 8.95            | 7.45            |
|                 | 18.0            | 9.10            | 7.58            |
| \text{Ca}^{2+}  | 0.0             | 8.20            | 6.83            |
|                 | 3.0             | 7.14            | 5.95            |
|                 | 6.0             | 5.56            | 4.63            |
|                 | 9.0             | 4.14            | 3.45            |
|                 | 12.0            | 3.48            | 2.90            |
|                 | 15.0            | 2.55            | 2.13            |
|                 | 18.0            | 0.94            | 0.79            |
\[ \frac{1}{[\text{LSH}]} \text{ vs. } \frac{1}{[\text{CoSalophen}^+]} \]

Equation (7): 

\[ [\text{HLSH}^+] = K [\text{LSH}] [\text{H}^+] \]  

From the mechanism

\[ \text{Rate} = k_3 [\text{CoSalophen}^+][\text{HLSH}^+] + k_5 [\text{CoSalophen}^+] [\text{LSH}] \]  

From Equation (7): 

\[ [\text{HLSH}^+] = K [\text{LSH}][\text{H}^+] \]  

Substituting Eq. (12) in Eq. (11)

\[ \text{Rate} = k_3 K [\text{CoSalophen}^+] [\text{LSH}][\text{H}^+] + k_5 [\text{CoSalophen}^+] [\text{LSH}] \]  

Equ. (14) is equivalent to Eq. (5) where \( k_5 = a = 0.16 \text{ dm}^3/\text{mol}/\text{s} \) and \( k_3 K = b = 464.16 \text{ dm}^6/\text{mol}^2/\text{s} \).

4. Conclusion

The reaction of \([\text{CoSalophen}^+]\) and LSH in DMSO: H$_2$O; 1:4 media, displayed a stoichiometry of 1:1 based on the mole ratio. Second order overall was established for the reaction and the hydrogen ion concentration effect revealed the activeness of both the protonated and deprotonated form of the reductant. Positive salt effect based on the Brønsted-Debye was established during the course of the reaction. Free radical was detected to take part while intermediary complex with substantial stability was not detected as the reaction is progressing. From the outcomes, the reasonable mechanistic pathway for the reaction is suggested to be outer-sphere.

Declarations

Author contribution statement

S. Abdulsalam: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

S. O. Idris: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

G. A. Shallangwa, A. D. Onu: Contributed reagents, materials, analysis tools or data.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

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