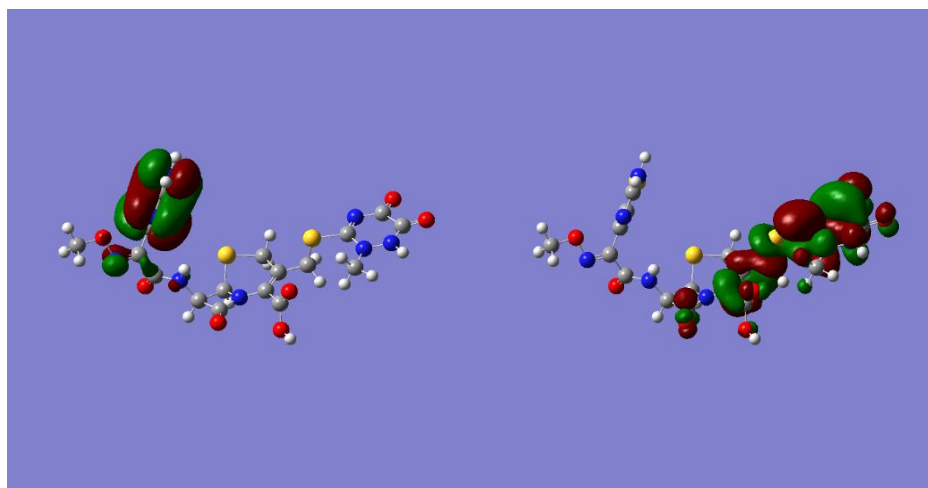


Cyclic Voltammetry of Aqueous CoCl_2 in the Presence of Ceftriaxone Disodium Salt (Cefs) at 298.65 K

Esam Arafa Gomaa^{a,*}, Mahmoud Nabil Abdel Hady^a, Mahmoud Hanfy Mahmoud^b, Doaa Ahmed El Kot^a

ARTICLE INFO	ABSTRACT
<p>Received: 21 October 2018 Revised: 2 November 2018 Accepted: 28 November 2018 Available online: 6 January 2019</p> <p>KEYWORDS</p> <p>Cyclic voltammetry Cobalt chloride Ceftriaxone disodium salt (Cefs) Oxidation reduction mechanisms Stability constants Kinetic parameters</p>	<p>Cyclic Voltammetry of Cobalt chloride with different concentrations was measured experimentally using DY2000 potentiostat. We used 0.1 M KCl as supporting electrolyte. Glassy carbon working electrode was used, purified and polished before use. The other two electrodes are platinum wire and Ag/AgCl electrode put in saturated KCl solution were used. The complex characters for the interaction of CoCl_2 with Ceftriaxone disodium salt (Cefs) were studied with the use of different Ceftriaxone disodium salt (Cefs) concentrations at 298.65 K. The oxidation, reduction mechanisms and the stability constants of the complexes formed are discussed. Scan rate and different kinetic parameters resulted from the interaction of CoCl_2 with Ceftriaxone disodium salt (Cefs) were also evaluated and their values were discussed.</p>

GRAPHICAL ABSTRACT



Cefs disodium salt HOMO in H_2O

Cefs disodium salt LUMO in H_2O

*Corresponding author's E-mail address: eahgomaa65@yahoo.com, Tel.: 00201065608647

^a Chemistry Department, Faculty of Science, Mansoura University, 35516 Mansoura, Egypt

^b Mathematics and Engineering Physics Department, Faculty of Engineering, Mansoura University, Mansoura, Egypt

Introduction

Complex interaction and stability constants for complexation of some metal salts with some organic ligands [1] can be explained by cyclic voltammetrically. Cobalt was found to form complexes by the interaction with nitrogen or hydroxyl groups in the ligands.

These can be studied by cyclic voltammetrically by measuring the shift in both anodic and characteristically cathodic peaks [2-10].

Here we prepared in our laboratory glassy carbon electrode which was used for measuring the redox behavior of CoCl_2 in the absence and presence of Ceftriaxone disodium salt (Cefs). The redox mechanisms and the stability constants for the complexes formed are discussed. Material Studio Package was used to perform Molecular Modeling Calculations by DMOL3 program [11].

Experimental

The used chemical like CoCl_2 were delivered from Sigma Aldrich. Co. Ceftriaxone disodium salt (Cefs) was provided from Pharco B international were used without any treatment to avoid the change in their characters. Potentiostat DY2000 apparatus was used for measuring the voltammograms at different scan rates. Purified water was used in practical work and duration with nitrogen for 10 minutes which were applied

to all the used solutions. Three electrode system was used for the development of cyclic voltammograms, Ag/AgCl electrode connected and filled with saturated KCl was used as the reference electrode. A platinum wire was used as the auxiliary electrode to save the cell from destruction. The glassy carbon electrode was polished with fine Al_2O_3 powder put above wool clean piece. The area of the used electrode is 0.0314 cm^2 . The Cobalt Ceftriaxone complex was prepared by using equimolar of Cobalt and Ceftriaxone Sodium solutions. They were refluxed for two and half an hour. The prepared complex was filtered, washed with Ethanol, and dried over CaCl_2 . The IR measurements were done for the formed complex.

Results and Discussion

Redox reactions of CoCl_2 in the absence of Ceftriaxone disodium salt (Cefs):

The electrochemical behavior, especially the redox processes of cobalt ions at the glassy carbon electrode were studied by measuring currents at the steady state.

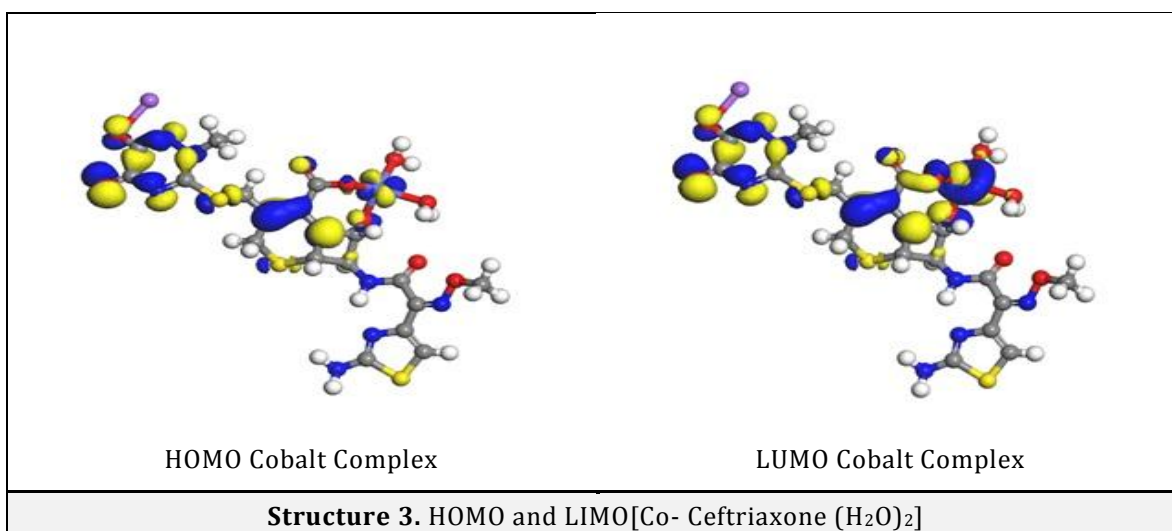
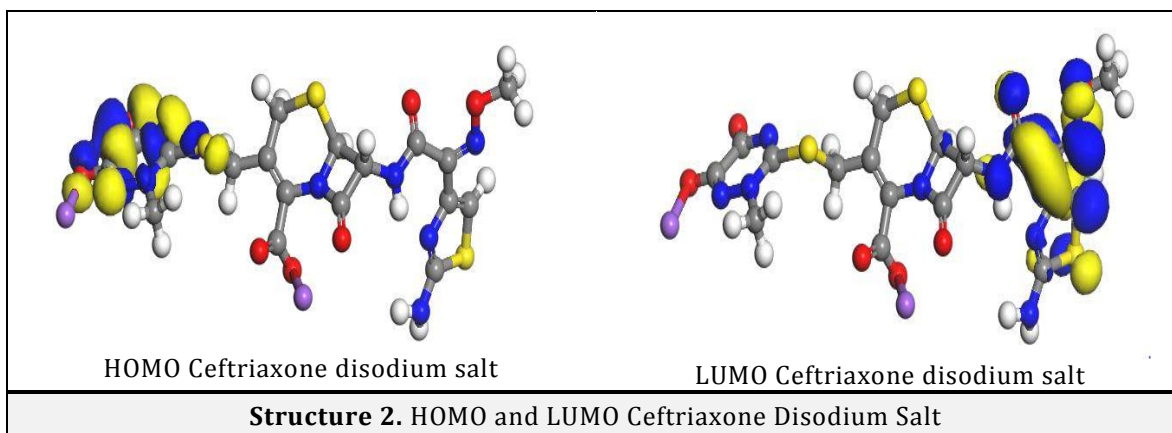
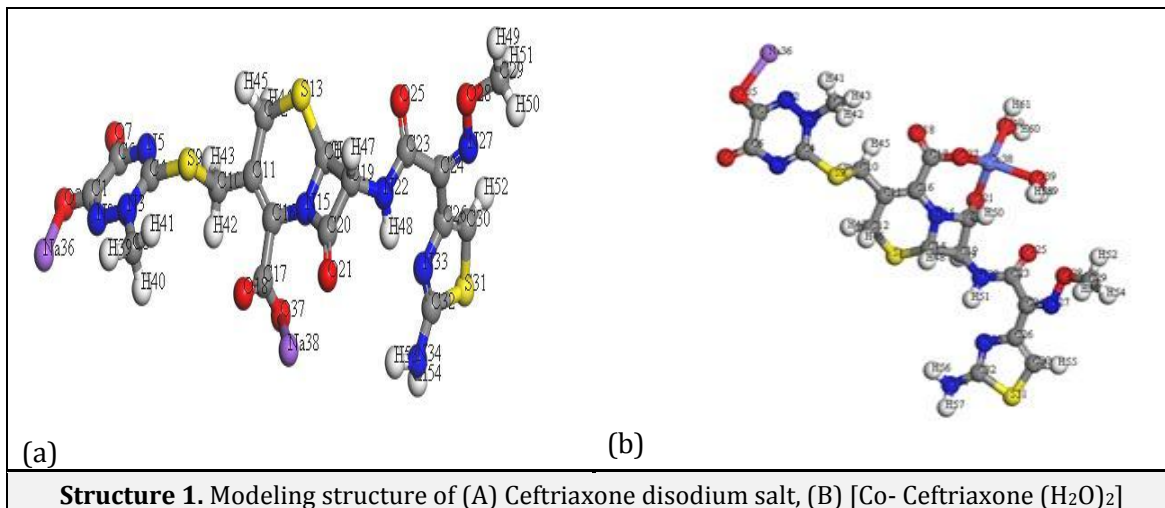
The cyclic voltammograms data were obtained and analyzed using equation (1) after Randles – Sevcik equation [6, 7, 9].

$$i_p = (2.69 \times 10^5) n^{3/2} A D^{1/2} \nu^{1/2} C \quad (\text{Eq. 1})$$

Where i_p is used currently in Ampere, A is the area of electrode surface in cm^2 , D is the diffusion coefficient in cm^2/sec , ν the scan

rate in v/s and C is the molar concentration

of depolarizer used.



The voltammograms were recorded using the working electrode from starting potential 1.5V till -1.5V.

Currents depended on the movement of the illustrative cobalt salt to the surface of the working electrode and the electron reaction of transfer. If the reaction took place reversibly, then the separation in the peak potentials, ΔE_p would close to a value of $58/n$ mV (at 25°C).

The potential difference can be calculated by the following equation [7-9]:

$$\Delta E_p = E_{pa} - E_{pc} \quad (\text{Eq. 2})$$

α_a : The Charge transfer coefficient of electrons which transfer in the rate determining step can be calculated by the following equation [7, 8]:

$$\alpha_a = 1.857 RT / (E_{pc} - E_{pc/2}) F \quad (\text{Eq. 3})$$

α coefficient is equal to 0.5 and $E_{pc/2}$ is half-wave potential of the cathodic peak.

The surface coverage Γ (The surface concentrations of electroactive species in mol.cm⁻²) can be calculated by applying the following equation [7, 9].

$$\Gamma = i_p / 4RT / n^2 F^2 A v \quad (\text{Eq. 4})$$

We can also, calculate the surface coverage from the quantity of charge which consumed during reduction or adsorption of the adsorbed layer by applying the following equation [10]:

$$Q = n F A \Gamma \quad (\text{Eq. 5})$$

The deviation from reversibility will happen if k (rate constant) is big.

Examples of irreversible reactions involve bond breaking and the reaction is known as an irreversible reaction.

Another important relation for reversible systems is the dependence of peak height on $(v)^{1/2}$, The square root of scan rate [12-19].

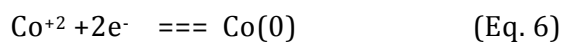
For quasi-reversible systems, the peak current is not proportional to $(v)^{1/2}$.

Some other researchers prefer to work with the potential at its half value for the peak current from the theoretical study to show that increase of potential rates, slow electron transfer makes a shift in peak potentials, this indicates no longer symmetric about the $E_{pc/2}$ for the reduction and oxidation reactions. Analysis of the redox reaction peaks was based on the relative heights of the anodic and cathodic peaks.

The effect of CoCl₂ concentrations was studied voltammetrically using 0.1M KCl as electrolyte supporting in the range from 1.5V to -1.5V at 298.65K. Their data has been given in Figure 2 and Table 1. We noticed one cathodic peak appears at ~ 1 V due to two electrons' reaction process.

The reduction of cobalt cations to zero valent ones at the glassy carbon electrode

can be explained as suggested mechanism:



The oxidation peak appeared and found at approximately $\sim 0.25\text{V}$ corresponding to the opposite reaction of reaction (6).

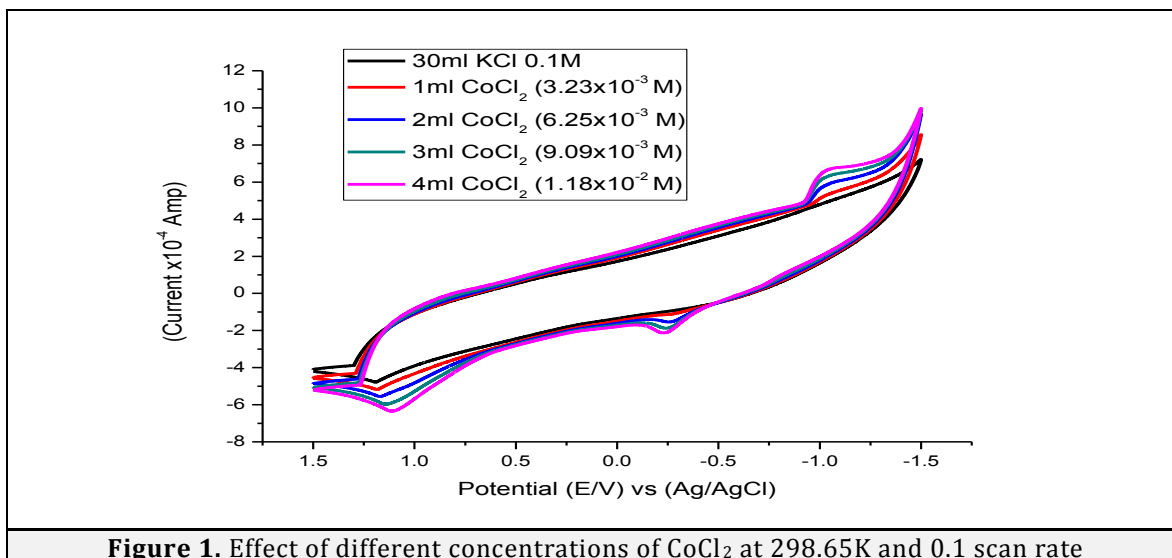


Figure 1. Effect of different concentrations of CoCl_2 at 298.65K and 0.1 scan rate

Table 1. Effect of different concentrations of CoCl_2 at 298.65K and 0.1 scan rate

[M] $\times 10^{-3}$	$E_{p,a}$	$E_{p,c}$	ΔE_p	$(-)I_{p,a}$ $\times 10^{-5}$	$I_{p,c}$ $\times 10^{-5}$	$I_{p,a}/I_{p,c}$	E°	D_a $\times 10^{-12}$	D_c $\times 10^{-12}$	αn_a	$K_s C$ $\times 10^{-6}$	Γ_c $\times 10^{-9}$	$(+)Q_c$ $\times 10^{-5}$	Γ_a $\times 10^{-9}$	$(-)Q_a$ $\times 10^{-5}$
3.23	-0.343	-1.008	0.665	2.19	3.04	0.721	-0.67	0.812	1.56	2.98 6	5.242	2.579	1.56	1.861	1.13
6.25	-0.291	-0.997	0.706	5.27	6.94	0.758	-0.64	1.248	2.17	1.83 8	6.340	5.895	3.57	4.472	2.71
9.09	-0.282	-0.983	0.701	7.56	9.82	0.770	-0.63	1.217	2.05	1.83 8	6.212	8.337	5.05	6.423	3.89
11.8	-0.254	-0.966	0.712	9.42	13.6	0.692	-0.61	1.126	2.35	6.82 7	6.303	11.54	6.99	7.997	4.85

Table 2. Effect of different scan rate of $1.18 \times 10^{-2}\text{M}$ CoCl_2 at 298.65K

ν	$E_{p,a}$	$E_{p,c}$	ΔE_p	$(-)I_{p,a}$ $\times 10^{-5}$	$I_{p,c}$ $\times 10^{-5}$	$I_{p,a}/I_{p,c}$	E°	D_a $\times 10^{-12}$	D_c $\times 10^{-12}$	αn_a	$K_s C$ $\times 10^{-6}$	Γ_c $\times 10^{-9}$	$(+)Q_c$ $\times 10^{-5}$	Γ_a $\times 10^{-9}$	$(-)Q_a$ $\times 10^{-5}$
0.1	-	-	0.71 2	9.42	13.6	0.69 29	-0.61	1.13	2.34 683	6.827	6.303	11.54 1	6.99	7.99732	4.85
0.05	-	-	0.75 2	5.14	7.28	0.70 59	-0.64	0.672	1.34 78	1.769 9	3.409	12.36 9	7.49	8.73078	5.29
0.02	-	-	0.72 3	2.32	7.07	0.32 79	-	0.342	3.17 895	1.448 1	2.257	30.03 6	18.2	9.848 8	5.97
0.01	-	-	0.69 4	1.91	5.06	0.37 74	-	0.463	3.25 06	3.413 5	1.731	42.95 3	26.0	16.21 2	9.82

we can notice from Figure 1 and calculated data in the Table 1 that by increasing concentration of cobalt ions is followed by the increase of anodic current I_{pa} , cathodic current I_{pc} , cathodic surface coverage Γ_c , anodic surface coverage Γ_a , the cathodic quantity of electricity Q_c and the anodic quantity of electricity Q_a . Also, electron transfer rate constant K_s showed a general tendency to large values by increasing $CoCl_2$ concentration.

Effect of scan rates of $CoCl_2$ alone was clear as presented in Figure 2 and calculated data in Table 2, using different scan rates (0.1, 0.05, 0.02, 0.01 V/sec).

We could notice by decreasing scan rates, both anodic and cathodic currents were decreased but other kinetic parameters like Γ_c , Γ_a , Q_c , and Q_a were increased.

we could deduce from the relation (i_p vs $v_{1/2}$) shown in Figure 3 that the electrochemical processes' diffusion were controlled.

Electrochemical behavior of Cobalt chloride in presence Ceftriaxone disodium salt (Cefs).

The effect of different concentrations of Ceftriaxone disodium salt (Cefs) on the redox behavior of $CoCl_2$ was illustrated in Table 3.

Also, one cathodic and one anodic peak were shown at the used temperature and

explained in Figure 4. The thermodynamic parameters for complexes K_c and ΔG_c were calculated and given in table (3) and (5) [15-27].

The standard transfer rate constant was calculated by the following equation:

$$K_s = k_o \exp(-\alpha n f / \frac{RT}{E - E^\circ}) \quad (\text{Eq. 7})$$

Where K_s was the forward rate constant, k_o was the electron transfer rate constant, E was the potential for reduction and α was the transfer coefficient. We consider $\alpha \cong 0.5$ as it depended on the surface free energies of the reactants and products.

The Gibbs free energy of interaction between cobalt chloride and ceftriaxone disodium could be calculated by the following equation:

$$\Delta G = -2.303 RT \log \beta_{MX} \quad (\text{Eq. 8})$$

By increasing the concentration of Ceftriaxone disodium salt (Cefs) as shown in Table 3 and Figure 4, E_{pa} shifted to less negative values but E_{pc} shifted to more negative values. Also, Cathodic parameters such as I_{pc} , D_c , Γ_c , and (+) Q_c were increased at first addition then they were decreased but other anodic parameters such as I_{pa} , D_a , Γ_a , and (-) Q_a were decreased.

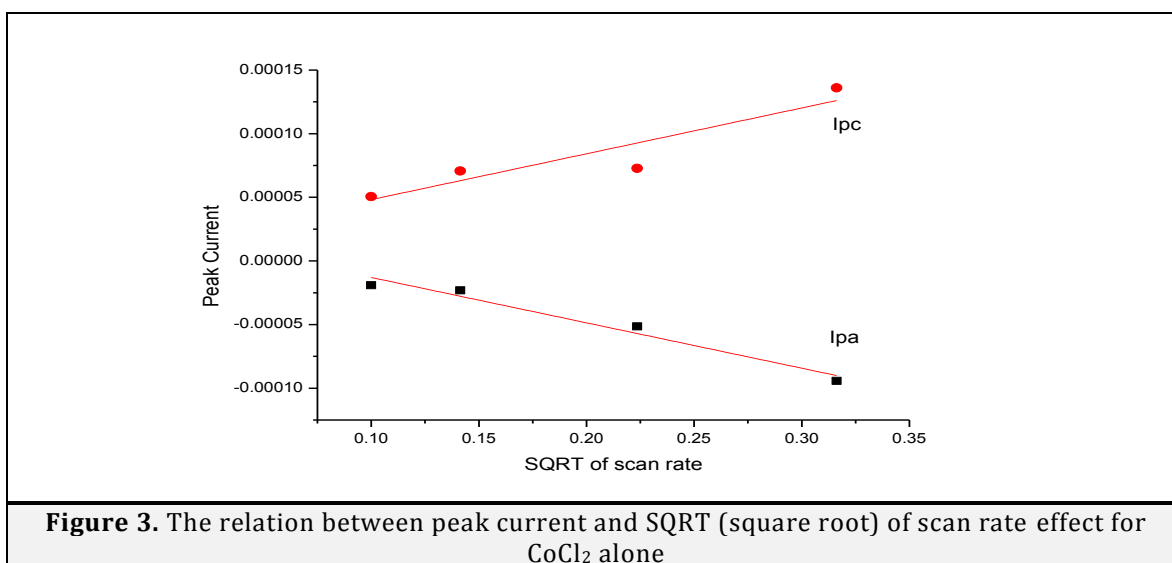
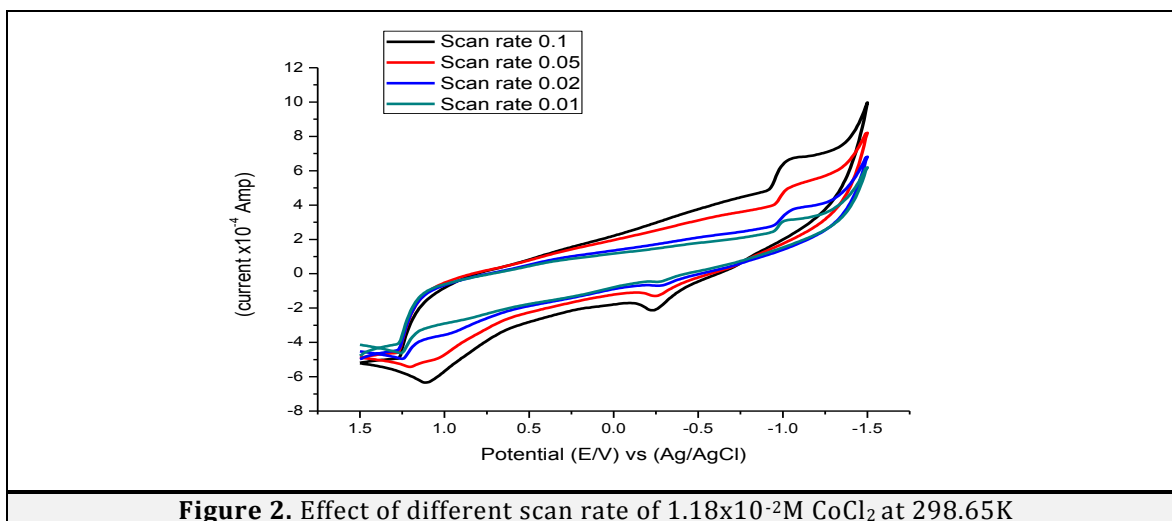


Table 3. Effect of different concentrations of Ceftriaxone disodium at 298.65K

[M] $\times 10^{-2}$	[L] $\times 10^{-3}$	$E_{p,a}$	$E_{p,c}$	ΔE_p	$(-)I_{p,a}$ $\times 10^{-5}$	$I_{p,c}$ $\times 10^{-5}$	$I_{p,a}/I_{p,c}$	E°	Da $\times 10^{-13}$	Dc $\times 10^{-13}$	α_{nac}	Ks C $\times 10^{-6}$	Γ_c $\times 10^{-9}$	(+) Qc $\times 10^{-5}$	Γ_a $\times 10^{-9}$	(-) Qa $\times 10^{-5}$
1.16	1.45	0.25 6	1.12 5	0.83 2	8.27	11.3	0.73 49	-0.690 5	8.960 7	16.6	0.9956	5.4573	9.563 03	5.79	7.028 11	4.26
1.14	2.86	0.23 2	1.14 9	0.91 7	5.74	4.39	1.14 815	-0.690 5	4.431	2.59	1.9115	2.8767	3.724 9	2.26	4.871 6	2.95
1.11	5.56	0.22 9	1.17 5	0.94 6	4.51	3.66	1.23 27	-0.702	2.900 5	1.91	1.2916	2.3973	3.108 5	1.88	3.831 97	2.32
1.07	9.33	0.21 2	1.19	0.97 8	2.41	2.58	0.93 397	-0.701	0.897 008	1.03	2.0778	1.5317	2.190 4	1.33	2.045 8	1.24

Effect of scan rates of CoCl_2 in presence of Ceftriaxone disodium by (2:1) molar ratio was clear as presented in Figure 5 and calculated data in Table 4, using different scan rates (0.1, 0.05, 0.02, 0.01 V/sec).

We noticed by decreasing scan rates, Both anodic and cathodic currents were

decreased but other kinetic parameters like Γ_c , Γ_a , Q_c , and Q_a were increased.

We could deduce from the relation (i_p vs $v_{1/2}$) shown in Figure 6 that the electrochemical processes' diffusion were controlled.

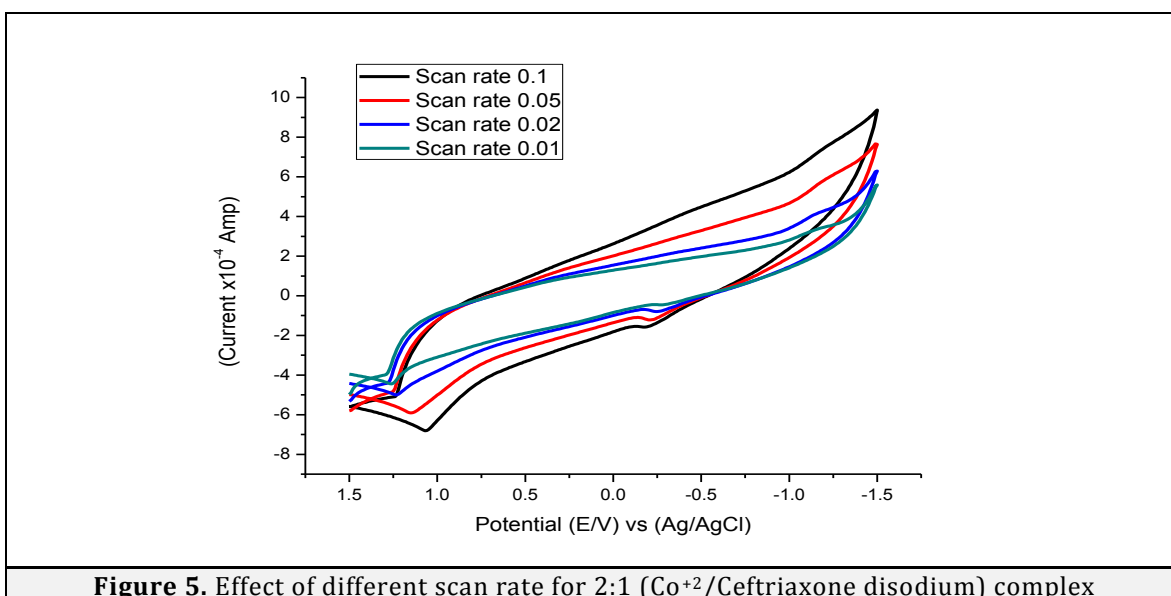
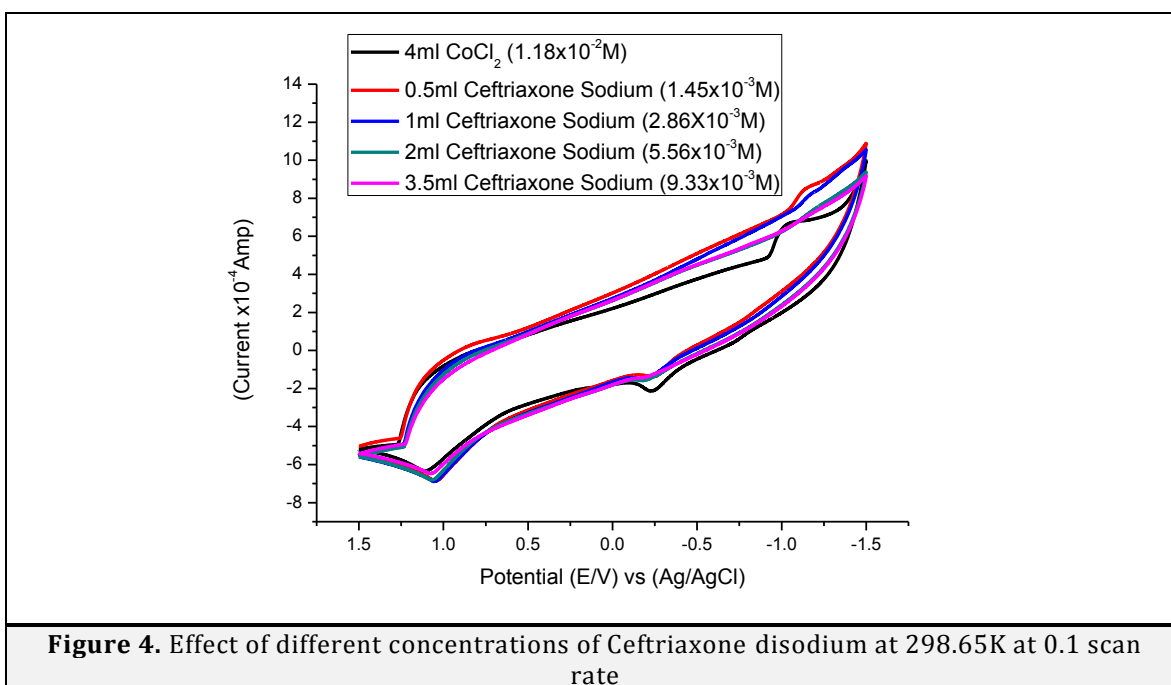
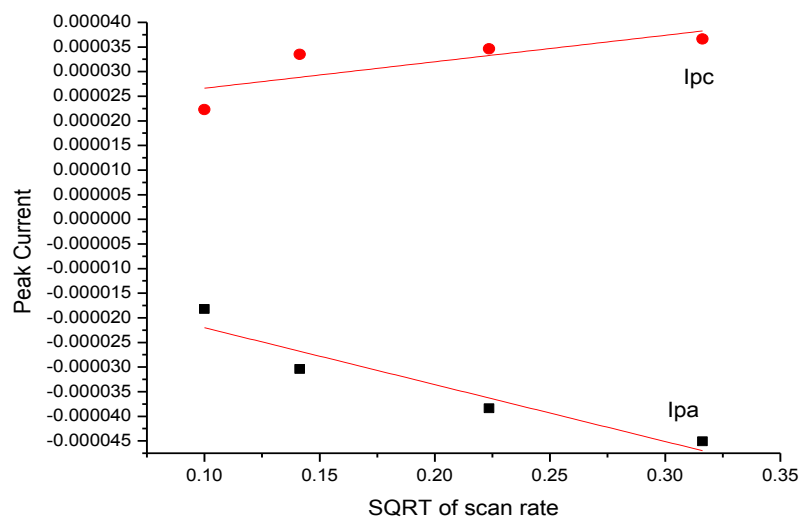


Table 4. Effect of scan rate 2:1 (Co²⁺/Ceftriaxone disodium)Complex

ν	$E_{p,a}$	$E_{p,c}$	ΔE_p	$(-)I_{p,a}$ $\times 10^{-5}$	$I_{p,c}$ $\times 10^{-5}$	$I_{p,a}/I_{p,c}$	E°	D_a $\times 10^{-13}$	D_c $\times 10^{-13}$	$\alpha n a$	$Ks C$ $\times 10^{-6}$	Γ_c $\times 10^{-9}$	$(+) Q_c$ $\times 10^{-5}$	Γ_a $\times 10^{-9}$	$(-) Q_a$ $\times 10^{-5}$
0.1	0.229	1.175	0.946	4.51	3.66	1.2327	-0.702	2.9005	1.9086	1.2916	2.397	3.1085	1.88	3.83197	2.32
0.05	0.233	1.165	0.932	3.84	3.46	1.10998	-0.699	4.2066	3.4143	1.9912	2.151	5.8797	3.56	6.52629	3.95
0.02	0.288	1.161	0.873	3.04	3.35	0.9062	0.7245	6.5771	8.0086	1.2576	1.883	14.238	8.63	12.9029	7.82
0.01	0.321	1.097	0.776	1.82	2.23	0.8174	-0.709	4.7181	7.0619	1.4481	1.187	18.9081	11.5	15.455	9.36

**Figure 6.** Relation between peak current and SQRT of scan rate on interaction between CoCl₂ and Ceftriaxone disodium**Table 5.** Stability constant for complex (Co²⁺/Ceftriaxone Sodium)

$[M] \times 10^{-2}$	$[L] \times 10^{-3}$	$(E_{p,1/2})_M$	$(E_{p,1/2})_C$	$\Delta E v$	J (L/M)	$\log \beta_j$	ΔG (KJ/mol)
1.16	1.45	-0.61	-0.684	0.074	0.125	2.8521	- 16.30901
1.14	2.86	-0.61	-0.6905	0.0805	0.25	3.3526	- 19.17107
1.11	5.56	-0.61	-0.702	0.092	0.5	4.2323	- 24.20146
1.07	9.33	-0.61	-0.701	0.091	0.875	4.8471	- 27.71727

The general trend for the Gibbs free energies of complexation and stability constant for the interaction between cobalt ions with ceftriaxone disodium salt forming complexes is increased by increasing L/M favoring more possibly to happen as shown in the Table 5. We also, studied the effect of scan rate on stability constant of the complex (2:1) (Co⁺²/Ceftriaxone disodium) as shown in the Table 6, we noticed that the general trend of stability constant (Log β_j) decreased by decreasing scan rates.

The IR and elemental analysis results

The IR spectrum of Ceftriaxone disodium showed six essential bands at 1742, 1649, 1606, 1537, 1399 and 3437 assigned to lactam carbonyl group $\nu(\text{C=O})$ [28], amide carbonyl group $\nu(\text{C=O-NH})$, azomethine nitrogen $\nu(\text{C=N})^*$ [29], $\nu(\text{COO})_{\text{asym}}$, $\nu(\text{COO})_{\text{sym}}$ [30] and $\nu(\text{NH})$, respectively. Ceftriaxone disodium; mp 236 °C. Anal. Calc. for $\text{C}_{18}\text{H}_{16}\text{N}_8\text{Na}_2\text{O}_7\text{S}_3$ (598.535): C, 36.12; H, 2.69; N, 18.72; Na, 7.68; O, 18.71; S, 16.07. Found: C, 36.01; H, 2.61; N, 18.6; O, 18.6; Na, 7.64; S, 16.08. Ceftriaxone disodium reacted as bidentate ligand in Co-Ceftriaxone complex, coordinating using lactam carbonyl group $\nu(\text{C=O})$, and $\nu(\text{COO})_{\text{asym}}$. This behavior of coordination was proved by the shift of the two bands to new wave

numbers. While; the other vibrations remained nearly at the same position. Besides that, the IR spectrum of the complex showed new two bands at 664 and 455 cm^{-1} which may be assigned to $\nu(\text{Co-O})$ [31] and $\nu(\text{Co-N})$ [32], respectively. Cobalt complex, pale brown color $[\text{Co}(\text{L})(\text{H}_2\text{O})_2]$; mp above 300°C. Anal. Calc. (647.518): C, 33.39; H, 3.11; N, 17.31; O, 22.24; S, 14.86; Co, 9.1 Found: C, 33.4; H, 3.01; N, 17.2; O, 22.21; S, 14.9; Co, 9.12. Where, the analysis of chloride content of the complex was determined using Volhard method and it was negative [33].

Computational Studies using DFT Method

Theoretical quantum mechanics calculations were done using Density Functional Theory (DFT) to predict the host-guest interaction between Ceftriaxone disodium and its Cobalt complex, this is shown in structure 1 which indicates the Molecular numbering structure of both Ceftriaxone disodium and Its Cobalt complex.

Molecular Parameters

Quantum parameters were determined of Ceftriaxone disodium salt and its cobalt complex as shown in the Table 7 such as, Total Energy, Binding Energy, Spin

Table 6. Effect of scan rate on stability constant for complex (2:1) (Co⁺²/Ceftriaxone Sodium)

ν	[M]x10 ⁻²	[L]x10 ⁻³	(E _{p,1/2}) _M	(E _{p,1/2}) _C	ΔE v	J (L/M)	Log β_j	ΔG (KJ/mol)
0.1	1.11	5.56	-0.61	-0.702	0.092	0.5	4.2323	-24.2015
0.05	1.11	5.56	-0.64	-0.699	0.059	0.5	3.11866	-17.8334
0.02	1.11	5.56	-0.6635	-0.7245	0.061	0.5	3.18615	-18.2194
0.01	1.11	5.56	-0.638	-0.709	0.071	0.5	3.5236	-20.1491

Table 7. Theoretical parameters for Ceftriaxone disodium and its Cobalt complex

Compound investigation	Theoretical parameters	Theoretical Data
Ceftriaxone disodium	Total Energy	-3179.925835 Ha
	Binding Energy	-9.250477 Ha
	Spin Polarization Energy	3.1205208 Ha
	Exchange- Correlation Energy	4.2229577 Ha
	Electrostatic Energy	-11.890909070 Ha
	Kinetic Energy	-4.7236894 Ha
	Sum of atomic Energy	-3170.6753570 Ha
	Dipole moment	8.24654 au
	E _{HOMO}	-4.774 ev
	E _{LUMO}	-2.369 ev
[Co-Ceftriaxone (H ₂ O) ₂]	Total Energy	-3337.998393 Ha
	Binding Energy	-10.1359306 Ha
	Spin Polarization Energy	3.5068866 Ha
	Exchange- Correlation Energy	4.6696072 Ha
	Electrostatic Energy	-14.4025548 Ha
	Kinetic Energy	-3.9280011 Ha
	Sum of atomic Energy	-3327.8624625 Ha
	Dipole moment	6.20795 au
	E _{HOMO}	-3.692 ev
	E _{LUMO}	-2.641 ev

Polarization Energy, Exchange-Correlation Energy, Electrostatic Energy, Kinetic Energy, Sum of Atomic Energy, Dipole moment, E_{HOMO} which was the energy of the highest occupied molecular orbital and E_{LUMO} which was the energy of the lowest unoccupied Molecular orbital. From the calculated data in Table 7 we could observe that:

1. The Binding energy of the Cobalt Ceftriaxone complex was higher than the binding energy of the Ceftriaxone

disodium which indicates the stability of the complex.

2. The dipole moment of Ceftriaxone disodium was higher than its cobalt complex indicating the activity of the free ligand.

3. The value of E_{HOMO} and E_{LUMO} were negative for both Ceftriaxone disodium and its cobalt complex indicating their stability. Also, they were presented in structure (2, 3).

4. The energy of HOMO was higher than

the energy of LUMO, so LUMO acted as electron donor but LUMO acted as an electron acceptor.

Conclusion

The redox mechanism for the solvation of CoCl_2 on using 0.1M KCl in the absence and presence of Ceftriaxone disodium salt (Cefs) was suggested based on the experimental data.

The thermodynamic parameters for L/M (Ligand/Metal), ceftriaxone disodium salt (Cefs) to Co^{+2} ratios were estimated and found to increase with the increase of Ceftriaxone disodium salt (Cefs) concentration which indicated the stability of the complex. Also, theoretical calculations were performed by the DFT method to support the experimental data.

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