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Redox Behavior of aqueous $CoCl_2.6H_2O$ in absence and presence of N-(benzo[d]thiazol-2-yl)-3-(2-(3,4-dihydronaphthalen-1(2H)-ylidene)hydrazinyl)-3-oxopropanamide (α-H 2 T) ligand at 298.15K

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Abstract: The kinetic and solvation parameters of $CoCl_2.6H_2O$ alone and in presence of (α-H_2T) ligand were evaluated. The glassy carbon electrode was used as working electrode with the other two electrodes, reference and auxiliary electrodes. The effect of cobalt concentrations on the estimated parameters was determined. In addition, the scan rate effect in absence and presence of (α-H_2T) ligand was studied. The impact of (α-H_2T) ligand concentrations on the estimated parameters was also evaluated. In addition to, the mechanism of the reaction was determined. Finally, the stability constants and Gibbs free energy for interaction of cobalt and the ligand were studied.

Keywords: kinetic, solvation, parameters ,$CoCl_2.6H_2O$, (α-H_2T) ligand **,** Stability constants,Gibbs free energy.

Introduction

Cobalt has many uses as it can be used as the main component in premium alloys, rechargeable batteries, and catalysts. It has magnetic properties, so it is mixed with Al and Ni to form potent magnets. Cobalt is utilized in electroplating as it is characterized by its attractive appearance, rigidity and resistance to corrosion. Cobalt chloride is exploited in electroplating, catalyst elaboration, porcelain, crock, painting of glass, and formation of vitamin B12. Moreover, it can be used as an additive to fertilizer and paste [1].

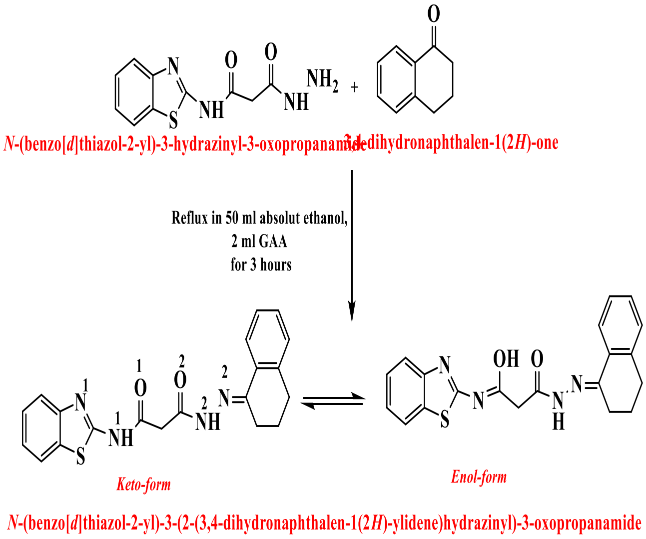
Schiff bases are compounds obtained by reaction of an aldehyde or ketone with primary amine under certain conditions. Schiff bases are known as azomethine or imine compounds, formed by replacing carbonyl group in aldehyde or ketone by amine or azomethine group [2. They have biological activities including antibacterial, antifungal, anti-inflammatory and antioxidant features. Azomethine group (RHC=R′) in Schiff,s bases show their pharmacological activities [3-8].

Schiff bases can form bonds through O, N or/ and S atoms [4, 5] so they are considered as bidentate or tridentate chelators and can also form four or six membered ring complexes. There are many studies on the stability of metal coordinating to bi-dentate Schiff bases O- and N- donors [4-7]. Tridentate donor ligands often form five or six coordinated complexes [8, 9].

Schiff bases are organic compounds that are largely utilized as catalysts, pigments, dyes, polymer stabilizers and intermediates in organic synthesis [10]. These compounds also show a wide range of biological activities [10, 11].

1. Experimental:

The ligand synthesis of (α-H_2T) was done as shown in the following schema:



Scheme 1: The outline synthesis of (α-H_2T) ligand

2.1. Chemicals

The used Cobalt chloride hexahydrate, sodium acetate have high purity and were obtained from Sigma Aldrich Company. Also, the new synthesized ligand (α-H_2T).

2.2. Cell and Instrument

The cell utilized consists of potentiostat PSTrace 5 attached to three electrodes. The electrodes are glassy carbon as working electrode with area 0.1256 cm^2, sliver/ sliver chloride as reference electrode and platinum wire as auxiliary electrode immersed in sodium acetate (0.1M) dissolved in 50% DMSO by volume DMSO/H2O. The nitrogen was pumped at each measurement to remove the oxygen gas.

3. Results and Discussion

3.1. Redox mechanism and cyclic voltammograms for different concentrations of $CoCl_2.6H_2O$

The oxidation and reduction mechanism of $CoCl_2.6H_2O$ was studied in 0.1M sodium acetate (50% DMSO) by volume DMSO/H2O at 298.15K. The potential range performed was from (-2.0 to 1.0) V. The formed voltammogram showed one cathodic peak at -1.3V and one anodic peak at 0.6 V [12, 13].

Reduction mechansim	
$Co^{+2} + 2e \rightarrow Co^0$	(1)
Oxidation mechansim	
$Co^0 \rightarrow Co^{+2} + 2e$	(2)

The impact of several concentrations (0.662, 1.320, 1.960& 2.600) $x10^{-3}$ M had been studied in 0.1M sodium acetate at 298.15K as displayed in Fig.1. The cathodic and the anodic peaks gradually increased by raising the concentration of cobalt ions which gives a proof that the reaction is supposed to be diffusion process. The anodic and cathodic diffusion coefficients are calculated from the relation between cobalt concentrations and peaks currents as in Fig.2. The values of diffusion coefficients are D_a= 1.483 $x10^{-4}$ cm^2/s, D_c= 16.775 $x10^{-4}$ cm^2/s.

Moreover, the solvation parameters like (Γ_c, Q_c, Γ_a & Q_a) increased as presented in Table (1). This demeanor could be explained as the increase in concentrations of the electro-active species [14].

The diffusion coefficient was determined by Randles-Sevick equation (3) [15-19] which elucidates the relation among current, diffusion coefficient and scan rate.

$$i_p = 0.4463\ n\ C\ A\ F\ (n\ \nu\ D\ F / RT)^{1/2} \quad (3)$$

i_p: The current of the peak, F: Faraday constant (96485.33 C/mol), A: The working electrode area (0.1256 cm^2), C: The bulk concentration of metal ion ($mol.L^{-1}$), D: Diffusion coefficient in $cm^2.S^{-1}$, ν: Scan rate ($V.S^{-1}$), R: Universal gas constant (8.314 $J.mol^{-1}.K^{-1}$)& T: The absolute temperature.

Thereafter, the difference between the cathodic and the anodic peak is calculated by the following equation (4) [18-23].

$$\Delta E_p = E_{pa} - E_{pc} \quad (4)$$

Therefore, the heterogeneous rate constant was estimated by equation (5) [18-23].

$$ks = 2.18\ [\alpha n_a\ D_C\ \nu\ F/RT]^{1/2} * \exp\ [n\ \alpha^2\ \Delta E_p\ F/RT] \quad (5)$$

(α): the coefficient of charge transfer, (n_a) the number of electrons participating in the step of determining rate, (F): Faraday constant, (T) The temperature in (K), suppose that α equals 0.5, the α_{na} can be calculated by the following equation (6) [19, 20].

$$\alpha_{na} = 1.857\ TR/\ F\ (E_{PC} - E_{PC/2}) \quad (6)$$

Additionally, (Γ) the surface coverage ($mol.cm^{-2}$) [18, 19] and the amount of charge (Q) [17] were evaluated from equations (7, 8)

$$\Gamma = i_p\ 4RT/n^2\ F^2\ A\ \nu \quad (7)$$

$$Q = n\ F\ A\ \Gamma \quad (8)$$

3.2. Impact of various scan rates for $CoCl_2.6H_2O$ alone

The influence of scan rates (0.02, 0.03, 0.04, 0.05& 0.10) $V.S^{-1}$ was determined at 298.15K for 2.6$x10^{-3}$ M of $CoCl_2.6H_2O$ as shown in Fig.3. The general trend is that the different solvation parameters like (Γ_c, Q_c, Γ_a & Q_a) become lower in values with the elevation of the scan rate demonstrating that the solvation mechanism is a diffusion one as shown in Table (2).

Also, the redox peaks currents increase with the increase of the scan rate. The relation between I_{pa} and I_{pc} with the SQRT of scan

rate is linear and can be used to calculate the diffusion coefficients of anodic and cathodic peaks as in Fig.4. This conduct demonstrated that the reaction is governed by the surface diffusion processes

[24].The values of diffusion coefficients are D_a= 6.304x10^{-11} $cm^2.s^{-1}$, D_c= 2.563x10^{-11} $cm^2.s^{-1}$.

3.3. Effect of (α-H_2T) ligand on the redox demeanor of $CoCl_2.6H_2O$

The influence of (α-H_2T) different concentrations on the electrochemical behavior of $CoCl_2.6H_2O$ was determined at 298.15K as shown in Fig.5. By increasing the concentration of (α-H_2T) ligand, the redox peaks currents and the kinetic parameters decreased. Also, the general trend of heterogeneous rate constant (ks) is to decrease by increasing hydrazine ligand (α-H_2T) concentration.

The behavior of decreasing the velocity of charge transfer indicates the binding between cobalt ions and (α-H_2T) [25] as observed in Table (3) The diffusion coefficients are calculated by using the relation between hydrazine ligand (α-H_2T) concentrations and peak currents as in Fig.6. The values are D_a=2.680x10^{-6} cm^2/s, D_c= 6.045x10^{-4} cm^2/s.

3.4. Impact of several scan rates in the existence of ligand (α-H_2T)

The influence of various scan rates (0.02,

0.03 0.04, 0.05 & 0.10) $V.S^{-1}$ for the interaction between cobalt ions and the (α-H_2T) ligand had been studied as offered in Fig.7 and Table (4). From the linear relation as in Fig.8, the diffusion coefficients are calculated and their values are: D_a= 3.078x10^{-11} cm^2/s, D_c=2.723 x10^{-11} cm^2/s.

3.5. The stability constant of for (Co- (α-H_2T))

The stability constant (Log βj) and Gibbs free energy (ΔG) have a tendency to increase with the increase of the (Ligand/Metal) ratio, confirming the ability to form a complex (Co-(α-H_2T)) as shown in Table (5). The scan rate effect was studied and it was found that the stability constant of the complex (Log βj) and the Gibbs free energy have largest value at scan 0.05 $V.S^{-1}$ as shown in Table (6). The stability constant (log β_{MX}) of the (Co-(α-H_2T)) complex was calculated from equation (7) [26-28].

$$\Delta E^\circ = 2.303(RT/\ F\ n) *(\ j \log C_{x+} \log \beta_{MX} = E^\circ_C - E^\circ_M \quad (9)$$

E°_C: is formal potential of the complex after every addition of (α-H_2T) (ligand), (E°_M) the formal potential of the last addition of metal (cobalt) before adding the (α-H_2T), (C_x) the ligand concentration in the solution, j (the coordination number of the complex)

The formal potential (E˚) was calculated from equation (8) [18-20].

$$E^\circ = (E_{pa} + E_{pc})/2 \quad (10)$$

The Gibbs free energy for the interaction between cobalt ions and the hydrazine ligand (α-H_2T) was determined from the stability constant by applying equation (9) [29-37]. The negative values of Gibbs free energies indicate that the reaction between cobalt ion and Schiff base ligand [Co-(α-H_2T)] is spontaneous.

$$\Delta G = -2.303\ RT \log \beta_{MX} \quad (11)$$

3.6. Mechanism of reaction

The mechanism of reaction is determined from the relation between $E_p/2$ /logν and scan rate ν and it is found that is followed IV mechanism that a reversible chemical reaction followed by reversible electron transfer [38] as shown in Fig.(9).

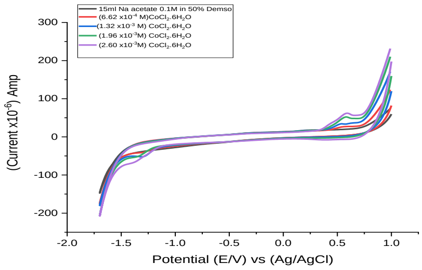


Fig.1. Cyclic voltammograms of different concentrations for $CoCl_2.6H_2O$.

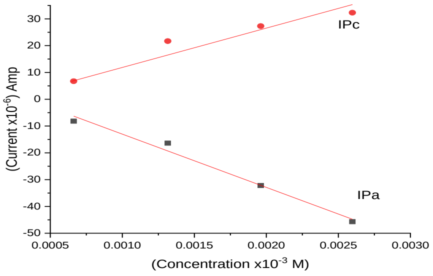


Fig.2. The relation between peak current and concentrations for different additions of cobalt chloride at scan rate 0.02$V.S^-$

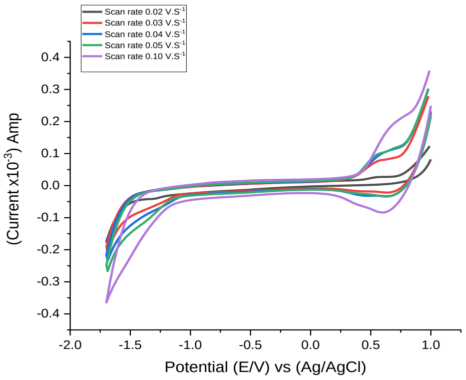


Fig.3. Effect of different scan rate for 2.6x10^{-3} M of $CoCl_2.6H_2O^{.1}$

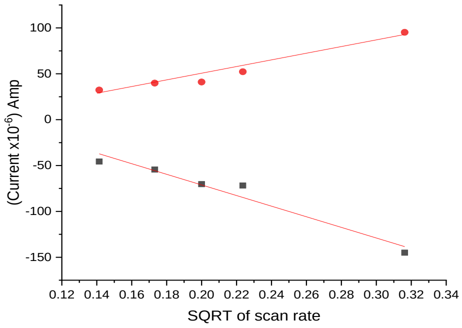
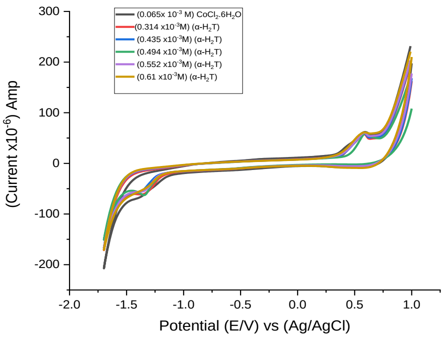


Fig.4. The relation between peak current and SQRT ($\nu^{1/2}$) of scan rate for redox peaks of $CoCl_2.6H_2O$ alone.



. **Fig.5.** Effect of several concentrations for (α-H_2T) at scan rate 0.02V.S^{-1}.

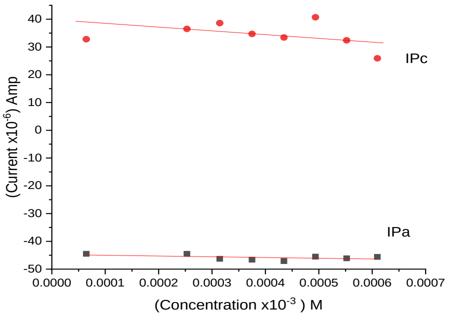


Fig.6. The relation between peak current and concentrations for different additions of (α-H_2T) at scan rate 0.02V.S^{-1}.

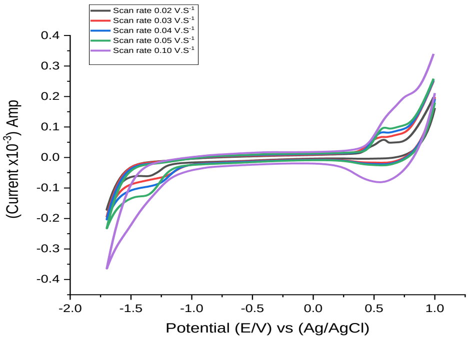


Fig.7. Effect of various scan rates for the formed complex [Co-(α-H_2T)].

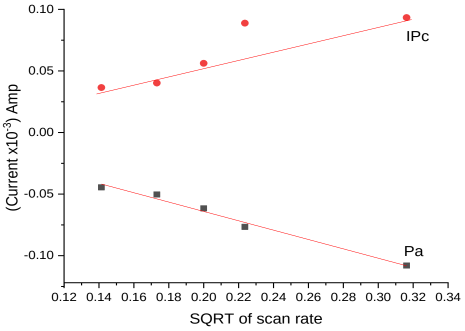


Fig.8. The relation between the current of peak and SQRT ($\nu^{1/2}$) of scan rate for redox peaks of cobalt chloride in presence of (α-H_2T).

Table (1) Kinetic and solvation parameters $CoCl_2.6H_2O$ alone at 0.02 scan rate 298.15K.

[M]x10^{3}mol.L^{-1}	**Ep,a (Volt)**	**Ep,c (Volt)**	**ΔEp (Volt)**	**(-)Ip,ax10^{6} Amp**	**Ip,cx10^{6} (Amp)**	**Ip,a/Ip,c**	**E° (Volt)**
0.662	0.527	-1.291	1.818	8.19	6.73	1.217	-0.382
1.32	0.528	-1.293	1.821	16.4	21.7	0.756	-0.383
1.96	0.56	-1.344	1.904	32.2	27.3	1.179	-0.392
2.60	0.602	-1.323	1.925	45.7	32.3	1.415	-0.361
D_ax10^{7} cm^{2}.s^{-1}	**D_cx10^{7} cm^{2}.s^{-1}**	**α_{nac}**	**ksc x10^{-12}**	**Γ_c x10^{9} mol.cm^{-2}**	**(+)Q_c x10^{5} (C)**	**Γ_ax10^{9} mol.cm^{-2}**	**(-) Q_a x10^{5}(C)**
8.395	5.67	1.363	3.93	0.713	1.73	0.868	2.10
8.528	14.9	0.769	5.08	2.300	5.57	1.738	4.21
14.803	10.6	0.561	18.4	2.894	7.01	3.413	8.27
16.993	8.49	0.430	21.7	3.424	8.30	4.844	11.70

Table (2) Effect of different scan rate for 2.6x10^{-3} M of $CoCl_2.6H_2O$ the redox peaks.

υ	Ep,a (Volt)	Ep,c(Volt)	ΔEp (Volt)	(-)Ip,a x10^6 Amp	Ip,c x10^6 (Amp)	Ip,a/Ip,c	E° (Volt)
0.02	0.602	-1.323	1.925	45.70	32.30	1.415	-0.361
0.03	0.531	-1.311	1.842	54.40	39.80	1.367	-0.390
0.04	0.547	-1.28	1.827	70.30	41.00	1.715	-0.367
0.05	0.542	-1.321	1.863	71.80	52.20	1.375	-0.390
0.10	0.644	-1.288	1.932	145.00	95.20	1.523	-0.322
Dax10^7 cm^2.s^{-1}	**Dc x10^7 cm^2.s^{-1}**	**α_{nac}**	**ksc x10^{-12}**	**Γ cx10^9 mol.cm^{-2}**	**(+) Qc x10^5 (C)**	**Γ a x10^{9}mol.cm^{-2}**	**(-) Q a x10^5(C)**
17.0	8.489	0.430	21.70	3.424	8.300	4.844	11.70
16.1	8.592	0.418	5.24	2.813	6.820	3.844	9.320
20.1	6.839	0.446	4.16	2.173	5.270	3.726	9.030
16.80	8.868	0.672	13.10	2.213	5.360	3.044	7.380
34.20	14.748	0.502	79.10	2.018	4.890	3.074	7.450

Table (3): Effect of different concentrations of (α-H_2T) on redox peaks at 0.02 (V.S^{-1}) scan rate.

[L] x10^3 mol.L^{-1}	Ep,a (Volt)	Ep,c(Volt)	ΔEp (Volt)	(-)Ip,a x10^6 Amp	Ip,c x10^6 (Amp)	Ip,a/Ip,c	E° (Volt)
0.065	0.613	-1.375	1.988	44.50	32.80	1.357	-0.381
0.314	0.596	-1.357	1.953	46.30	38.60	1.199	-0.381
0.435	0.562	-1.396	1.958	47.10	33.40	1.410	-0.417
0.494	0.587	-1.339	1.926	45.50	40.70	1.118	-0.376
0.552	0.576	-1.348	1.924	46.10	32.40	1.423	-0.386
0.610	0.557	-1.332	1.889	45.60	25.90	1.761	-0.388
Da x10^7 cm^2.s^{-1}	**Dc x10^7 cm^2.s^{-1}**	**α_{nac}**	**ksc x10^{-12}**	**Γ c x10^9 mol.cm^{-2}**	**(+) Qc x10^5 (C)**	**Γ a x10^9 mol.cm^{-2}**	**(-) Q a x10^5(C)**
16.30	8.870	0.548	85.20	3.477	8.430	4.717	11.40
18.593	12.90	0.795	62.70	4.092	9.920	4.908	11.90
19.728	9.92	0.513	48.60	3.540	8.580	4.993	12.10
18.640	14.90	0.823	40.50	4.314	10.50	4.823	11.70
19.372	9.57	0.628	27.30	3.434	8.320	4.887	11.80
19.187	6.19	0.663	11.40	2.745	6.650	4.834	11.70

Table (4) Effect of different scan rate for redox peaks of the formed complex [Co-(α-H_2T)].

υ	Ep,a (Volt)	Ep,c(Volt)	ΔEp (Volt)	(-)Ip,a x10^6 Amp	Ip,c x10^6 (Amp)	Ip,a/Ip,c	E° (Volt)
0.02	0.561	-1.339	1.9	44.50	36.50	1.219	-0.389
0.03	0.51	-1.245	1.755	50.30	40.20	1.251	-0.368
0.04	0.54	-1.276	1.816	61.60	56.20	1.096	-0.368
0.05	0.561	-1.356	1.923	76.60	88.80	0.885	-0.397
0.10	0.607	-1.351	1.923	108.00	93.30	0.885	-0.397
Da x10^7 cm^2.s^{-1}	**Dc x10^7 cm^2.s^{-1}**	**α_{nac}**	**ksc x10^{-12}**	**Γ c x10^9 mol.cm^{-2}**	**(+) Qc x10^5 (C)**	**Γ a x10^{9}mol.cm^{-2}**	**(-) Q a x10^5(C)**
16.960	11.4101	0.575	17.900	3.869	9.380	4.717	11.40
14.446	9.227	0.446	1.030	2.841	6.890	3.555	8.62
16.249	13.525	0.502	5.010	2.979	7.220	3.265	7.91
288.692	368.344	0.472	227.00	3.765	9.130	3.248	7.87
288.692	368.344	0.472	322.00	1.978	4.790	2.290	5.55

Table (5) Stability constant for (Co- (α-H_2T)) complex

(Ep,1/2)M	(Ep,1/2)C	ΔE (volt)	j(L/M)	log βj	ΔG (KJ/mol)
-0.361	-0.381	0.021	0.025	0.873	-4.982
-0.361	-0.364	0.004	0.075	0.622	-3.552
-0.361	-0.381	0.020	0.125	1.489	-8.499
-0.361	-0.401	0.041	0.150	2.333	-13.318
-0.361	-0.417	0.057	0.175	3.023	-17.258
-0.361	-0.376	0.016	0.200	1.785	-10.191
-0.361	-0.386	0.026	0.225	2.270	-12.959
-0.361	-0.388	0.027	0.250	2.466	-14.080

governed. The concentration impact of ligand (α-H_2T) on the solvation and kinetic parameters is determined and it is found tendency for the complexation [Co-(α-H_2T)] by the increasing of Gibbs free energy and stability constant values with (L/M) ratios. The redox demeanor of cobalt species was studied, it is found that the electrochemical mechanism of the process is a diffusion

Table (6) Effect of scan rate on stability constant for the (Co- (α-H_2T)) complex.

υ	**(Ep,1/2)M**	**(Ep,1/2)C**	**ΔE (volt)**	**log βj**	**ΔG (KJ/mol)**
0.02	-0.027	-0.389	0.362	18.833	-107.514
0.03	-0.390	-0.368	-0.023	5.836	-33.316
0.04	-0.367	-0.368	0.002	6.647	-37.948
0.05	-0.027	-0.397	0.370	19.087	-108.961
0.10	-0.322	-0.397	0.075	9.115	-52.034

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