



Cyclic Voltammetric studies of the interaction of cupric chloride with (Z)-4-oxo-4-((4-selenocyanatophenyl)amino) but-2-2nonic acid, (Chal) in KCl solutions using glassy carbon electrode.



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Abstract

To predict and analyze the complexation behavior between the chalcogen ligand (Chal) (Z)-4-oxo-4-((4-selenocyanatophenyl)amino) but-2-2nonic acid, and metal ion in aqueous solution cyclic voltammetric studies of copper chloride salt [CuCl₂] in absence and presence of chalcogen ligand (Chal), (Z)-4-oxo-4-((4-selenocyanatophenyl)amino) but-2-2nonic acid were performed. From these studies, some values were evaluated (the values of solvation and kinetic parameters [E_p (peak potential), I_p (peak current), D (Diffusion coefficient), ΔEP (peak potential difference), E_{1/2} (half wave potential), k_s (electron transfer rate constant), Γ (surface coverage) and Q_a (quantity of electricity)]. the effect of different scan rate and concentration of above the calculated quantities were evaluated and discussed.

Keywords: Cyclic voltammetry, chalcogen ligand, (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid I_p (peak current), E_{1/2} (half wave potential), k_s (electron transfer rate constant).

1. Note

- 1-Estimation of many cyclic voltammetry data for CuCl₂ alone and in the presence of (Z)-4-oxo-4-((4-selenocyanatophenyl)amino) but-2-2nonic acid, (Chal) ligand.
- 2- Estimation of many kinetic parameters for solvation.
- 3- Evaluating the different energy parameters for the salt and the complex interaction between the metal and ligand (Chal).
- 4- Evaluation of different solvation parameters for copper ions alone and in presence of (Chal) ligand in 0.1 M KCl.

2. Introduction

One of the most popular electrochemical technique is Cyclic voltammetry which used in a number of electrons transferred through oxidation or reduction process ,determination reactions mechanism , formal potential, a system stoichiometry, heterogeneous rate constants and diffusion coefficient of electroactive species. Cyclic voltammetry is simple, high sensitive and rapid technique. It is called cyclic because of the current is measured as a response of the applied potential, starting at the initial potential (E_i) and the

potential value varying in a linear manner up to the end value (E_f) final potential. At the end value of the potential, the direction of the potential scan is reversed and the scan takes place in the opposite direction at the same potential range [1-3].

3. Experimental:

1.Chemicals:

Water used in solutions is bidistilled with a specific conductivity (0.07 μS cm⁻¹ at 298.15 K). Copper chloride and KCl, were supplied from Sigma-Aldrich. Copper chloride used without any improvement but before using potassium chloride it was dried for 5 h. prepared chalcogen ligand, nitrogen was supported by Talkha Fertilizer Co.

2. Preparation of chalcogen ligand, (Chal), (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid:

Selenium dioxide(.67g) was added with stirring to the solution 0.2g malononitrite in 2ml DMSO, after 5 min the mixture become reddish(exothermic reaction with vigorous gas evolution after 15 min ,the mixture was diluted with 6 ml water, yellow precipitate was formed after cooling, precipitate was filtered, dried, crystallized from benzene[5].

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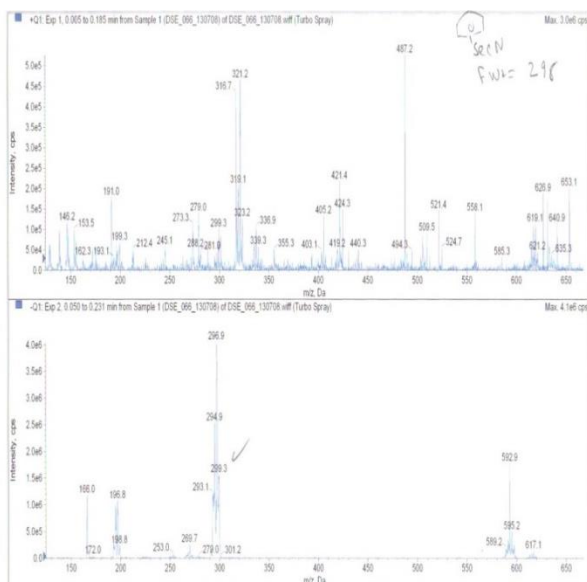
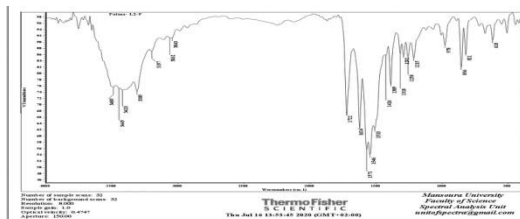
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3. Cyclic voltammetry measurement:

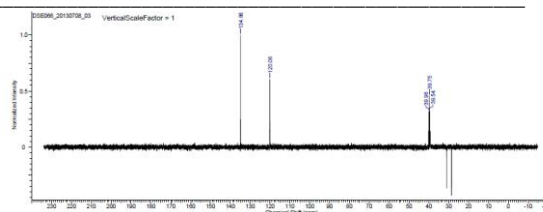
Cyclic voltammetry was performed using DY2000 multichannel Potentiostat connected with the electrochemical cell which consists of three electrodes Pt wire (0.5 mm diameter) as a counter electrode, Ag/AgCl (saturated 0.1 M KCl) as a standard electrode and glassy carbon electrode (GCE) as a working electrode with diameter surface area ($A = 0.503 \text{ cm}^2$), and the solutions was stirred by a magnetic stirrer. The solutions temperature inside the electrochemical cell is kept constant throughout the experiment. The working electrode was polished to a mirror-like surface after each run. After polishing the electrodes were rinsed with bidistilled water. The electrodes were placed in solutions consisting of the copper chloride concentration of stock (0.01 M) in potassium chloride (KCl 0.1 M). Before each run, an oxygen removal was performed by passing a high purity of nitrogen for 10 minutes. The data and graphs plot were analyzed by using Origin lab software (4).

Characterization of prepared compounds

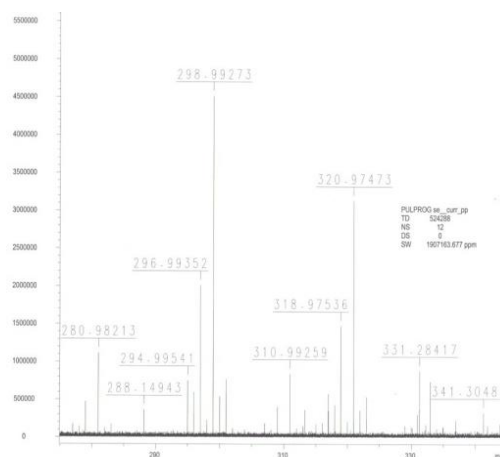
FT-IR



High resolution mass spectra



¹³C NMR



¹H NMR

• Results and Discussion

Cyclic voltammetry of CuCl_2 in absence of (Z)-4-oxo-4-((4-selenocyanatophenyl)amino) but-2-2nonic acid, (Chal) ligand.

The cyclic voltammetry for the redox behavior of CuCl_2 in 0.1M KCl at 298.15K was studied in range of from 0.5 to -0.8V. The reduction process proceeds in the range 0.5 to -0.8 V and the oxidation in the range -0.8 to 0.5V. Also effect of different scan rate was studied. The effect of different concentrations of CuCl_2 is presented in Fig. (1) and showed the change of Cu(II) to Cu(0) by the two reduction steps at 0.15 V and -0.4 V are clear in Figs.(1&2). One electron mechanism was suggested for each reduction step. CuCl_2 shows also two oxidation peaks at -0.1V and 0.35V indicating the reverse of the reduction process as explained in next suggested mechanism:



The electrochemical kinetic parameters like electron transfer rate constant k_s , cathodic surface coverage (Γ_c), anodic surface coverage (Γ_a), cathodic quantity of electricity (Q_c), anodic

quantity of electricity (Qa), diffusion coefficients for anode and cathode and α_{na} are presented in Table (1) [6-20]. The data given in Table (2) indicates the increase in all solvation and kinetic parameters by more adding CuCl_2 in solution indicating diffusion mechanism.

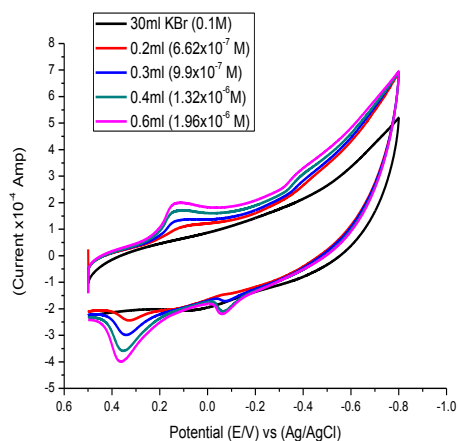


Fig.1. Effect of different concentrations of copper chloride at 298.15K and scan rate 0.1V.S^{-1} .

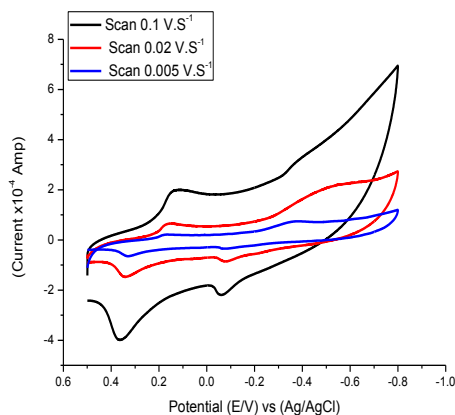


Fig.2. Effect of different scan rates of $1.96 \times 10^{-3}\text{M}$ copper chloride at 298.15K.

Table (1): Effect of different concentrations of copper chloride for first redox peaks at 298.15K and scan rate 0.1V.S^{-1}

[M] $\times 10^3$ mol.L ⁻¹	Ep,a Volt	Ep,c Volt	ΔE_p Volt	(-) Ip,a $\times 10^4$ Amp	Ip,c $\times 10^4$ Amp	Ip,a/Ip,c	E° Volt	Da $\times 10^4$ cm ² .s ⁻¹	Dc $\times 10^4$ cm ² .s ⁻¹	α_{na}	ksc	Γ_c $\times 10^8$ mol.cm ⁻²	(+) Qc $\times 10^5$ C	Γ_a $\times 10^8$ mol.cm ⁻²	(-) Qa $\times 10^5$ C
0.662	0.3069	0.1418	0.1651	0.225	0.278	0.81002	0.2243	1.5826	2.41	1.7122	0.459	0.9195	2.79	0.74483	2.26
0.990	0.3285	0.1515	0.177	0.510	0.444	1.1477	0.2400	3.6363	2.76	2.2391	0.632	1.4707	4.46	1.6879	5.11
1.32	0.3397	0.1679	0.1718	0.936	0.751	1.2464	0.2538	6.9359	4.46	2.2608	0.767	2.4856	7.53	3.0980	9.39
1.96	0.3472	0.1519	0.1953	1.96	0.954	2.05995	0.2496	13.772 4	3.25	1.9245	0.762	3.1581	9.57	6.5055	19.7

Table (2): Effect of different concentrations of copper chloride for second redox peaks at 298.15K and scan rate 0.1V.S^{-1}

v V.S ⁻¹	Ep,a Volt	Ep,c Volt	ΔE_p Volt	(-)Ip,a $\times 10^4$ Amp	Ip,c $\times 10^4$ Amp	Ip,a/Ip,c	E° Volt	Da $\times 10^4$ cm ² .s ⁻¹	Dc $\times 10^4$ cm ² .s ⁻¹	α_{na}	ksc	Γ_c $\times 10^8$ mol.cm ⁻²	(+)Qc $\times 10^5$ C	Γ_a $\times 10^8$ mol.cm ⁻²	(-) Qa $\times 10^5$ C
0.1	0.0767	0.3659	0.2892	0.442	0.352	1.2553	0.2213	0.6968	0.442	2.5311	0.823	1.1657	3.53	1.4633	4.43
0.02	0.0924	0.4282	0.3358	0.172	0.312	0.5491	0.2603	0.525	1.7414	1.4113	0.868	5.1727	15.70	2.8401	8.60
0.005	-0.099	0.3422	0.2432	0.0090	0.178	0.5061	0.2206	0.579	2.2628	1.5023	0.203	11.7927	35.70	5.9678	18.1

Table (3): Effect of different scan rates of $1.96 \times 10^{-3}\text{M}$ copper chloride at 298.15K for first redox peaks

v V.S ⁻¹	Ep,a Volt	Ep,c Volt	ΔE_p Volt	(-)Ip,a $\times 10^4$ Amp	Ip,c $\times 10^4$ Amp	Ip,a/Ip,c	E° Volt	Da $\times 10^4$ cm ² .s ⁻¹	Dc $\times 10^4$ cm ² .s ⁻¹	α_{na}	ksc	Γ_c $\times 10^8$ mol.cm ⁻²	(+)Qc $\times 10^5$ C	Γ_a $\times 10^8$ mol.cm ⁻²	(-)Qa $\times 10^5$ C
0.1	0.3472	0.1519	0.1953	1.96	0.954	2.05995	0.2496	13.772	3.25	1.9245	0.762	3.1581	9.57	6.5055	19.7
0.02	0.3257	0.1751	0.1506	0.556	0.359	1.5492	0.2504	5.5200	2.2984	2.3641	0.204	5.9425	18.0	9.2063	27.9
0.005	0.3111	0.1914	0.1197	0.203	0.110	1.8416	0.2513	2.9300	0.8646	4.0853	0.0060	7.2896	22.1	13.4244	40.7

Table (4): Effect of different scan rates of $1.96 \times 10^{-3}\text{M}$ copper chloride at 298.15K for second redox peaks

[M] $\times 10^3$ mol.L ⁻¹	Ep,a Volt	Ep,c Volt	ΔE_p Volt	(-) Ip,a $\times 10^4$ Amp	Ip,c $\times 10^4$ Amp	Ip,a/Ip,c	E° Volt	Da $\times 10^4$ cm ² .s ⁻¹	Dc $\times 10^4$ cm ² .s ⁻¹	α_{na}	ksc	Γ_c $\times 10^8$ mol.cm ⁻²	(+) Qc $\times 10^5$ C	Γ_a $\times 10^8$ mol.cm ⁻²	(-) Qa $\times 10^5$ C
0.6.62	0.0384	-0.4241	0.4625	0.0798	0.224	0.3571	-0.1929	0.1994	1.56	3.2342	9.84	0.7403	2.24	0.2644	0.801
0.990	-0.0893	-0.4006	0.3113	0.215	0.253	0.8483	-0.2405	0.6447	0.896	3.3994	1.69	0.8378	2.54	0.7107	2.15
1.32	-0.0788	-0.3769	0.2981	0.390	0.310	1.2561	-0.2279	1.2044	0.763	3.3266	1.35	1.0278	3.11	1.2910	3.91
1.96	-0.0767	-0.3659	0.2892	0.442	0.352	1.2553	-0.2213	0.6968	0.442	2.5311	0.823	1.1657	3.53	1.4633	4.43

From scan rate effect the different kinetic parameters mentioned before was done and included in Table (3, 4) and their effect were presented in Fig.(2) explaining that the reactions under or redox system is diffusion controlled. Figs (3) and (4) show straight line relations between peak currents for both anodic and cathodic peaks an square root of scan rate for both the first and second Cu ions peak couples (the first and the second couples).

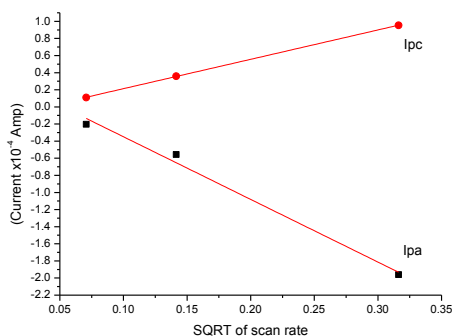


Fig.3. The relation between peak current and SQRT of scan rate for first redox peaks of copper chloride.

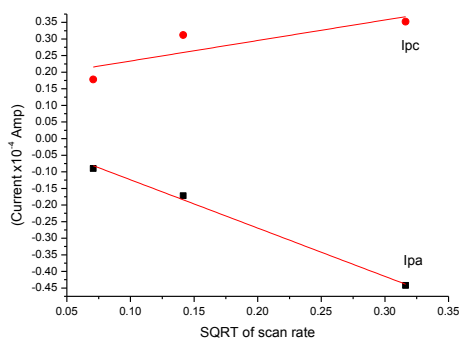


Fig.4. The relation between peak current and SQRT of scan rate for second redox peaks of copper chloride.

Electrochemical behavior of CuCl₂ in presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) ligand at 298.15K.

We used (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) as ligand by different concentrations ranging from 0.614x10⁻³ to 2.82x10⁻³ M.

From Fig. (5), by increasing (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) concentration shift in CuCl₂ waves specially reduction waves to more negative potentials. Also positive potential shift in oxidation peaks are observed indicating complex behavior observed and complexation between (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) and copper chloride.

The stability constant and Gibbs free energies of complexation were calculated and their values for 1:1 complex, metal to ligand ratio and represented in Table (5). These data are obtained from CuCl₂ wave analysis for the first reduction wave with the first oxidation peak. Also, the second reduction peak with the second oxidation peak.

The obtained thermodynamic parameters evaluated are representing in Table (5, 6).

The scan rate wave studied and from Fig. (6) supported the quasireversible and diffusion control of the reaction under study.

On drawing the relation between $\frac{i_{pa}}{i_{pc}}$ versus scan rate (v) for complex 1:1 (molar ratio) we can support the diffusion controlled system of redox reactions.

For more calculations, the stability complexation constant (Bc) and the Gibbs energies of complexation (Gc) [21-31] were evaluated following the redox peak and the data are given in Table (7).

From the data given in Table (7, 8) we conclude complexation reaction between the salt and (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal).

Most analyzing date obtained for CuCl₂ in presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal) are less than CuCl₂ a one data indicating complex reaction.

Most data obtained by analysis of the peaks are grater on analyzing the first peak couple than the second one, indicating more easily redo process for the first couple of peak process.

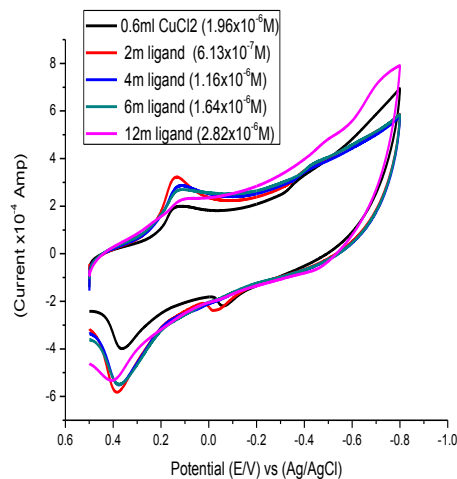


Fig.5. Effect of different concentrations of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) at 298.15K and scan rate 0.1V.S-1.

Table (5): Effect of different concentrations of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid , (Chal) ,for first redox peaks at scan rate 0.1V.S⁻¹

[L] x10 ³ mol.L ⁻¹	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-) Ip,a x10 ⁴ Amp	Ip,c x10 ⁴ Amp	Ip,a/Ip,c	E° Volt	Da x10 ⁴ cm ² .s ⁻¹	Dc x10 ⁴ cm ² .s ⁻¹	αnac	ks x10 ²	Γ c x10 ⁸ mol.cm ⁻²	(+) Qc x10 ⁵ C	Γ a x10 ⁸ mol.cm ⁻²	(-) Qa x10 ⁵ C
0.614	0.3608	0.158	0.2028	2.17	1.84	1.1764	0.2594	19.1	13.8	1.6693	1.58	6.1073	18.5	7.1848	21.8
1.16	0.352	0.1481	0.2039	2.10	1.41	1.4868	0.2501	20.1	9.09	1.5628	1.25	4.6746	14.2	6.9504	21.1
1.64	0.3525	0.1531	0.1994	1.63	1.09	1.4933	0.2528	13.6	6.10	1.5072	0.96 4	3.6207	11.0	5.4069	16.4
2.82	0.3807	0.1433	0.2374	1.26	0.859	1.4643	0.2620	10.95	5.11	1.2386	1.17	2.8456	8.62	4.1668	12.6

Table (6): Effect of different concentrations of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid , (Chal) for second redox peaks at scan rate 0.1V.S⁻¹

[L] x10 ³ mol.L ⁻¹	Ep,a	Ep,c	ΔEp	(-) Ip,a x10 ⁴	Ip,c x10 ⁴	Ip,a/Ip,c	E°	Da x10 ⁴ cm ² .s ⁻¹	Dc x10 ⁴ cm ² .s ⁻¹	αnac	ksc	Γ c x10 ⁸ mol.cm ⁻²	(+) Qc x10 ⁵ C	Γ a x10 ⁸ mol.cm ⁻²	(-) Qa x10 ⁵ C
0.613	-0.0437	-0.380	0.3366	0.626	0.301	2.0788	-0.212	1.59	0.367	2.9476	1.30	0.9968	3.02	2.072	6.28
1.16	-0.0538	-0.388	0.3346	0.338	0.278	1.2176	-0.2211	0.5212	0.352	3.4755	1.35	0.9192	2.78	1.119	3.39
1.64	-0.0725	-0.423	0.35	0.199	0.215	0.9252	-0.2475	0.2015	0.235	4.7523	1.51	0.7111	2.15	0.658	1.99
2.82	-0.0565	-0.464	0.4072	0.009	0.168	0.5124	-0.2601	0.0514	0.196	4.6572	2.41	0.5570	1.69	0.285	0.865

Table (7): Effect of different scan rate of 1:1 (Cu-(Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid) complex for first redox peak

v V.S ⁻¹	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-)Ip,a x10 ⁴ Amp	Ip,c x10 ⁴ Amp	Ip,a/Ip,c	E° Volt	Da x10 ⁴ cm ² .s ⁻¹	Dc x10 ⁴ cm ² .s ⁻¹	αna	ksc	Γ c x10 ⁻⁸ mol.cm ⁻²	(+)Qc x10 ⁵ C	Γ ax10 ⁻⁸ mol.cm ⁻²	(-)Qa x10 ⁵ C
0.1	0.3525	0.1531	0.1994	1.63	1.09	1.4933	0.2528	13.6	6.10	1.5072	0.964	3.6207	11.0	5.4069	16.4
0.02	0.3054	0.1715	0.1339	0.560	0.344	1.6255	0.2385	0.0008	0.0003	1.9166	0.178	5.7019	17.3	9.2684	28.1
0.005	0.3042	0.1749	0.1293	0.356	0.205	1.7388	0.2396	0.0013	0.0004	1.2830	0.0827	13.546	41.0	23.5531	71.4

Table (8): Effect of different scan rate of 1:1 (Cu-(Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid) complex for second redox peak

v V.S ⁻¹	Ep,a Volt	Ep,c Volt	ΔEp Volt	(-)Ip,a x10 ⁴ Amp	Ip,c x10 ⁴ Amp	Ip,a/Ip,c	E° Volt	Da x10 ⁴ cm ² .s ⁻¹	Dc x10 ⁴ cm ² .s ⁻¹	αna	ksc	Γ c x10 ⁻⁸ mol.cm ⁻²	(+)Qc x10 ⁵ C	Γ ax10 ⁻⁸ mol.cm ⁻²	(-)Qa x10 ⁵ C
0.1	-0.0725	-0.4225	0.35	0.199	0.215	0.9252	-0.2475	0.2015	0.235	4.7523	1.51	0.7111	2.15	0.6579	1.99
0.02	-0.095	-0.3842	0.2892	0.0849	0.0548	1.5483	-0.2400	0.1839	0.0767	4.1957	0.197	0.90759	2.75	1.4052	4.26
0.005	-0.0693	-0.439	0.3697	0.0553	0.0489	1.1299	-0.2542	0.3118	0.2442	0.1829	0.0820	3.2391	9.81	3.6600	11.1

Most of scan rate cyclic voltammetry data for CuCl₂ in presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal) are smaller than that of CuCl₂ alone the discussed medium indicating the complexation as a result for the interaction of CuCl₂ with (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal).

Electrochemical behavior of CuCl₂ in (Z)-4-oxo-4- The obtained thermodynamic parameters evaluated are representing in Table (10).

The scan rate wave studied and from Fig. (6) supported the quasireversible and diffusion control of redox reaction under consideration.

On comparing the different thermodynamic parameters mainly the stability constants and Gibbs free energies of complex interaction [9-33] for the reaction of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal) plus copper chloride by analysis of the two couples of peaks at different scan rates, we noticed almost equal values were obtained by analysis of the first and second couples of peaks as the data represented in Tables (9, 10).

The Gibbs free energies of complexation for 1:1 complex ratio between metal ions and (Chal) ligand gave approximately similar data by analyzing the first and second couple of copper ions peaks. This indicates strong complex is formed between the two reacting species.

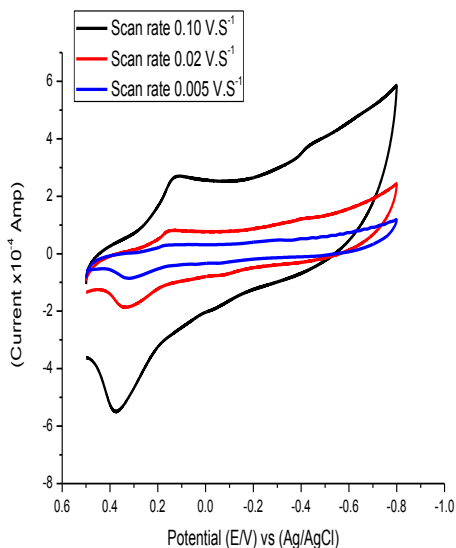


Fig.6. Effect of different scan rates for 1:1 ratio between copper and (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal) at 298.15K.

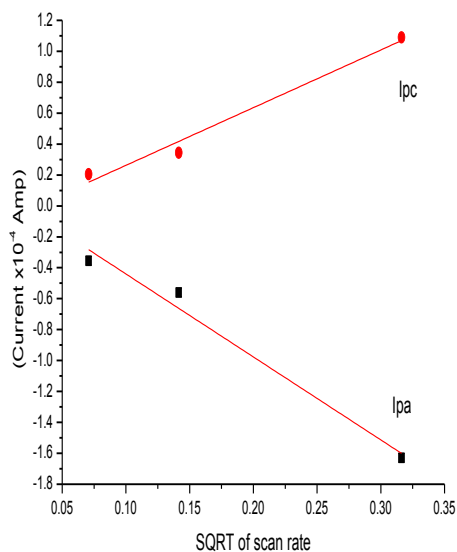


Fig.7. The relation between peak current and SQRT of scan rate for first redox peaks of copper chloride in presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal).

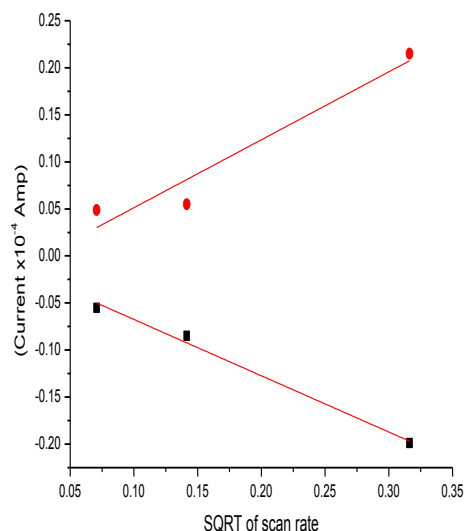


Fig.8. The relation between peak current and SQRT of scan rate for second redox peaks of copper chloride in presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2 nonic acid, (Chal).

Table (9): Stability constant for [Cu-(Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid] by analysis of the first peak couple.

(Ep,1/2)M Volt	(Ep,1/2) C Volt	ΔE Volt	J (L/M)	log β_j	ΔG kJ mol ⁻¹
0.24955	0.2594	0.00985	0.3333	1.2413	-6.9173
0.24955	0.25005	0.0005	0.6667	1.9667	-10.9598
0.24955	0.2528	0.00325	1	2.8416	-15.8356
0.24955	0.262	0.01245	2	5.31601	-29.6249

Table (10): Stability constant for [Cu-(Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid] by analysis of the second peak couple.

(Ep,1/2)M Volt	(Ep,1/2)C Volt	ΔE Volt	j (L/M)	log β_j	ΔG kJ mol ⁻¹
-0.2213	-0.212	-0.0093	0.333	2.261	-5.070
-0.2213	-0.2211	-0.0002	0.667	4.330	-10.892
-0.2213	-0.2475	0.0262	1	5.631	-18.050
-0.2213	-0.2601	0.0388	2	9.093	-32.167

3. Conclusions

CuCl₂ peaks are explained in absence and presence of (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) as a ligand. Different solvation parameters were evaluated by analysis of the two couples of peaks for copper ions. The thermodynamic complexation properties were evaluated and found insures the complexation interaction between copper chloride and (Z)-4-oxo-4-((4-selenocyanatophenyl) amino) but-2-2nonic acid, (Chal) studied here.

4. Conflicts of interest

The authors declare that they have no conflict with anybody about this work.

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