

Synthesis and Characterization of [Benzyl Hydrogen-4-(Dimethylamino) Benzylidene Carbon Hydrazonodithioate] and its Metal Complexes with [Co(II), Ni(II) and Cd(II)] Ions and Study Their Application as a Corrosion Inhibitor

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Abstract: The formation of Co(II), Ni(II) and Cd(II)-Complexes (C_1-C_3), respectively were studied with the synthesized Schiff base [Benzyl hydrogen-4-(dimethylamino) benzylidene carbon hydrazonodithioate] derived from reaction of benzyl hydrazine carbodithioate and 4-(dimethylamino) benzaldehyde. The suggested structures of the ligand and its complexes have been determined by using CHN analyzer, thermal analysis, FT-IR, UV-Visible, 1H NMR. Referring to these studies, the ligand coordinates as bidentate with metal ions through nitrogen atom of azomethane and sulfur atom of thiol group. The ratio of Metal to Ligand (M: L) as (1:2) and the suggested geometry of complexes was tetrahedral.

Key words: Schiff base ligand, bidentate, azomethine, corrosion inhibitors, polarization curves, tetrahedral

INTRODUCTION

Schiff bases are typically formed by the condensation reaction of a primary amine with carbonyl group of aldehyde or ketone. Schiff bases have two binding atoms or more and are known as bidentate or polydentate ligands (Nejo, 2009).

The aromatic Schiff bases are stable in aqueous acid or base solution according to their resonance whilst aliphatic Schiff bases unstable and decompose to carbonyl and primary amine compounds (Dawood and Saleh, 2007).

The importance of Schiff bases lies in the broad nature of industrial implementations and biological performance (Silveru and Parthasarathy, 2012). Benzyl hydrazine carbodithioate (SBDTC) is entertaining because to the reality that their derivatives have the ability to modify them in different ways by let in of various substituents (Break *et al.*, 2013). The Schiff bases have been used in drugs and the form of a particular molecule is known to be the most important factor affecting drug activity. The four main factors affecting drug properties are its capability to chelate with metal ions, its lipophilicity, steric effects and electronic effects (Al-Karkhi, 2014). This research includes the preparation and identification of recent Schiff base Ligand (L) proceeds from reaction benzyl hydrazine carbodithioate with 4-(dimethylamino) benzaldehyde and its complexes with Co(II), Ni(II) and Cd(II).

MATERIALS AND METHODS

All chemical materials used are of highest purity and used as obtained from the manufacture. Melting points were determined by using (Gallenkamp melting

point apparatus), UV-Vis. Spectra were performed in ethanol using (Shimadzu UV-1800) in the range 200-1100 nm and IR-spectra recorded on a (Shimadzu FTIR-8400 sec) in the range (400-4000 cm^{-1}) using KBr disk where all these measurements done at Department of Chemistry/Collage of Science/University of Baghdad. The 1H NMR spectra were gained on the (Advance III 400 Bruker 400 MHz) using DMSO as solvent at Iran/Isfahan University. The element analysis (CHN) was gained on a (Euro EA 3000) at College of Education for Pure Science (Ibn Al-Haitham), University of Baghdad.

Preparation of benzyl hydrazine carbodithioate

(SBDTC): Potassium hydroxide (5.6000 g, 0.1 mole) dissolved in 35 mL of ethanol 95% to this solution, hydrazine hydrate (4.9 mL, 0.1 mole) was added and the mixture was cooled in an ice-salt bath to 0°C. Carbon disulfide (6.1 mL, 0.1 mole) was added drop wise with constant stirring over a period of 1 h. The two layers that subsequently formed were separated using a separating funnel. The light brown lower layer was dissolved in 40% ethanol (35 mL) below 5°C. The mixture was kept in an ice-bath and to it, benzyl chloride (11.6 mL, 0.1 mole) was added drop wise with vigorous stirring. The sticky white which formed is filtered off, washed in ethanol and left to dry overnight in a vacuum desiccator over anhydrous silica gel (Al-Karkhi, 2014; George and Sarkis., 2006).

Synthesis of schiff base ligand [Benzyl hydrogen-4-(dimethylamino) benzylidene carbon hydrazonodithioate] (L): A solution of (1.98 g, 0.01 mole from benzyl hydrazine carbodithioate (SBDTC)

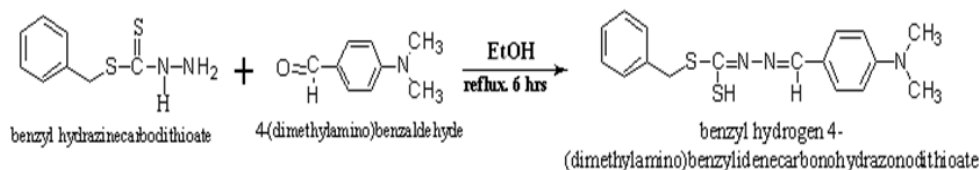


Fig. 1: Synthesis route of benzyl hydrogen-4-(dimethylamino) benzylidene carbon hydrazonodithioate (L)

in 50 mL of hot ethanol (99.9%) with catalyst (glacial acetic acid) was added to solution of 4-(dimethylamino) benzaldehyde (1.49 g, 0.01 mole) in 25 mL of hot ethanol (99.9%). The mixture was heated with stirring (reflux 6 h). The reaction mixture was then cooled in a snow bath to reach an integrated crystal. The resulting red product was washed with refrigerated ethanol and then dried using a vacuum drying container with the presence of anhydrous silica (Fig. 1). (Mughrabi *et al.*, 2010).

Synthesis of metal complexes: The synthesized complexes that have been prepared were in mole ratio (1:2) (M:L). Schiff base (L) (0.0658 g, 0.0002 mole) is dissolved in the absolute ethanol (10 mL of ethanol) and heated at 70°C with stirring, so, get a clear solution. A half molar of metal acetate dihydrate (0.0001 mole) [cobalt acetate dihydrate (0.0213 g), cadmium acetate dihydrate (0.0207 g) and nickel acetate dihydrate (0.0153 g) has been dissolved in absolute ethanol (10 mL) and then heated. Both solutions were mixed together and heated with stirring (reflux 6 h). Then leave the mixture cool down and filter out the formed precipitate.

RESULTS AND DISCUSSION

The Schiff base ligand was prepared by the condensation reaction. The element micro analysis data of the prepared (L) and its complexes with [Co(II), Ni(II) and Cd(II)] ions are in good congruence with the calculated data. The expected formula of new prepared complexes is scheduled in Table 1. The solvothermal decomposition of the prepared complexes using the hydrothermal method can be carried out where the complexes are considered as a precursor and the remaining material is expected to be (cobalt, copper and cadmium) sulfides as nanoparticles.

Infrared of ligand and its metal complexes

Infrared Spectrum of prepared Benzylhydrazine Carbodithioate (SBDTC): The infrared spectrum of SBDTC was presentation absorption peaks at (3303 and 3388 cm^{-1}) attributed to stretching vibration symmetric and asymmetric of free $\nu(\text{NH}_2)$ group (Asiri *et al.*, 2010; Csw *et al.*, 2013; A-Hasani *et al.*, 2011). The band appeared at 3178 cm^{-1} was attributed to stretching

vibration of $\nu(\text{NH})$ (Asiri *et al.*, 2010; Xiaoyang *et al.*, 2012). The band observed at (1610 cm^{-1}) was assigned to vibration of $\nu(\text{C}=\text{N})$. The sharp band appeared at 1047 cm^{-1} was assigned to vibration of $\nu(\text{C}=\text{S})$ (Chan *et al.*, 2008). The band appeared at 1346 cm^{-1} was attributed to vibration of $\nu(\text{N}-\text{N}-\text{C})$. The band appeared at 952 cm^{-1} was attributed to vibration of mixed $\nu(\text{C}-\text{N})$ and $\nu(\text{N}-\text{N})$ (Ameram and Othman, 2013). The IR spectrum of SBDTC is shown in Fig. 2.

Infrared spectrum of p-dimethylaminobenzaldehyde:

The infrared spectrum of (PDMAB) was showed absorption band at 1662 cm^{-1} attributed to stretching vibration of free $\nu(\text{C}=\text{O})$ group. The IR spectrum of p-dimethyl amino benzaldehyde was shown in Fig. 3 and 4.

IR of ligand [Benzyl hydrogen-4-(dimethylamino) benzylidene carbon hydrazonodithioate]:

The IR spectrum of the Ligand (L) show up a sharp absorption band at 1608 cm^{-1} which was attributed to stretching vibration of $\nu(\text{C}=\text{N})$. The band appeared at 3018 cm^{-1} was attributed to vibration of $\nu(\text{OH})$ of water (Saleem and Sultan, 2014). The sharp peak appeared at 954 cm^{-1} was attributed to $\nu(\text{C}-\text{S})$ (Alwan, 2012). IR spectrum of ligand showed no more $\nu(\text{N}-\text{H})$ peak but do not exhibits a $\nu(\text{C}=\text{S})$ band, since, the thion group was relatively unsteady in the monomeric form and it has the ability to change into more stable thiol form by enethiolization process. Benzyl hydrazine carbodithioate have been showed a band which appeared at 3303 and 3388 cm^{-1} attributable to the stretching vibration of $\nu(\text{NH}_2)$ group which disappeared in the ligand while the peak of carbonyl $\nu(\text{C}=\text{O})$ showed at 1662 cm^{-1} in the p-dimethylaminobenzaldehyde which was disappeared in the ligand. These indications refer to the formation of the ligand. The IR spectrum of this ligand is shown in Fig. 4.

FT-IR Spectra of L-complexes: The IR spectrum of Ligand (L) was show up a sharp absorption band at 1608 cm^{-1} which referred to stretching vibration of $\nu(\text{C}=\text{N})$ which was change location to (1604, 1602 and 1598 cm^{-1}) in the cobalt, nickel and cadmium complexes, respectively (Aliyu and Sani, 2011; Pulak and Sukalyan, 2011). IR spectra of ligand and its complexes show no

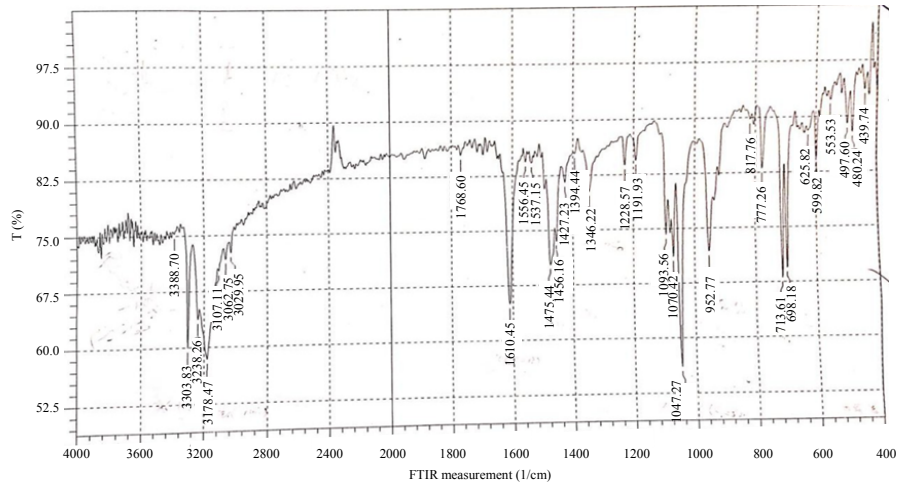


Fig. 2: The IR spectrum of benzyldithiocarbamate

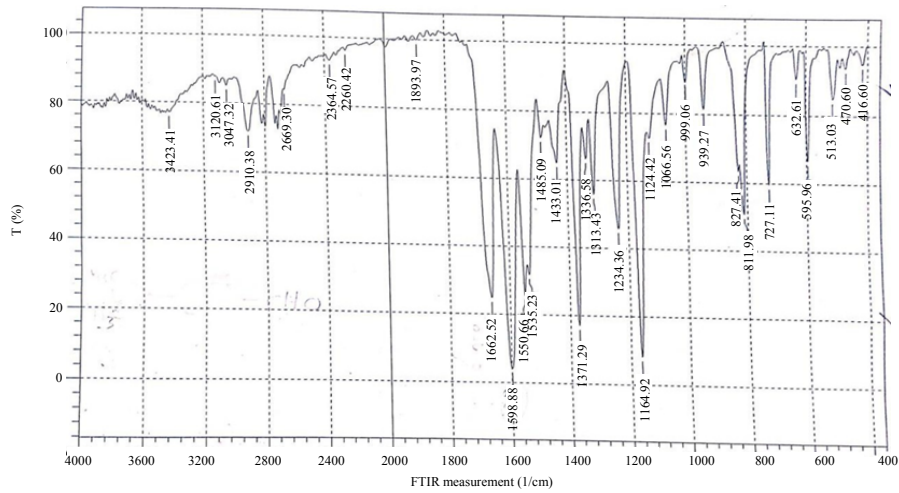


Fig. 3: The IR spectrum of p-dimethyl amino benzaldehyde

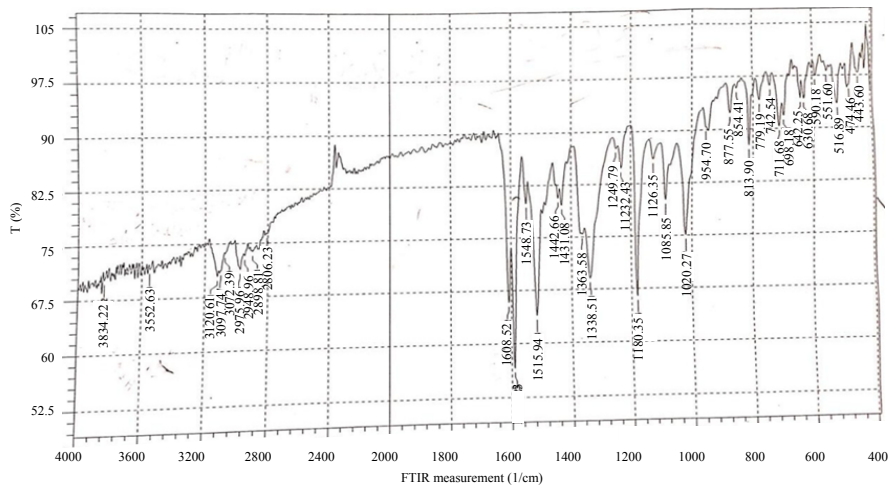


Fig. 4: The IR spectrum of ligand [Benzyldithiocarbamate-4-(dimethylamino)benzylidene] (L)

Table 1: Physical properties and analytical data of ligand and its metal complexes

Symbol	Color	Formula structure	Mwt. (gmol^{-1})	mp ($^{\circ}\text{C}$)	Micro element analysis found (Cal.)		
					C (%)	H (%)	N (%)
L	Red	$[\text{C}_{17}\text{H}_{19}\text{N}_3\text{S}_2] \cdot 0.5\text{H}_2\text{O}$	338.0	88-90	60.89 (60.35)	6.02 (5.92)	12.93 (12.42)
Co(L)_2	Dark-brown	$[\text{C}_{34}\text{H}_{36}\text{N}_6\text{S}_4\text{Co}] \cdot 0.5 \text{EtOH} \cdot 0.5\text{H}_2\text{O}$	764.9	132-135	55.06 (54.9)	5.92 (5.22)	10.69 (10.98)
Ni(L)_2	Brown	$[\text{C}_{34}\text{H}_{36}\text{N}_6\text{S}_4\text{Ni}]$	734.6	>210	57.97 (55.53)	5.36 (4.90)	11.97 (11.40)
Cd(L)_2	Yellow	$[\text{C}_{34}\text{H}_{36}\text{N}_6\text{S}_4\text{Cd}] \cdot 2\text{H}_2\text{O}$	822.4	136-140	48.76 (49.61)	4.38 (4.86)	10.67 (10.27)

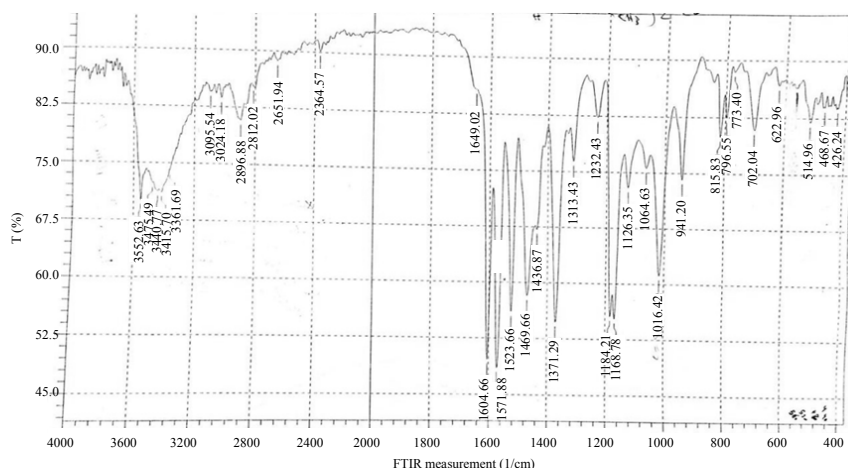


Fig. 5: The IR spectrum of cobalt complex

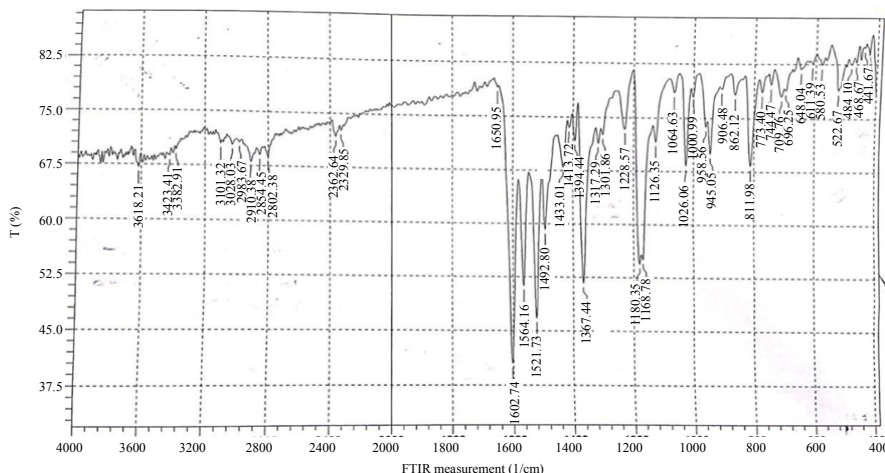


Fig. 6: The IR spectrum of nickel complex

more $\nu(\text{N-H})$ peak but do not exhibit a $\nu(\text{C}=\text{S})$ band, since, the thion group was relatively unstable in the monomeric form and has a tendency to change into more stable thiol form (SH) which disappeared when the complexes are formed (Tarafder *et al.*, 2002) while absorption bands appeared at 568, 580 and 568 cm^{-1} were referred to $\nu(\text{M-N})$ for complexes, respectively (Asiri and Badahdah, 2007; Mohamed *et al.*, 2006). The absorption bands which pointed at 426, 420 and 426 cm^{-1} were

pointed out to stretching vibrations of $\nu(\text{M-S})$ for ligand complexes, respectively (Uddin *et al.*, 2014). These observations indicate that the complexation occurred between the Schiff base and the metal ion in the solid state. Finally, observed that the weak absorption bands show up at 3409.91-3446.56 cm^{-1} were pointed out to $\nu(\text{H}_2\text{O})$ and $\nu(\text{OH})$ (Mohammed, 2011; Adedibu and Joshua, 2011) (Fig. 5-7).

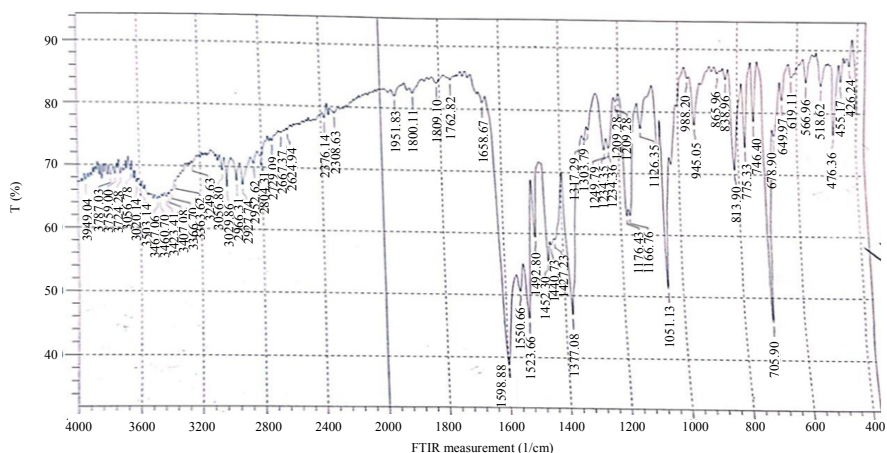


Fig. 7: The IR spectrum of cadmium complex

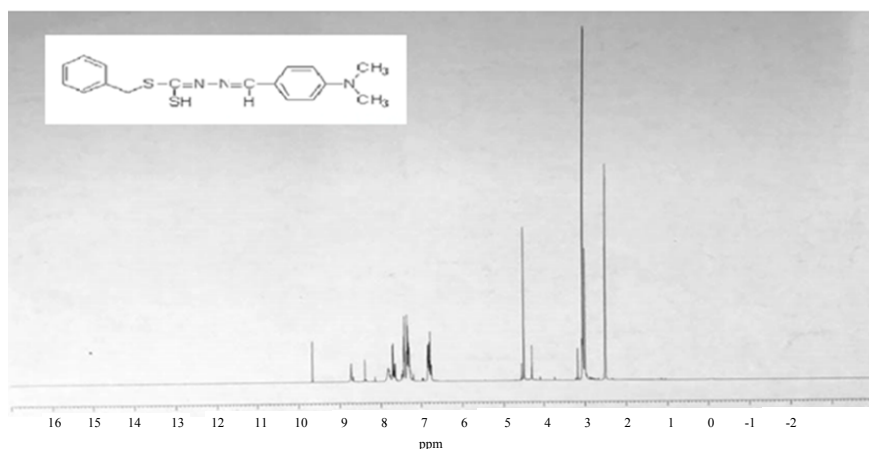


Fig. 8: The ¹H NMR spectrum of ligand [Benzyl hydrogen-4-(dimethylamino) benzylidene carbon hydrazonodithioate]

Table 2: FT-IR spectral data of ligand and its metal complexes

Symbol	$\nu(\text{C}=\text{N} \text{ cm}^{-1})$	$\nu(\text{M}-\text{N} \text{ cm}^{-1})$	$\nu(\text{M}-\text{S} \text{ cm}^{-1})$
L	1608	--	--
Co(L) ₂	1604	568	426
Ni(L) ₂	1602	580	420
Cd(L) ₂	1598	568	426

The IR spectra of L-complexes are shown in Fig. 5-7 and the spectral data listed in Table 2. IR, electronic spectra, magnetic moment, molar conductance measurements and thermogravimetric analysis, it can be concluded that Schiff base acts as a mononegative charge and type of bidentate (NO) ligand in all complexes.

¹H NMR spectrum of Ligand (L): The ¹H NMR spectrum of the ligand was recorded in DMSO-d₆ as a solvent. The synthesized ligand has been shown multiple signals at $\delta(6.8-7.8 \text{ ppm})$ which refer to aromatic ring. The peak pointed out at $\delta(8.7 \text{ ppm})$ pointing to the proton of azomethine group (CH = N) (Mohammed, 2011). The

peak that appeared at $\delta(9.7 \text{ ppm})$ was indicated presence of the proton of SH group (Mahajan *et al.*, 2007). Apparent peaks at $\delta(4.3-4.6 \text{ ppm})$ were referred to chemical shift of S-CH₂ proton (Alwan, 2012). The weak peak appeared at $\delta(3 \text{ ppm})$ refers to a presence of H₂O (Ammar and Alaghaz, 2013; Parkash *et al.*, 2008). The peak appeared at $\delta(2.5 \text{ ppm})$ was assigned to CH₃ (Adedibu and Joshua, 2011). The ¹H NMR spectrum of L was shown in Fig. 8.

Electronic spectra: The electronic spectra of the ligand and its metal complexes have been established in ethanol at room temperature. The ligand showed up absorption bands at 243, 280 nm as signs to ($\pi \rightarrow \pi^*$) and ($n \rightarrow \pi^*$) transitions, respectively. These peaks were changed to 241, 281 nm in the cobalt complex, 244 nm in the nickel complex and 243, 295 nm in the cadmium complex while the band which appeared at 313 nm assigns to ($n \rightarrow \pi^*$) transition in the nickel

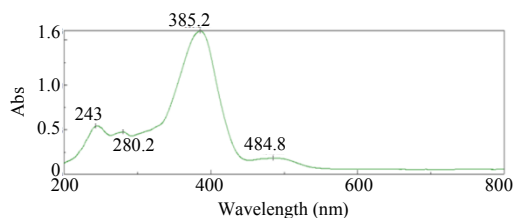


Fig. 9: The electronic spectrum of the Ligand (L)

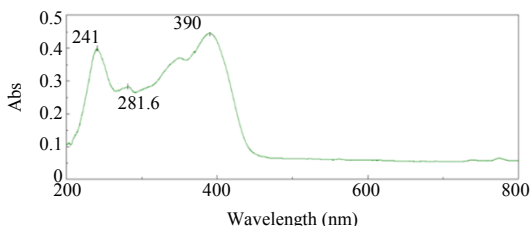


Fig. 10: The electronic spectrum of the cobalt complex

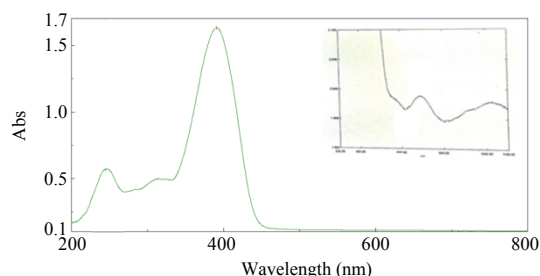


Fig. 11: The electronic spectrum of the nickel complex

complex (Foust and Ford, 1972). The peaks appeared at 385, 484.5 nm were referred to Intra Ligand Transitions (ILCT) which have been shifted to 390 nm in the cobalt and nickel and 387 nm in the cadmium complexes. The cobalt complex was shown up two peaks at (740 and 768 nm) assigned to d-d transitions $A_{2g} \rightarrow T_{2g}(P)$ and $A_{2g} \rightarrow T_{1g}(F)$ while nickel complex was showed three peaks at (692, 941 and 1029 nm) assigned to d-d transitions [$T_{1g} \rightarrow T_{2g}(F)$, $T_{1g} \rightarrow T_{1g}(P)$ and $T_{1g} \rightarrow A_{2g}(F)$] while cadmium complex showed no d-d transitions because the d-orbitals are filled (Foust and Ford, 1972; Bilge *et al.*, 2009). The ratio of Metal to Ligand (M: L) as (1:2) and the geometry of complexes was tetrahedral.

The electronic spectra of the ligand and its complexes explained in Fig. 9-12 and the spectral data are scheduled in Table 3 and the suggested structure of metal ion (Co (II), Ni (II) and Cd (II) complexes have a tetrahedral geometry as shown in Fig. 13.

Application/corrosion inhibitors: Ligand (L) and its synthesized complexes were used as an inhibitors for the

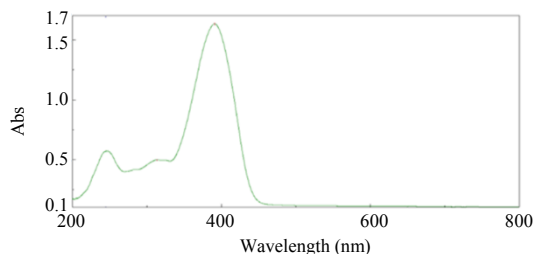


Fig. 12: The electronic spectrum of the cadmium complex

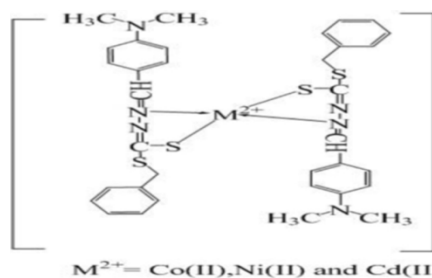


Fig. 13: Suggested structure of metal ion (Co (II), Ni (II) and Cd (II) complexes

Table 3: The electronic spectral data of ligand and its metal complexes

Symbol	Wave length λ (nm)	Wave No. (cm^{-1})	Assignment
L	243	41152	$\pi \rightarrow \pi^*$
	280	35714	$n \rightarrow \pi^*$
	385	25974	ILCT
	484	20661	ILCT
Co(L) ₂	241	41493	$\pi \rightarrow \pi^*$
	281	35587	$n \rightarrow \pi^*$
	390	25641	CT (L→M)
	740	13513	$A_{2g} \rightarrow T_{2g}(P)$
Ni(L) ₂	768	13020	$A_{2g} \rightarrow T_{1g}(F)$
	244	41152	$\pi \rightarrow \pi^*$
	313	31948	$n \rightarrow \pi^*$
	390	25641	CT (L→M)
Cd(L) ₂	692	14450	$T_{1g} \rightarrow T_{2g}(F)$
	941	10615	$T_{1g} \rightarrow T_{2g}(P)$
	1029	9718	$T_{1g} \rightarrow A_{2g}(F)$
	243	41152	$\pi \rightarrow \pi^*$
	295	33898	$n \rightarrow \pi^*$
	387	25839	CT (M-L)

corrosion of Stainless Steel (S.S) in seawater (3.5% NaCl soln.). The corrosion study was achieved using potentiostate type M/alb 200 (Germany Sartorius Model). All corrosion parameters were calculated from Tafel extrapolation curves. Both anodic and cathodic polarization curves were shifted (Tafel slopes ba, bc) as shown in Fig. 14 and Table 4. It can be seen that in presence of L and the three complexes, the corrosion current densities (i_{corr}) are reduced and the Inhibition efficiency (I%) can be calculated using the following equation (Khalil *et al.*, 2012, 2018):

Table 4: Corrosion parameters for CS with different inhibition complexes in 3.5% NaCl at 25°C temperature

Compound	E _{corr} (mV)	I _{corr} (μAcm ⁻²)	-bc (mV/Dec)	ba (mV/Dec)	WL (gm ⁻² d)	PL (mm y ⁻¹)	IE (%)
Blank	276.07	101.23	91.90	113.90	8.39	1.130	
L	288.90	58.85	97.30	96.20	4.74	0.640	41.865
Co(L) ₂ (N1)	315.10	48.61	86.30	112.30	3.91	0.529	51.981
Ni(L) ₂ (N2)	303.80	57.26	91.10	119.50	4.61	0.623	43.436
Cd(L) ₂ (N3)	303.70	62.73	88.80	100.10	5.05	0.682	38.500

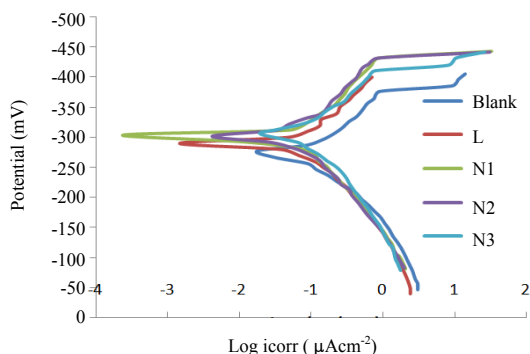


Fig. 14: The corrosion inhibition of Ligand (L) and its complexes(N1-N3)

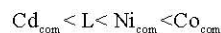
$$I\% = [I_{corr}^0 - I_{corr} / I_{corr}^0] * 100$$

Where:

I_{corr}^0 : The corrosion current density in uninhibited solutions

I_{corr} : The corrosion current density in inhibited sea water solutions (in presence of L or each of the three synthesized complexes)

bc: cathodic Tafel slope, ba: anodic Tafel slope, WL: Weight Loss, PL: Penetration Loss, IE%: Inhibition Efficiency. The inhibition efficiency are ranging between 38.5-51.98 at 298 K, the highest I% occur between cobalt complex and the lowest value obtained by cadmium complex and the inhibition percentage efficiencies sequences are as following:



The corrosion inhibitors of ligand and its complexes possess significant to moderate activity as shown in Fig. 14 and the results of corrosion obtained (corrosion parameters of stainless steel alloy at 298 K) were listed in Table 4.

CONCLUSION

In this study, new ligand complexes of Co(II), Ni(II) and Cd(II) have been synthesized. The spectroscopic studies of all complexes point out that the ligand is a bidentate and coordinated with metal ion through sulphur atom and nitrogen of imine group. Thus, from the results presented the suggested structure of metal

complexes have a tetrahedral geometry. The compounds prepared have been shown inhibition against seawater (3.5% NaCl soln.).

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