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COMPLEXATION BEHAVIOR OF NEWLY SYNTHESIZED SCHIFF BASE N-(4-METHOXYPHENYL-4\-CYANOBENZILIDENE) AS CORROSION INHIBITION

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Abstract

Complexes of Nickel (II), Copper (II), Zinc (II) and Cadmium (II) ions with Schiff base derived from 4-cyanobenzaldehyde and 4-methoxyaniline were synthesized and investigated by several techniques using elemental analysis, FT-IR, UV-visible, ^{1}HNMR, ESR, Magnetic susceptibility. The results revealed that, the synthesized ligand coordinate with the central metal ions through the azomethine nitrogen. Investigation of the stereochemistry of these complexes suggests the existence of tetrahedral geometries for Ni (II), Zn (II) and Cd (II) complexes where's pesedotetrahedral geometries as expected for Cu (II) complex. The effect of the Schiff base ligand (1) and its corresponding Zn complex on the corrosion behaviors of C-steel in 0.5 M HCl acid solution were studied by weight loss measurements. The inhibitive effect of ligand (1) and its stable tetrahedral Zn complex is argued to their adsorption over the C-steel surface. Key word; Transition metal complexes, Schiff base, Corrosion inhibitor

1. Introduction

Schiff base ligands have been extensively studied in coordination chemistry mainly due to their facile syntheses, easily steric, electronic properties and good solubility in common solvents [1]. Transition metal complexes with nitrogen donor Schiff bases are of particular interest [2] because of their ability to possess unusual configurations, be structurally labile and their sensitivity to molecular environments [3]. Schiff bases can accommodate different metal centers involving various coordination modes allowing successful synthesis of homo- and heterometallic complexes with varied stereochemistry [4]. This feature is employed for modeling active sites in biological systems [5]. Additionally, they have wide applications in fields such as, antibacterial, antiviral, antifungal agents [6], homogeneous or heterogeneous catalysis [7] and magnetism [8], anticancer [9]; corrosion inhibition [10]. In view of this, we described the synthesis and characterization of Schiff base and their Nickel (II), Copper (II), Zinc (II) and Cadmium (II) complexes. Corrosion inhibition of some metal complexes including zinc was evaluated using mild steel [11]. It is shown that a complex protective film is formed in presence of zinc.

2. Experimental

2.1. Physical techniques

The melting point measured by the electrothermal melting point, Griffine Gearge made in UK; Elemental analyses for carbon, hydrogen and nitrogen were determined by Perkin-Elmer 2400 at Cairo University; FT-IR Mattson 5000 FTIR spectrometer using potassium bromide at Mansoura University; spectral UV-Visible range (200-900) nm using Perkin Elmer Lambda 35 UV/Vis spectrometer at Al-Azhar University; ^{1}HNMR spectra in D_6-DMSO with TMS as internal standard were obtained from a Jeol-FX-90Q Fourier NMR spectrometer at Cairo University; Mass Spectra were performed by a Shimadzu-GC-MS-QP1000 EX using the direct inlet system Al-Azhar University (RCMB); Metal contents (% wt) were estimated by Atomic Spectrometry (UNICAM-SOLAAR) at Cairo Oil Refining Company (CORC); Magnetic Susceptibility (χ_g) measured using a magnetic susceptibility balance of models Johnson Metthey and Sherwood at Mansoura University, the ESR spectra was carried out on Bruker-EMX-(X-bands) spectrometer working in the expand 9.7 GHZ with 100 KHZ frequency and microwave power 1.008 mW and modulation and amplitude 4GAUSS at Egyptian Atomic Energy Authority (EAEA) and the chloride content were estimate using (Volhard’s methods) [12].

2.2. Materials

The chemicals used in present work, 4-cyanobenzaldehyde, 4-methoxyaniline, nickel (II) Chloride hexahydrate, Cuppric (II) acetate monohydrate, Zinc (II) Chloride, Cadmium (II) Chloride monohydrate, nitric acid, Hydrochloric acid, ethanol, methanol, diethyl ether, petroleum ether (60-80), acetone and chloroform were BDH and Merck chemicals. Corrosion tests were performed on a C-steel of the following percentage compositions are (wt. %): 0.07 (C), 0.39 (Mn), 0.022 (P), 0.0001 (S), 0.01 (Si), 0.03 (Al), 0.04 (Cr), 0.0009 (Mo), 0.04(Ni), 0.016 (Co), 0.073(Cu), 0.0006 (Nb), 0.003 (Ti), 0.017 (V), 0.013 (W), 0.003 (Pb) and reminder iron. Solution of 0.5M HCl was prepared by dilution of analytical grade 37% HCl with bidstilled water, which used as corrosive media in this study. The C-steel specimens used in weight loss measurements were cut into 1.6 cm^2 coupons. The concentration range of employed range of employed inhibition was (10, 50, 100, 200) ppm in 0.5M HCl.

2.3. Preparation of ligand

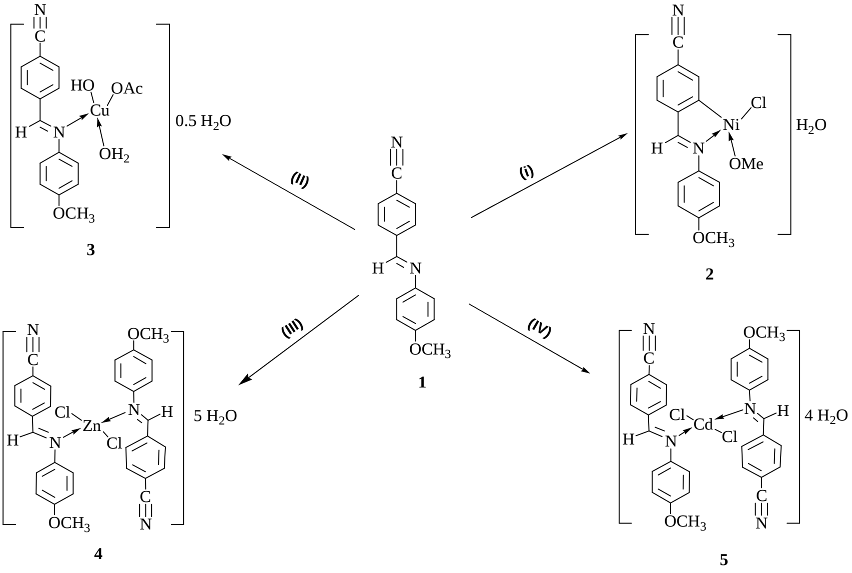
The Schiff base ligand and its complexes **(scheme 1)** used under study were prepared as 4-methoxy aniline 5.06 gm (0.041 mol) dissolved in absolute ethanol ($30cm^3$) was mixed with the 4-cyanobenzaldehyde, 5.25 gm (0.041 mol) in ($20cm^3$) absolute ethanol about 10 min. Then mixture was stirred for 1 hour under reflux. The crystals of the desired ligand was collected by filtration through a Buchner funnel and recrystallized from Petroleum sprite (60–80) and finally dried at room temperature.

2.4. Preparation of solid complexes

All the complexes were prepared by mixing equimolar amounts of ligand and with the metal (II) acetate [$M=Cu^{II}$] and chlorides [$M=Ni^{II}$, Zn^{II}, Cd^{II}] in 50 cm^3 methanol. The mixture was heated under reflux for 7 hrs. The resulting solid complexes (60–90 % yield) were filtered out, washed several times with water, hot methanol followed by ether until the filtrate becomes colorless and finally, the complexes were washed with diethyl ether and dried in vacuum desiccators over anhydrous $CaCl_2$.

2.5. Weight loss method

In this study C-steel sheet of 2.0 cm ×2.0 cm × 0.4 cm with an exposed total area 1.6 cm^2, in triplicate were mechanically polished successively with different grades emery papers, rinsed with bidistilled water, wash by acetone and finally dried between filter paper. The immersion time at 25 ºC for 24 hrs, specimens were immersed in 50 ml 0.5M HCl solution containing various concentrations of the studied inhibitors, the mass of the specimens before and after immersion was determined using an analytical balance of 0.01 mg accuracy. Experiments were carried out in triplicate and average of the triplicate values was used.



Reagent: (I) $NiCl_2.6H_2O/CH_3OH$; (II) $Cu(OAc)_2.H_2O/CH_3OH$; (III) $ZnCl_2/CH_3OH$; (IV) $CdCl_2.\ H_2O/CH_3OH$

Scheme 1: The proposed chemical structures of the complexes

Table 1: Analytical and physical data of ligand and its complexes.

Compound	Molecular Formula	Molecular Weight M^+ Calcd/ (Found)	M.P. °C	Color	Elemental Analysis Calculated / (Found)				
					C %	H %	N %	Cl %	M %
Ligand	$C_{15}H_{12}N_2O$	236.27	119	Green	76.25 (76.27)	5.12 (5.22)	11.86 (11.82)	-	-
Ni^{II}	$C_{16}H_{16}ClN_2NiO_3$	377.02	175	Greenish	50.78 (51.01)	4.26 (5.07)	7.40 (7.43)	9.30	15.51 (15.60)
Cu^{II}	$C_{17}H_{19}CuN_2O_{5.5}$	402.89	190	Dark Brown	50.68 (50.70)	4.75 (4.70)	6.95 (6.90)	-	15.77 (15.65)
Zn^{II}	$C_{30}H_{34}Cl_2N_4O_7Zn$	698.11	170	Yellow	51.55 (51.60)	4.90 (3.70)	8.02 (8.00)	10.10	9.36 (9.45)
Cd^{II}	$C_{30}H_{26}CdCl_2N_4O_4$	727.92	240	Dark Green	49.50 (49.89)	4.43 (4.24)	7.70 (7.57)	9.70	15.44 (15.30)

Table 2: IR spectral data of ligand and its complexes

Comp.	ν_{OH} (H_2O)	ν_{CH^-} aromatic	ν_{CH^-} aliphatic	$\nu_{C\equiv N}$	$\nu_{C=N}$	ν_{OAc}		$\nu_{C=C}$	ν_{Ph-N}	ν_{M-C}	ν_{M-N}	ν_{M-X}
						ν_{as}	ν_s					
Ligand	-	2958	2838	2225	1619	-	-	1502, 1461, 1407	1030	-	-	-
Ni^{II}	3461	2927	2840	2221	1578	-	-	1504, 1461, 1442, 1405	1027	557	473	418
Cu^{II}	3444	2933	2838	2221	1612	1574	1363	1502, 1461, 1442, 1407	1027	-	472	-
Zn^{II}	-	2958	2836	2219	1581	-	-	1502, 1459, 1442, 1407	1029	-	470	414
Cd^{II}	3415	2923	2850	2222	1611	-	-	1485, 1446, 1409	1029	-	468	410

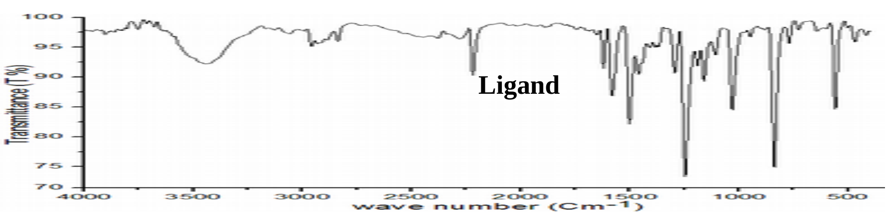
Table 3: Electronic spectra ^{1}HNMR and magnetic moment data of all compounds:

Formula	λmax (nm) Assignment	^{1}HNMR δ ppm	μ_{eff} (B.M)	Structure
Ligand	(260-289), (318-348) (π-π*) C=N, Ar. ring	8.75 (C=N, s, H), 3.32-3.79 ($-OCH_3$) 6.98-7.94 (Ar, m, 8H)	-	-
[Ni-(L)-OMe.Cl] H_2O	445($^3T_1 \rightarrow {}^1T_{2(F)}$)	-	3.47	Tetrahedral
[Cu-(L)-OAc)(OH)] $0.5H_2O$	493 (L→M)CT	-	1.74	Pseudo-Tetrahedral
[Zn-$(L)_2$- 2Cl] 5 H_2O	392 (L→M)	8.09 (C=N, s, 1H), 3.16-3.78 ($-OCH_3$, s, 3H) 6.99-7.39 (Ar, m, 8H)	D	Tetrahedral
[Cd-$(L)_2$-2Cl] $4H_2O$	323 (L→M)	8.09 (C=N, s, 1H), 3.41-3.78 ($-OCH_3$, s, 3H) 6.99-7.98 (Ar, m, 8H)	D	Tetrahedral

s: singlet, m : multiplet, Ar: Aromatic ring, D: Diamagnetic

3. Results and discussion

All the analytical, physical and spectroscopic data of the Schiff bases **(scheme 1)** and their isolated metal complexes are given in **Tables (1-3)**. The complexes are air stable for a long time and are soluble in DMF and DMSO.



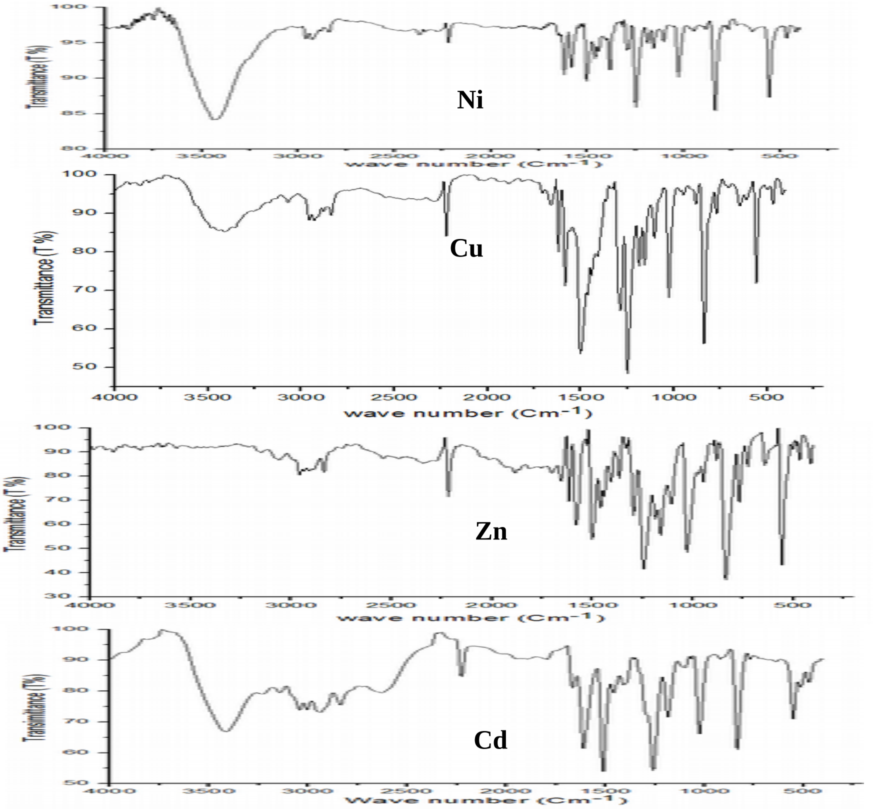


Fig. 1: FT-IR Spectra of Ligand, Ni, Cu, Zn and Cd

3.1. FT-IR Spectroscopy

The positions of the significant FT-IR bands of free ligand and its metal complexes are summarized in Table 2. The FT-IR spectra show in **(Fig. 1)** of free ligand show a strong band at 1619 cm^{-1} assignable to ν(C=N) of the azomethine [13]. The observation of this band confirms the formation of the azomethine linkage. The infrared spectrum of the free Schiff base shows strong band at 2225 cm^{-1} are assigned to C≡N [14].

3.1.1. Ni^{II} complex

The FT-IR spectral data for this complex show in **(Fig. 1)** indicate that the ligand coordinating to metal ions via the azomethine with displacement of the protons from the latter groups k a five member ring around the metal ion. For Ni^{II} shows the downfield shift of the C=N 1619 cm^{-1} in the free ligand to 1583 cm^{-1} assignable to the coordinated imino group. A new band observed at 557 and 473 cm^{-1} which may be due to **$\nu_{(Ni\text{-}C)}$** and **$\nu_{(Ni\text{-}N)}$** that attributed to **$\nu_{(Ni\text{-}N)}$** vibrations supporting the participation of the nitrogen atom of the azomethine group of the ligand in the complexation with metal ions [15]. The presence band at 2221 cm^{-1} assignable to ν(C≡N) and peaks at (3650-3200) cm^{-1} are attributable to coordinated water molecules [16].

3.1.2. Cu^{II}, Zn^{II} and Cd^{II}complexes

For Cu^{II}, Zn^{II} and Cd^{II} complexes show in **(Fig. 1)** the assignment of the important bands was made and recorded in **(Table 2).** In order to give conclusive idea about the structure of metal complexes, the main IR bands were compared with those of the free ligand, shows the downfield shift of the C=N of a Cu^{II}, Zn^{II} and Cd^{II}complexes (1612, 1581 and 1611) cm^{-1} as respectively, assignable to the coordinated imino group. A new band observed at 472 and 470, 468 cm^{-1} which may be due to $\nu_{Cu\text{-}N}$, $\nu_{Zn\text{-}N}$ and $\nu_{Cd\text{-}N}$ [17]. The presence band at 2221 cm^{-1} assignable to ν(C≡N). All complexes showed broadband around (3461-3415) cm^{-1} due to ν(OH) from water molecules and this band is absent in the ligand.

3.2. ^{1}HNMR Spectra

The ^{1}*HNMR* spectra of the ligand and complexes (*Zn^{II} and Cd^{II}*) [18-20] were recorded to confirm the binding of the Schiff base to the metal ions. The spectra of the complexes showed a singlet in the region δ (7.94-8.09) ppm, which has been assigned to azomethine proton (HC=N) **(Table 3)**. The position of azomethine signal in the complexes is downfield in comparison with the free ligands, suggesting deshielding of the azomethine proton due to its coordination to metal ions through the azomethine nitrogen. In the region δ (6.98-7.94) ppm were assigned the chemical shifts for hydrogen of symmetrical aromatic ring of ligand. Protons on the methoxy group ($-OCH_3$) resonance in the region of δ (3.16-3.79) ppm as singlet pattern.

3.3. Electronic spectra and magnetic studies

The assignments of the observed electronic absorption bands of the Schiff bases and its metal complexes [21–23] as well as the geometry and magnetic data of the formed complexes are shown in **(Table 3)**. In fact, the appearance of several bands in the electronic spectra of free ligand (1), exhibit bands in the range of 260-289 and 318-348 nm assigned to the π-π^* and n-π^* transitions respectively. The azomethine group and is shifted to longer wavelength on coordination through azomethine nitrogen in all the complexes [24]. The Ni^{II}-Schiff base complex spectrum displays absorption band at 445 nm owing $^3T_1 \rightarrow {}^1T_{2(F)}$ transition and suggesting a tetrahedral geometry, The tetrahedral geometry is confirm by measured magnetic moment (μ_{eff}=3.47 B.M) which is harmony with the reported value, μ_{eff}=3.47 B.M, for tetrahedral complex Ni. The electronic spectrum of Cu^{II}-Schiff base complex exhibits an L→M(C.T) charge transfer band at 493 nm. This complex presumably has a pesedotetrahedral configuration, and geometry is confirm by measured magnetic moment (μ_{eff}=1.74 B.M). The electronic spectrum of Zn^{II}-Schiff base complex exhibits an M→L charge transfer band at 392 nm. This complex presumably has a tetrahedral configuration. This complex presumably has a pesedotetrahedral configuration. The electronic spectrum of Cd^{II}-Schiff base complex exhibits an M→L charge transfer band at 323 nm. This complex presumably has a tetrahedral configuration.

3.5. ESR Spectra

The electron spin resonance (ESR) also frequently called electron paramagnetic resonance (EPR) and electron magnetic resonance is a branch of absorption spectroscopy is which radiation of microwave frequency is absorbed by paramagnetic samples.

The Copper complexes give poorly resolved ESR spectra at room temperature, even though it is a d^{10} system. Very few d^{10} systems that exhibit room temperature ESR spectra are known in the literature Copper (II) (d^{10}) ion being a non-Kramer’s ion, ESR spectra is observable, generally at low temperatures [25]. However, there are very few reports in the literature about the study of this d^{10} ion at room temperature. Most of the reported work concerns the study of this ion at 77 K or 4 K. There are a few extra resonance lines, which might have been arisen from forbidden transitions. Generally, the ESR spectra of a powdered Cu^{II} **(Fig. 2)** exhibit a very broad signal associated to Cu^{II} with a g//-factor of about 2.088. In this case, the electronic fine structure is large compared to the electron Zeeman interaction leading to a single line. The g factor deviates strongly from complex (**2)** because of strong spin-orbit coupling. The ESR spectral parameters of Copper (II) in complexes and reveal that the complexes having a peseduotetrahedral geometry around Cu^{II} ion. These data are well consistent with other reported values.

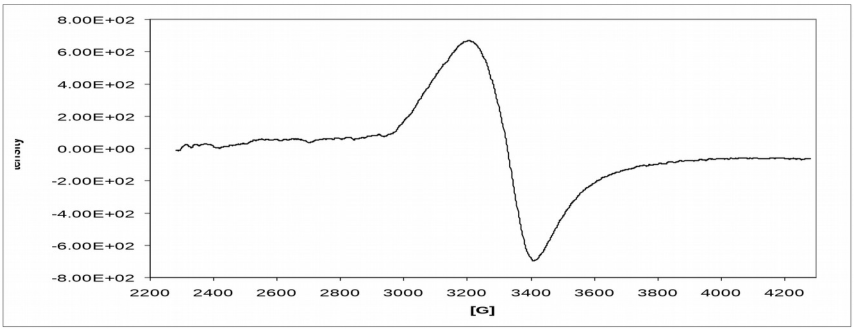


Fig. 2: ESR Spectrum of complex Cu^{II}

3.6. Mass Spectra

Mass spectrometry show in **Figs. (3-5)** has been successfully used to investigate molecular species in solution [26, 27]. The pattern of mass spectrum gives an impression of the successive fragmentation of the target compound with the series of peaks corresponding to the various fragments. Their intensity gives an idea of stability of fragments. The recorded mass spectra of the ligand and their Cu^{II} and Zn^{II} complexes and molecular ion peaks have been used to confirm the proposed formula. The mass spectrum of ligand showing peaks at 236 (82%), 221 (100%), 192 (32%), 76 (37%), 50 (25%) respectively, corresponding to the molecular ion $[M]^+$. The ligands finally fragment to 50 (25%) that correspond to $[C_4H_3]^+$. The mass spectra of complexes Cu^{II} and Zn^{II} have a common base peak 221 (100%), which shows a peak of ligand. In complex Cu^{II} the fragmentation pattern shows peaks at 236 (71%) is $[L]^+$ and 299 (0.14%) shows $[L+Cu^{II}]^+$. In complex Zn^{II}, the fragmentation peak at 372 (0.14%) shows $[L+Zn^{II}+(Cl)_2]^+$ and 236 (82%) shows $[L+Zn^{II}]^+$.

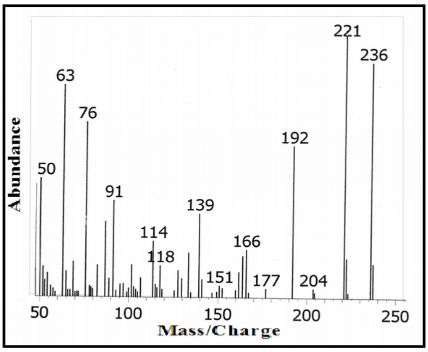


Fig. 3: Mass Spectrum of Ligand

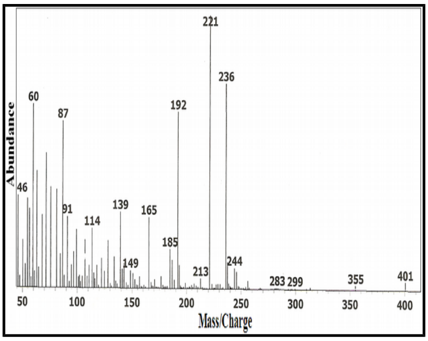


Fig. 4: Mass Spectrum of Cu complexes

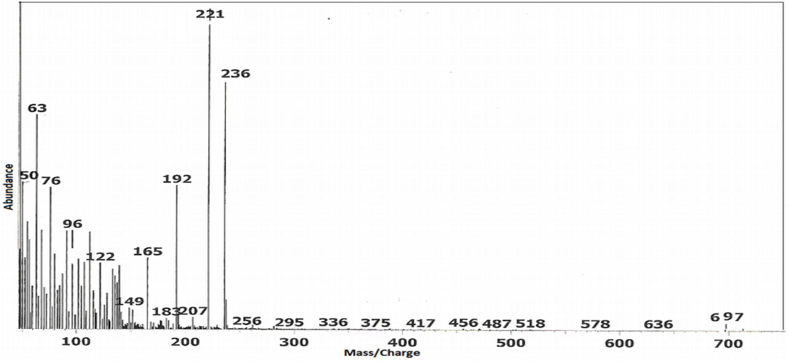


Fig. 5: Mass Spectrum of Zn complexes

3.7. Corrosion inhibition

The inhibitive efficiency of ligand (1) and Zn (4) complex show in **(Fig. 6)** on the dissolution of C-steel in 0.5 M HCl are investigated **(Tables 4)**. The degree of surface coverage (θ), the percentage inhibition efficiency (IE %) and the corrosion rate (C.R.) were calculated as follows [28]:

The value of corrosion rate is calculated from the following equation

$$C.R(mpy) = \frac{kW}{DAT} \qquad (1)$$

Where k is the corrosion constant= 3445.15, W is the loss of weight after corrosion (mg), A is the total area of the coupon (cm^2), T is the corrosion time (hr) and D is the coupon density = 7.87 (gm/cm^3).

The degree of surface coverage (θ) and the inhibition efficiency, IE %, are calculated using the equations

Surface coverage $\theta = \frac{w^{o} - w}{w^{o}}$ (2)

Where; w^0 and w are the weight loss in the absence and presence of inhibitor, respectively.

Inhibition Efficiency IE % = θ x 100 (3)

The inhibition efficiency of additive compound depends on many factors, which include the number of adsorption sites and their charge density, molecular size, heat of hydrogenation, mode of interaction with the metal surface, and formation of metallic complexes. The weight loss data were determined and listed in Table 4. Careful inspection of these results showed that, by increasing the inhibitor concentration, both (θ) and (IE %) were increased while (CR) was reduced. So, the dissolution of C-steel in the presence of the investigated inhibitor can be interpreted on the basis of interface inhibition mode, the inhibitor acts effectively at the metal solution interface [29]. Weight loss data determination after 24 hrs. Yielded convincing evidence for the application of ligand (1) as an active corrosion inhibitor Than Zn complex (4). The maximum efficiencies at higher ligand (1), Zn complex (4) concentration values are 81, 74, respectively. These results indicate a reasonable corrosion inhibition for specimens.

Table (4): Corrosion parameters for C-Steel in 0.5 M HCl in the absence and presence of different concentrations of Ligand (1) and Zn complexes at 25ºC.

Inhibitor	Conc. (M)	Weight Loss mg cm^{-2}	Rate of corrosion mpy	θ	Inhibition efficiency η%
Ligand (1)	Blank	0.200	-	-	-
	10	0.098	0.021	0.40	40
	50	0.085	0.018	0.58	80
	100	0.039	0.008	0.76	76
Zn	200	0.033	0.007	0.80	80
	10	0.011	0.084	0.20	20
	50	0.010	0.078	0.26	26
	100	0.005	0.036	0.65	65
	200	0.003	0.027	0.74	74

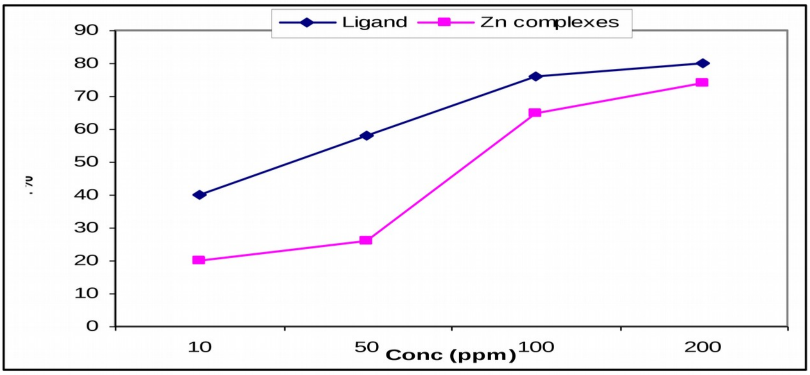


Fig. 6: Variation of inhibition efficiency with different concentration ligand and Zn-complex on C-steel surface in 0.5 M HCl at different concentrations of at 25 ºC.

4. Conclusion

From previous data; elemental analysis, infrared, electronic spectroscopy, ^{1}HNMR, ESR and mass spectrometer of metal ion complexes, the following can be drawn concerning the ligating property of the Schiff base as well as the stereochemistry of their corresponding complexes. The infrared spectral data reveal one mode of complexation through nitrogen atom of azomethine group. The electronic spectra exhibit two kinds of geometries; Tetrahedral for Ni^{II}, Zn^{II}, Cd^{II} and Psedotetrahedral for Cu^{II} complex. The suggested structural formulas of the metal-Schiff base complexes under investigation are given **(Scheme 1)**. Studied Schiff base and Zn complex are excellent inhibitors and act as the mixed type inhibitor for C-Steel corrosion in the 0.5 M HCl acid solution specially in high concentrated, corrosion inhibition efficiencies are in the ligand more than Zn complex. Inhibition efficiencies of these compounds increase by an increase in the inhibitor concentration of environment temperature.

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