

ORIGINAL ARTICLE

Preparation & Characterization of Complexes Containing Beta Lactam & Study of the Effect of Corrosion Inhibition Using Iron Resistant Stainless Steel

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Metal (II) complexes of Co, Ni, Cu and Zn with Amoxicillin derivative (L) were synthesized and identified by elemental analysis CHNS Uv-Vis, FTIR, TGA, metal analysis AA, magnetic susceptibility and conduct metric measurement. by analysis the ligand behaves as a bidentate. All complexes that have nickel, copper, cobalt and zinc ions were proposed as octahedral geometry shape. Protection study of the corrosion behavior of carbon steel surface was conducted with several of the zinc derivative concentrations. Experimentally, the inhibition efficiencies (%IE) in the saline solution 3.5% NaCl was examined using measurements of potentiometric polarization. The outcomes revealed that the %IE of carbon steel corrosion by amoxicillin Derivatives was 72.87%.

Keywords: corrosion resistance, elemental analysis CHNS, Uv-Vis and FTIR spectroscopies, Amoxicillin derivative

INTRODUCTION

"The class of compounds, so-called imines, are often referred to as Schiff bases, named after the German chemist Hugo Schiff. Schiff bases contain the azomethine group ($R_1-C=N-R_2$) and are typically formed by the condensation of a primary amine with an active carbonyl compound"¹. At coordination chemistry, Schiff bases with high chelating tendency across various range of transition metals ions, have gained much attention². Particularly, Schiff base metal chelates with multi-donors are potent π ligands that lead to form stable complexes. For example, complexes were appeared important properties as different from chemical^{3,4} or physical⁵. They have scale- range of pharmacological activities^{6,7}. Mounting evidences indicate that the antimicrobial and anticancer activities are enhanced upon chelation. Over the past decades, transition metal ion complexes and Schiff bases there has been a great interest because they have multi advantages in several fields. Schiff bases are famed as azomethine group that produced through a simple condensation reaction of amine with aldehyde compounds⁸. Such bases are interesting compounds that successfully coordinating with variable metal ions through different mode of bonding^{7,10}. These complexes have a pivotal role in biological, electrochemical and catalytic fields.

Transition metals can exhibit a wide variety of coordination properties and reactivity, which can be used to form complex with organic drugs as ligands. Transition metal complexes exhibit unique and interesting properties such as changing oxidation states and the ability to form specific interactions with other biomolecules^{11,12}. Thus, the drugs that contain antibiotics and metals could prevent resistance development of microbes, enable novel action mechanisms and enhance the antimicrobial activity of these drugs.^{13,14,15} For this reason we aimed to produce a new class of antibiotic contains amoxicillin as amino antibiotic and cinnamaldehyde as the aldehyde this type of antibiotic can be classified as Schiff base. Amoxicillin is one of the penicillin antibiotics that is utilized to treat bacterial infections including chest infections and dental abscesses. It can also be employed together with other medical drugs to treat stomach ulcers.^{16,17}

Cinnamaldehyde ($C_6H_5CH=CHCHO$) is an organic compound that exist normally at cinnamon genus bark and its responsible for cinnamon flavor and odor¹⁸. It is a phenylpropanoid that is naturally synthesized by the shikimate pathway.¹⁹ Corrosion inhibitor is a chemical substance that interacts with the surface or environment of a metal to which is exposed and acts to protect surface of the metal from corrosion²⁰. The inorganic mixes have chromate, nitrites and mixes have hetro particles (O, P, S, N), π bonds were best and productive²¹. Mixes containing both nitrogen and chloro molecules may give fantastic hindrance, contrasted and mixes containing just nitrogen or chloro particle²². Heterocyclic mixes, for example, anti-

infection (drug drugs) may give superb restraint. These particles relies principally upon certain actual properties of the inhibitor particle, for example, utilitarian gatherings, steric factors, electron thickness at the contributor iota and electronic structure of the atoms^{23,24}. A couple of scientists have been accounted for the utilization of antibacterial medications as consumption inhibitors because of that of essence of oxygen, nitrogen and sulfur as well as in their systems dynamic focuses, high solvency in water, high atomic size, non-harmful —environmentally friendly| erosion inhibitors, significant in organic responses and medications that might be effortlessly created and sanitized^{25,26}.

Corrosion Potential (E_{corr}) Measurements: The electrochemical system consists of a potentiostate, three electrodes, with a controlled computer and standard corrosion cell. The thermostat was among the most widely, using to control the temperature of (3.5% NaCl) which is 30 degrees Celsius and flows by the external vessel the corrosion cell, as well as the three electrodes, are depicted in Figure 1. are:

1. The first is reference electrodes based on its potential consist of AgCl, Ag, KCl, and the outer solution filled with the prepared Saline solution (3.5% NaCl). The reference electrode, a lugging tube placed at the distance 2mm from working electrode.
2. The second electrode is auxiliary consist of high purity platinum rod with 0.6 mm in diameter and 10 cm in length.
3. The third is working electrode (Carbon steel) which was mounted in the working electrode load with 1 cm^2 surface area the opening uncovered to the acidic solution.



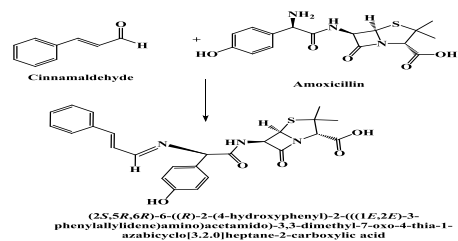
Figure 1: (a) The three electrodes and the corrosion cell

MATERIALS AND METHOD

The next chemical materials were obtainable trading products, amoxicillin $C_{16}H_{19}N_3O_5S$, metal salts $CoCl_2 \cdot 6H_2O$, CH_3COOH ,

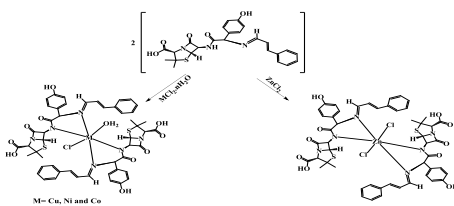
NiCl₂·6H₂O, ZnCl₂ and (CuCl₂·2H₂O), gained from F-897ewluka, sigma Aldrich. FTIR was detected in the range of 4000-200 cm⁻¹ with KBr disk and 4000-200 cm⁻¹ with CsI disk on a Shimadzu-3800 Spectro-meter. The electronic spectral data were detected by using Shimadzu160 Spectro-photometer. Melting point apparatus of Gallencamp MF-B-600.01 was used. Molar Conductivity was used to measure the conductivity of the complexes at room temperature in freshly prepared 10-3 M in absolute ethanol using coring conductivity meter 220. TGA investigations of all previous forms were done on Perkin-Elmer Pyris Diamond TGA. spectra were registered by employing Bruker 500-MHz Spectro-meter

Synthesis of Ligand L: This ligand was synthesized by adding 0.365g, 0.0009 mol from amoxicillin with 5 ml ethanol with continuous stirring, then adding 0.132g, 0.0009 mol cinnamaldehyde which dissolved in 5 ml ethanol, about 3 drops of glacial acetic acid were added onto the mixture, then the solution left in order to perform refluxing for about 3 hrs. A yellow solution is produced which kept at room temperature to be dried and gained a yellow precipitate.



Scheme 1: synthesis of ligand L1

Synthesis of Cu²⁺, Co²⁺, Ni²⁺ And Zn²⁺ Complexes: The complexes of this ligand (L1) were synthesized by the following approach, reaction of 0.19 g, 0.0002 mol from ligand dissolved in 10 ml ethyl alcohol with each of the next metal salts 0.047 g CuCl₂·2H₂O, 0.04 g CoCl₂·6H₂O, 0.047 g NiCl₂·6H₂O and 0.027 g ZnCl₂, were dissolved in 10 ml ethanol. The reactions were carried out into round flask with 25 ml as volume in 2:1 ligand: metal ratio. Then the solution of ligand was mixed with each metal salt solution, mixtures were left for 3 hrs. refluxing then kept at room temperature till being cooled, solvent evaporates and precipitation accomplished.



Scheme 2: Synthesis of complexes

RESULT AND DISCUSSION

All physical properties are ill suited in Table (1.). The complexes are characterized with stability at room temperature with various melting points and different colors.

Table 1: element micro analysis and some physical properties of ligand and complexes

Compounds	Elemental analysis Found, (Cal.) %				M.P °C	Color	% Yield	µscm-1
	C	H	N	M				
C ₂₂ H ₂₅ N ₃ O ₅ S(L)	66.32 (65.66)	5.22 (5.12)	8.19 (7.28)	---	199	Dark Yalow	80%	----
CoC ₂₀ H ₃₂ N ₄ O ₁₀ SCl ₂ (Co-L)	51.76 (53.54)	3.97 (4.29)	6.60 (6.13)	8.99 (8.69)	179	Brown	77%	17.24
Ni C ₂₀ H ₃₂ N ₄ O ₁₀ SCl ₂ (Ni-L)	50.03 (50.84)	3.83 (4.66)	6.11 (5.93)	9.29 (8.28)	207	Light Brown	82%	19.49
Cu C ₂₀ H ₃₂ N ₄ O ₁₀ SCl ₂ (Cu-L)	48.30 (49.24)	3.93 (4.79)	5.00 (5.62)	7.69 (8.78)	216	Brown	74%	13.77
Zn C ₂₀ H ₃₂ N ₄ O ₁₀ SCl ₂ (Zn-L)	50.62 (50.37)	3.75 (4.69)	4.99 (5.86)	8.69 (9.13)	228	Yellow	89%	15.98

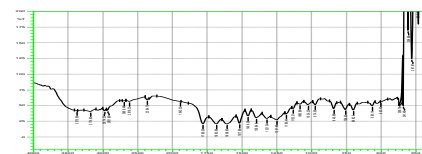


Figure 1: FT-IR spectrum of the ligand L

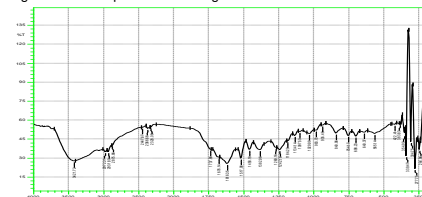


Figure 2: FT-IR spectrum of Nickel complex

FT-IR Studies: The FT-IR spectrum, figure (1) of the formed ligand showed a unique absorption band at 1608 cm⁻¹ belongs to the azo-methene group which absent in the spectrum of both starting materials, this evidence supports the formation of ligand through the binding between the NH₂- amino group of amoxicillin and the aldehydic group of cinnamaldehyde. Other indication proves the formation of ligand through the amino group is the absent of asymmetrical stretching band of NH₂- group in addition to the appearance of many stretching absorption bands at 3242 cm⁻¹, 3066 cm⁻¹ and 2974 cm⁻¹ those bands belong to the stretching vibrational mode of N-H amino group, C-H aromatic and C-H aliphatic groups respectively. Other distinguishable absorption band is that which appeared at 1736 cm⁻¹ belongs to C=O of ester. In addition to various vibrational modes of carbonyl group at (1736 and 1673) cm⁻¹ which belong to carbonyls of ester and amide respectively [27]. When observe the FT-IR spectra of all complexes we find that in addition to the presence of origin bands that belonging to the ligand with some modifications including shifting in some bands including C=N by 7 cm in each of zinc, cobalt and copper complexes and by 8 cm for nickel complex and N-H group by 12 cm, 10 cm and 9 cm in each of zinc, cobalt and both nickel and copper respectively which proves that the

complexes coordinated with ligand through these groups and the appearance of others such as M-N, M-O and M-C l which

contributed to the coordination behavior with metal figure (2) for Ni complex and Table (2)

Table 2: FT-IR spectral records of the ligand L and its complexes

Comp.	N-H	C-H Aliph.	C-H Arom.	C=O amide	C=O est	C=N	H ₂ O	M-O	M-N	M-Cl
L1	3242	3066	2974	1673	1736	1608	3373	--	--	---
Zn L1	3230	3067	2978	1674	1737	1601	3423	---	576	385
Co L1	3232	3063	2960	1670	1738	1601	3410	409	518	390
Ni L1	3231	3060	2972	1676	1737	1600	3421	422	555	389
Cu L1	3231	3065	2970	1674	1738	1601	3388	430	520	392

Uv-Vis Studies: The data of UV-Vis spectrum in figure (3) and table (3) for the synthesized ligand noticed that the next electronic transitions $\pi \rightarrow \pi^*$ at 230 nm which corresponded to 43478 cm^{-1} wave number and $n \rightarrow \pi^*$ at 295 nm corresponded to 33898 cm^{-1} .

The results of electronic spectra of complexes synthesized detected the following transitions for cobalt complex ${}^4T_{1g} \rightarrow {}^4T_{2g}$, $4T_{1g} \rightarrow {}^4A_{2g}$ and ${}^4T_{1g} \rightarrow {}^4T_{1g(P)}$ at 861 nm equals to 11614 cm^{-1} , 542 nm equals to 18450 cm^{-1} and 531 nm equals to 18832 cm^{-1} respectively. While

transitions for Nickel complex (figure4) ${}^3A_{1g} \rightarrow {}^3T_{2g}$ was at 892 nm equals to 11211 cm^{-1} , ${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$ was at 631 nm equals to 15841 cm^{-1} and ${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$ was at 432 nm equals to 23148 cm^{-1} . Transitions for copper complex $2E_g \rightarrow {}^2T_{2g}$ was at 890 nm equals to 11236 cm^{-1} in addition to charge transfer transitions at 301 nm, 33223 cm^{-1} and 244 nm, 40984 cm^{-1} . Zinc complex gave only charge transfer transitions at 275 nm and 292 nm equals to 36364 cm^{-1} and 34247 cm^{-1} respectively. In conclusion octahedral geometries were recorded at all these complexes.[28]

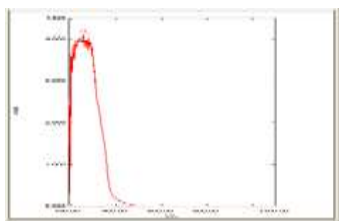


Figure 3: The Uv-Vib. Spectrum of Liagand (L)

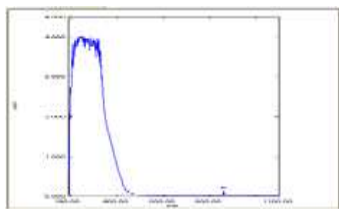


Figure 4: The Uv-Vib. Spectrum of Nickle Complex

Table 3: Uv- Visb spectral records of the ligand L and its complexes

Compound	λ max nm	ν cm^{-1}	Assignment	Geometries
L1	230	43478	$\pi \rightarrow \pi^*$	--
	295	33898	$n \rightarrow \pi^*$	
Co L1	861	11614	${}^4T_{1g} \rightarrow {}^4T_{2g}$	Oh
	542	18450	$4T_{1g} \rightarrow {}^4A_{2g}$	
	531	18832	${}^4T_{1g} \rightarrow {}^4T_{1g(P)}$	
Ni L1	892	11211	${}^3A_{1g} \rightarrow {}^3T_{2g}$	Oh
	631	15841	${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$	
	432	23148	${}^3A_{2g} \rightarrow {}^3T_{1g(P)}$	
Cu L1	890	11236	${}^2E_g \rightarrow {}^2T_{2g}$	Oh
	310	33223	C.T	
	244	40984	C.T	
Zn L1	275	36364	C.T	Oh
	292	34247	C.T	

TGA: The results of complexes are summarized in Table (4). The results reveal a good correspondence with the structures offered from the analytical data Table (1). The complexes TGA curves mention to three steps of ligand decomposition. In firstly, the weight loss corresponds to the 4CH₃ group and chloride group of the ligand. Secondly, the thermal decomposition of C₅₀H₅₂N₆O₁₆SCl₂ of ligand. Third, refer to MO residue (M=Co, Ni, Cu and Zn). Besides these stages, complexes that have coordinated water in CuL and NiL complexes and out of sphere molecules of water appeared extra step of decomposition at all complexes prepared.

Table 4: Thermal analyses data for complexes of the ligand

Comp.	Dissociation Stages	Temp range in TG °C	Weight loss Found (Cal.) %	Decomposition assignment.
CoL	Stage I	64-150	3.38 (3.29)	Out of sphere water
	Stage II	150-382	11.50 (11.93)	- 4CH ₃ , -2Cl
	Stage III	382-559	70.10 (72.22)	- C ₅₀ H ₅₂ N ₆ S ₂ O ₁₁
	Stage IV	> 559	6.3 (6.78)	CoO residue.
NiL	Stage I	64-150	2.20 (2.37)	Out of sphere water Coordination water molecule
	Stage II	150-252	3.32(3.19)	-2Cl, -4CH ₃ .
	Stage III	252-382	11.49 (11.55)	- C ₅₀ H ₅₂ N ₆ S ₂ O ₁₁ .
	Stage IV	382-559	70.81 (70.92)	NiO residue.
CuL	Stage I	64-150	0.99 (0.87)	Out of sphere water Coordination water molecule
	Stage II	150-252	3.22 (3.32)	-2Cl, -4CH ₃ .
	Stage III	252-382	12.17 (12.04)	- C ₅₀ H ₅₂ N ₆ S ₂ O ₁₁ .
	Stage IV	382-559	71.59 (71.69)	CuO residue.
ZnL	Stage I	64-150	0.99 (0.87)	Out of sphere water Coordination water molecule
	Stage II	150-252	3.22 (3.32)	-2Cl, -4CH ₃ .
	Stage III	252-382	12.17 (12.04)	- C ₅₀ H ₅₂ N ₆ S ₂ O ₁₁ .
	Stage IV	382-559	71.59 (71.69)	ZnO residue.
	Stage V	> 559	7.50 (7.30)	

Potentiostatic Polarization Measurement: The Potentiostatic Polarization curves for Carbon steel without and with a new (zinc complex) immersed in 3.5% NaCl solution at temperatures 298K are shown in figures .2 and the results are listed in Table 1.

Table 1: Polarization parameters for different concentrations of metal complexes at Carbon steel in 3.5% M NaCl at 298 K.

Conc. mM	-E _{corr} /mV	i _{corr} /μA.cm ⁻²	-bc/mV/Dec	ba/mV/Dec	W.L. g.m ⁻² .d ⁻¹	P.L mm.y ⁻¹	R _p Ω.cm ²	IE%	CR mpy
Blank	579.5	310.93	90.5	65.2	77.7	0.961	52.92	0	143.61
100	495.5	120.67	120.5	90.2	20	0.534	185.62	61.19	55.73
200	498.0	86.40	138.9	79.8	16	0.332	254.71	72.21	39.90
300	528.3	84.37	173.2	101.8	14	0.301	329.97	72.87	38.97

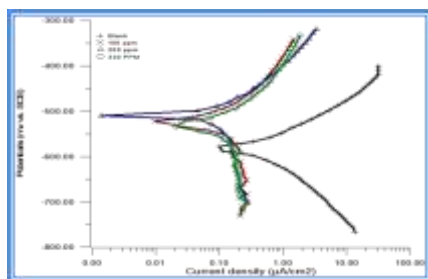


Figure 2: Potentiostatic Polarization curves for Carbon steel, whether uncoated and coated with different metal complexes concentrations in 3.5% NaCl solution at 298 K. Corrosion potential values, corrosion current densities, E_{corr}, cathodic and anodic i_{corr} slopes, bc and ba on the Tafel. R_p polarization resistance which is determined by equation (1), CR corrosion rate determined by equation (2), the inhibition efficacy of a new Amoxicillin derivative can be calculated by the following equations (3) [29].

$$R_p = \left(\frac{B}{i_{corr}} \right) \dots \dots \dots (1)$$

$$R_p = \frac{2.303 (\beta_a + \beta_c) i_{corr}}{\beta_a \beta_c}$$

$$CR = 0.13 \left(\frac{e}{\rho} \right) i_{corr} \dots \dots \dots (2)$$

e: Chemical equivalent ρ: density of mild steel

$$(\%) = \left(\frac{i_{corr} - i_{inh\ corr}}{i_{corr}} \right) \times 100 \dots \dots \dots (3)$$

Where i_{corr} and i_{inhcorr}. The corrosion current density without and with the inhibitor, respectively, is referred to. The inhibition efficiency improved as the concentration of the new Amoxicillin derivative was increased, owing to the complicated impact of the new Amoxicillin derivative on the anodic and cathodic electrochemical corrosion reactions, as a new Amoxicillin derivative includes heteroatoms such as nitrogen, sulfur, and oxygen atoms, as well as the planarity [30].

CONCLUSION

In this article we worked up to prepare a Schiff base ligand derived from amoxicillin and cinnamaldehyde and making use of this ligand in preparing a series of metal complexes including Co, Cu, Zn and Ni metal ions then identify the formation of these compounds by analytical and spectroscopic techniques which in turn prove the bidentate behavior of ligand and octahedral geometry of all complexes. This work describes the corrosion inhibition of Carbon Steel by a new Amoxicillin derivative drugs. Results from Experiments resulting from potentiodynamic measurements was subjected to statistical analysis. The efficiency of inhibition results displayed that the usage of a new Amoxicillin derivative drugs Metal Complex for the prevention of corrosion in Saline solution, may decrease the corrosion rate from 143.61 - 38.97 (mpy) and raise in polarization resistance from 52.92 to 329.97 (Ω.cm²) also the inhibition efficiency goes up 72.87%, because of the adsorption of inhibitor from the solution on the surface of Carbon Steel in Saline solution.

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